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Modeling of Size Effects in Metallic Samples of Confined Volumes

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MODELING OF SIZE EFFECTS IN METALLIC SAMPLES OF CONFINED VOLUMES

A Dissertation

Submitted to the Graduate Faculty of the Louisiana State University and Agricultural and Mechanical College in partial fulfillment of the requirements for the degree of Doctor of Philosophy

in

The Department of Civil and Environmental Engineering

by

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The target of this dissertation was highly interdisciplinary in a way that required getting trainings in many fields of mechanics of materials, atomistic processes in metals, high performance computing, and computational chemistry, which make me stretching beyond my previous knowledge. It would be impossible without the help and support of the people I now wish to acknowledge.

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ABSTRACT

In material science, size effects is described as the variation of material properties as the sample size changes. In this dissertation, the size dependency of the material strength is addressed as size effects. The size effects underlying mechanisms depend on the nature of the considered material. In the case of crystalline metals, size effects in crystalline metals are governed by the dislocations, as the primary deformation mechanism, and their interactions with one another and other defects such as grain boundaries. In this dissertation, the size and strain rate effects of fcc metallic samples of confined volumes are investigated during the nanoindentation and pillar compression experiments using large scale atomistic simulations. Examples of possible benefits include better understanding, controlling, and accelerating the development in new micro- and nano-technology such as microelectromechanical systems (MEMES), nano-coatings, thin films, nanocomposites, ultrafine grain bulk materials, and multilayer systems.

First, the effects of different boundary conditions on the simulation of nanoindentation are investigated using Molecular Dynamics (MD). Next, the available theoretical models of size effects during nanoindentation are evaluated using atomistic simulation. In the next step, the MD simulation is incorporated to investigate the governing mechanism of size effects in a nanoscale single crystal Ni thin film during indentation. The effects of grain boundary (GB) on the sources of size effects are then investigated during the nanoindentation test.

In the next step, the different mechanisms of size effects in fcc metallic samples of confined volumes are studied during high rate compression tests using large scale atomistic simulation. Different mechanisms of size effects, including the dislocation starvation, source exhaustion, and dislocation source length effect are investigated for pillars with different sizes. Furthermore, the size and strain rate effects are then investigated using the observed dislocation length distribution. Finally, the hardening mechanisms in fcc metallic structures during high rate deformations are studied by incorporating the dislocation network properties.
CHAPTER 1
INTRODUCTION: SIZE EFFECTS IN MATERIALS

The variation of material properties as the size of the sample changes is commonly termed as size effects. In the current dissertation, the focus is on the dependency of material strength on the sample size. Depending on the material nature, the size effects mechanism may vary. Here, the size effects in crystalline fcc metals are investigated. In the case of fcc metallic samples, the interaction of dislocations with one another, other defects such as grain boundaries, and free surfaces controls the size effects. Generally, size effects in fcc metals can be divided to two different categories of internal and external size effects. The internal size effects is generally related to some internal length scales such as grain size or mean free path of dislocations. The external size effects, however, is the dependency of material strength on some external length scale such as pillar diameter and indentation depth. In the general cases, the combined external and internal size effects are also available such as the nanoindentation response of a polycrystalline metals.

1.1. Pillars

The experiment on micropillars have been introduced by Uchic and his coworkers (2003,2004,2005). They used the focused ion beam machining to build the micropillars with various sizes. They observed huge size effects in pillars compression test in a way that decreasing the pillar diameter increases its strength. The results for compression experiment on Ni micropillars at room temperature have been presented in Figs. 1.1 and 1.2. Fig. 1.1 shows that the pillar with the diameter of 5 μm is about three times stronger than bulk material sample. The number is about 15 times for diameter of 0.5 μm (Fig. 1.2).

Three groups of Uchic et al. (2009), Kraft et al. (2010), and Greer and De Hosson (2011) have reviewed the experimental and numerical investigation of size effects in pillars. They summarized the experimental procedures, observed results, theoretical models, and numerical simulation of pillar compression test. The pillar compression test schematic is illustrated in Fig. 1.3. The main
difference between the pillar compression test and the conventional large scale one is that the sample is attached to the bulk substrate. In addition to the micropillar compression test, the tension experiment has been also incorporated to study the size effects in pillars. A scanning electron micrograph (SEM) image of a Cu pillar during tension test is shown in Fig. 1.4.

![SEM image of Cu pillar during tension test](image)

**Fig. 1.1.** Response of pure Ni pillars during uniaxial compression experiment at room temperature. (a) Stress-strain curves for pillars with diameters that vary from 40 to 5 μm and a bulk single crystal having approximate dimensions of 2.6 × 2.6 × 7.4 mm. (b) A scanning electron micrograph (SEM) image of a pillar with the diameter of 20 μm at 4% strain. (c) A SEM image of 5 μm diameter pillar at 19% strain during a rapid burst of deformation (After Uchic et al., 2004).

FCC metals have attracted most of the attention in the case of size effects in pillars. Au pillars are of specific interest due to the effect of oxide layers on the test response. The size effects in FCC pillars can be described as follows:

\[ \sigma = A d^{-n} \]  

(1.1)

Where \( \sigma \) is the sample yield or flow stress, \( d \) is the pillar diameter, \( A \) is a constant, and \( n \) is the power-law exponent which is in the range of 0.61-0.97.
Fig. 1.2. Response of Ni3Al-Ta pillars during uniaxial compression experiment at room temperature. (a) Stress-strain curves for pillars with diameters that vary from 20 to 0.5 μm and a bulk single crystal having approximate dimensions of $2.5 \times 2.5 \times 7.5$ mm. (b) A SEM image of a pillar with the diameter of 20 μm at 10% strain during the rapid burst of deformation. (c) A SEM image of 1 μm diameter pillar after the experiment, where the top of the sample is completely sheared off during the rapid strain burst (After Uchic et al., 2004).

The following equation can capture the size effects in the cases of pure FCC metals:

$$\frac{(\tau - \tau_0)b_{ref}}{K_s b_{metal}} = Bd^{-n}$$

where $\tau$ is the yield or flow shear stress, $\tau_0$ is the reference stress, $K_s$ is the anisotropic shear modulus, $b_{ref}$ is the Burgers vector of reference metal, $b_{metal}$ is the Burgers vector of the considered metal, and $B$ is a constant. Uchic et al. (2009) gathered the available data for compression test on FCC pillars (Fig. 1.5). They assumed that the reference stress is negligible, i.e. $\tau_0 \approx 0$, and they selected Ni as the reference metal, i.e. $b_{ref} = b_{Ni}$. Accordingly, the best fit was obtained using $n = 0.6$. In order to explain the size effects observed during the pillar compression experiment, the theoretical model of source truncation, source exhaustion hardening, and weakest link theory have been introduced to explain the nature of size effects.
Fig. 1.3. (a) Schematic of the pillar compression experiment using a nonoindenter with a flat punch. (b) Schematic of the flow response of a microcrystal oriented for single slip. (c) SEM image of pure Ni pillar with the diameter of 5 μm. (d) SEM image of Ni pillar with the diameter of 5 μm after testing (After Uchic et al., 2009).

Fig. 1.4. A SEM image of FIB-fabricated Cu pillar with a diameter of 8 μm during the tension experiment at different strains of: (a) 0.8% (b) 5.4% (After Kiener et al., 2008).
Fig. 1.5. Variation of normalized flow or yield shear stress $\tau b_{Ni}/K_s b_{metal}$ as a function of pillar diameter for different FCC metals. The original experimental data has been reported by Uchic et al. (2004), Dimiduk et al. (2005), Frick et al. (2008), Greer et al. (2005), Greer and Nix (2005), Volkert and Lilleodden (2006), Kraft and Volkert (2006), Ng and Ngan (2008), and Kiener et al. (2006) (After Uchic et al., 2009).

For micropillars, the free surfaces modify the dislocation structure in a way that double-ended dislocations are changed into the single ended sources. The length of the dislocation sources in the micropillars is a function of pillar diameter, and larger pillars have larger source lengths. The required stress to activate a dislocation source is a function of source length in a way that the shorter dislocation source needs larger stresses to be activated. Consequently, the larger the pillar the weaker it is. Parthasarathy et al. (2007) and Rao et al. (2007) have named this mechanism as source truncation. Parthasarathy et al. (2007) described the critical resolved shear stress (CRSS) as follows:
\[ \text{CRSS} = \frac{\alpha G b}{\bar{\lambda}_{\text{max}}} + \tau_0 + 0.5 G b \sqrt{\rho} \]  

(1.3)

where \( \bar{\lambda}_{\text{max}} \) is the mean effective source length, which is related to the pillar diameter, \( \alpha \) is a constant, \( G \) is the shear modulus, \( b \) is the Burgers vector, \( \tau_0 \) is the friction stress, and \( \rho \) is the dislocation density. Parthasarathy et al. (2007) assumed that the upper limit of CRSS is the stress required to move the partial dislocations:

\[ \text{CRSS}(\text{max}) = \frac{\gamma}{b} \]  

(1.4)

where \( \gamma \) is the stacking fault energy. The comparison between the model proposed by Parthasarathy et al. (2007) and micropillar compression test on Ni (Uchic et al., 2005; Dimiduk et al., 2005) and Au (Greer et al., 2005; Volkert and Lilleodeen, 2006) is presented in Fig. 1.6 which demonstrates the capability of the model to capture the reported size effects.

Fig. 1.6. Variation of critical resolved shear stress (CRSS) versus the pillar diameter for: (a) Ni (b) Au. The dotted lines denote the lower and upper standard deviations from the mean as predicted by the model. The original experimental data has been reported by Uchic et al. (2005), Dimiduk et al. (2005), Greer et al. (2005), and Volkert and Lilleodeen (2006) (After Parthasarathy et al., 2007).

In the case of metallic samples of confined volumes, if the available mobile dislocation content may not be sufficient to sustain the imposed strain, the required stress to sustain the plastic flow should be increased. Accordingly, increasing the dislocation density decreases the material strength by providing more mobile dislocations. In the case of micropillar compression test, the dislocation
can escape from the free surface, or source shutdown and mechanical annealing may reduce the available dislocation content. Consequently, higher stresses should apply to induce the target plastic deformation. The resulting hardening mechanisms is commonly called as exhaustion hardening (see, e.g., Rao et al., 2008). In the case of very small pillars, the sample may lose all its dislocation content which leads to very high stresses and commonly termed as dislocation starvation (Greer et al., 2005), which is governed by source-limited activations.

Another mechanism to capture the size effect during micropillar compression experiment is the weakest link theory (Norfleet et al., 2008). The weakest link theory relates the increase in sample strength as its size decreases to the increase in the strength of the weakest slip plane. Norfleet et al. (2008) investigated the weakest link theory during micropillar compression test of Ni pillars with diameters from 1 μm – 20 μm (Fig. 1.7). By measuring the dislocation content in bulk samples and micropillars, they relate the flow shear stress $\tau$ to the lattice-friction stress $\tau_0$, source hardening constant $k_s$, average dislocation source length $\bar{\lambda}$, average strength of the forest dislocations $k_f$, and the scalar density of the forest dislocations $\rho_f$ as follows:

$$
\tau = +k_s G \frac{\ln(\bar{\lambda}/b)}{(\bar{\lambda}/b)} + k_f G b \ln \left( \frac{1}{b \rho_f/2} \right) \sqrt{\rho_f/2}
$$

where $\rho_f = R \rho$ and $R$ is a constant varies with sample geometry and strain value. Due to the difficulties in accurate estimation of $R$, Norfleet et al. (2008) assumed two value of $R = 1/2$ and $R = 3/4$ to form the lower and upper bounds of material flow shear stress. The experimental variation of proportional limit as the pillar diameter changes is compared with that of predicted by Eq. (1.5), both upper and lower bounds, in Fig. 1.8. In the case of samples with diameter larger than ~5 μm, Eq. (1.5) can capture the size effects. However, the model underestimate the shear stress for smaller samples. Accordingly, Eq. (1.5) neglects a hardening term. In order to remove this gap, Norfleet et al. (2008) modified the dislocation density in forest hardening mechanisms term. The modified results are
presented in Fig. 1.9. Although the modification reduces the gap between the experimental results and theoretical prediction, the model still underestimates the sample strength.

![Diagram of stress versus strain for pure Ni (269)-oriented pillars with different diameters](image)

Fig. 1.7. Variation of stress versus strain for pure Ni (2\text{6}9)-oriented pillars with different values of diameters (After Norfleet et al., 2008).

Norfleet et al. (2008) stated that the gap between the experimental results and proposed model can be described using the weakest link theory. They concluded that there is a mean-field limit $\xi^*$ for any crystalline metals. The conventional forest hardening mechanisms can capture the interaction of dislocations for sample with the length scale larger than $\xi^*$. On the other hand, the mean field law breaks down for sample with the length scale smaller than $\xi^*$. In this case, the dislocation length distribution is altered due to the interaction of dislocations with free surfaces, and the largest dislocations, which are the weakest planes, are not available anymore. Accordingly, the stronger forest hardening mechanism is available for samples with the length scales smaller than $\xi^*$. 

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Fig. 1.8. Variation of proportional limit versus pillar diameter for experiment and lower and upper bounds of Eq. (1.5) (After Norfleet et al., 2008).

Fig. 1.9. Variation of proportional limit versus pillar diameter for experiment and lower and upper bounds of Eq. (1.5) corrected by increasing the dislocation density at smaller samples (After Norfleet et al., 2008).
1.2. Nanoindentation

Nanoindentation is a popular experiment to extract the material properties at micro and nanoscale. The nanoindentation experiment includes a hard indenter which impose displacement on the material surface and keep penetrating. The indenter force is then recorded along the indentation. Unlike the conventional indentation test, in which only a hardness number is obtained, the hardness is not constant and may vary during nanoindentation. From the variation of hardness versus the indentation depth, the size effects in materials can be investigated. Fig. 1.10 depicts the variation of variation of hardness versus the indentation depth for the single crystal Cu indented by a Berkovich diamond indenter. It shows that as the indentation depth increases, the hardness decreases.

Fig. 1.10. Nanoindentation of a (111) single crystalline Cu sample indented with a Berkovich diamond indenter (a) A SEM micrographs of sample at indentation depth of 1500 nm (b) Variation of hardness versus the indentation depth (After McElhaney et al., 1998).

To unravel the underlying mechanisms of size effects during nanoindentation, the concept of geometrically necessary dislocations (GNDs), which was introduced by Ashby (1970), are commonly adopted. Ashby (1970) stated that dislocations could be categorized to two groups. The first one,
which is called geometrically necessary dislocations (GNDs), is formed to sustain the imposed
displacement and for the sake of compatibility. The second type, which is called statistically stored
dislocations (SSDs), is a group of dislocations trapping each other in a random way. Various
researchers such as Stelmashenko et al. (1993), DeGuzman et al. (1993), Nix and Gao (1998),
Swadener et al. (2002), and Pugno (2007) have tried to capture the size effects during
nanoindentation by incorporating the concept of GNDs (Fig. 1.1). In Fig. 1.1, the SSDs are excluded;
however, they contribute to the indentation hardness. During the nanindentation, the compatibility
of deformation between the sample and indenter is guaranteed by the nucleation and movement of
GNDs. Accordingly, the total length of GNDs can be obtained based on the indentation depth \( h \), contact
radius \( a_p \), Burgers vector of GNDs \( b_G \), and indenter geometry as follows:

\[
\lambda_G = \int_0^{a_p} 2\pi r \left( \frac{dh}{dr} \right) dr
\]

\( dh/dr \) is a term which include the geometry of indenter. For instance, \( dh/dr = \tan(\theta) = h/a_p \) for a
conical indenter. Consequently, \( \lambda_G \) for a conical indenter can be obtained as below:

\[
\lambda_G = \int_0^{a_p} 2\pi rh \frac{b_G}{b_G a_p} dr = \frac{\pi a_p h}{b_G} = \frac{\pi a_p^2 \tan(\theta)}{b_G}
\]

After obtaining the total dislocation length, the geometry of plastic volume should be defined.
The plastic volume is usually assumed to be a hemisphere which has a radius \( R_{pz} \) proportional to the
contact radius \( a_p \), i.e., \( R_{pz} = f a_p \), where \( f \) is a constant. Accordingly, the volume of plastic volume
can be obtained as below:

\[
V = \frac{2}{3} \pi R_{pz}^3 = \frac{2}{3} \pi f^3 a_p^3
\]

In the next step, the GNDs dislocation density can be obtained as below:

\[
\rho_G = \frac{\lambda_G}{V}
\]

Finally, the Taylor hardening model can relate the dislocation density to the indentation hardness as
follows:
\[ H = 3\sqrt[3]{3\alpha G b_s\sqrt{\rho}} \quad (1.10) \]

where \( \rho \) is the total dislocation density, which include the dislocation densities of GNDs \( \rho_G \) and SSDs \( \rho_S \) and their interaction. Abu Al-Rub and Voyiadjis (2004) assumed a simple form for the total dislocation density and obtained the nanoindentation hardness as follows:

\[ H = 3\sqrt[3]{3\alpha S G b_s \left[ \rho_S^{\beta/2} + \left( \frac{\alpha_G^2 b_G^2 \rho_G}{\alpha_S^2 b_S^2} \right)^{\beta/2} \right]^{1/\beta}} \quad (1.11) \]

where \( \beta \) is a material constant the indices \( G \) and \( S \) represent GNDs and SSDs parameters, respectively.

To demonstrate the capability of the model presented by Abu Al-Rub and Voyiadjis (2004), one can consider the case of conical indenter in which \( \rho_G = 3\tan(\theta)/2f_3 a_p b_G \). As the indentation depth increases, the contact radius \( a_p \) also increases which decreases the GNDs density \( \rho_G \). Decreasing \( \rho_G \) leads to the hardness reduction according to the model presented by Abu Al-Rub and Voyiadjis (2004). Accordingly, the model can capture the typical size effects during nanindentation, which is the case for conical and Berkovich indenters (Fig. 1.10).

Fig. 1.11. Axisymmetric rigid conical and spherical indenters. The imposed deformation is accommodated by GNDs nucleation, and the dislocation loops are assumed to be circular (After Abu Al-Rub and Voyiadjis, 2004).
1.3. Objectives of study

Dislocation network properties control the size effects in crystalline metals. In order to investigate the underlying mechanisms of size effects in metals, the governing atomistic processes should be studied. The molecular dynamics (MD) can be effectively incorporated to model the size effects in metals with full atomistic details. In this dissertation, the size effects during nanoindentation and micropillar compression tests are addressed using large scale atomistic simulation. During the nanoindentation and micropillar compression experiments, the dislocation network evolution is investigated using MD. Accordingly, the relation between the size effects and dislocation network can be extracted.

In order to use the atomistic simulation to model the nanoindentation, the effects of selected boundary conditions on the md simulation results should be thoroughly investigated. Accordingly, the appropriate form of boundary conditions, which induces the least artificial properties, is selected to do the atomistic simulation modelling of nanoindentation experiment. Next, the size effects theories developed by previous researchers, such as the theoretical prediction of dislocation length, dislocation density, plastic volume size, and hardness, are evaluated using the atomistic simulation results. In the next step, the size effects mechanisms for shallow and deep indentation depths are investigated using MD simulation. The coupling effects of grain boundary and nanoindentation depth is a problem that should be carefully addressed, and atomistic simulation can be a great tool to address it.

Beside the nanoindentation, the size effects in micropillar compression test have also gained many attentions. Accordingly, the large scale atomistic simulation is incorporated here to study the size effects mechanisms for pillars with various sizes. Furthermore, the coupling effects of applied strain rate and pillar size are addressed here incorporating large scale atomistic simulations. The dislocation network properties are extracted from the atomistic simulation results. Accordingly, the coupling effects of strain rate and pillar size are captured using the dislocation network properties.
Finally, the microstructural investigation of the hardening mechanism in fcc crystals during high rate deformations is conducted using large scale atomistic simulation. The effect of strain rate is then captured using the evolution of dislocation network.

1.4. Chapters content

The current dissertation consists of three major parts. The first part includes Chapters 2, 3, 4, and 5 is devoted to study the size effects during nanoindentation experiment using large scale atomistic simulation for both single crystal and bi-crystal samples. In this part, the theoretical models of size effects are investigated using the results of MD simulation. Also, the underlying mechanisms of size effects during nanoindentation experiment are addressed using MD simulation. In the second part, which contains Chapters 6 and 7, the coupling effects of strain rate and pillar size are studied using large scale atomistic simulation during micropillar compression test. In the Last part, which contains Chapter 7, the microstructural investigation of the hardening mechanism in fcc bulk crystals is addressed during high rate deformations using large scale atomistic simulations.

In Chapter 2, the effect of different boundary conditions on the simulation of nanoindentation is investigated using Molecular Dynamics (MD). The MD simulations of nanoindentation on Ni single crystal thin films are conducted using various boundary conditions and thicknesses. The coupling effects of indenter size and boundary conditions are studied using the spherical indenter with different radii. Silicon substrate is used as one of the boundary conditions. Three different atomic potentials including embedded-atom method (EAM), Tersoff, and Lennard–Jones (LJ) are incorporated to model the atomic interactions of Ni-Ni, Si-Si, and Ni-Si, respectively. The effect of boundary conditions on the elastic behavior of thin films with various thicknesses is investigated. The results show that the effect of boundary conditions on the elastic response is a function of the film thickness and indenter radius, such that as the thickness increases and the indenter radius decreases, the boundary conditions effects become less significant. Next, the nucleation and early evolution of dislocations in the simulated samples is addressed. Three different patterns of
dislocation structures are observed which are governed by two mechanisms of indentation and bending. Finally, the incipient plasticity in thin films with different boundary conditions and thicknesses is investigated. The results show that the dislocation structure controls the mean contact pressure at the onset of plasticity.

In Chapter 3, the size effects during nanoindentation in Ni thin films are investigated using large scale atomistic simulation. The main focus of this chapter is to evaluate the available theoretical models of size effects during nanoindentation using atomistic simulation. First, the dislocation nucleation and evolution in the simulated samples are studied. In the next step, the plastic zone size is obtained for each sample at several indentation depths incorporating the dislocation visualization. The results show that the plastic zone size divided by the contact radius is not a constant factor and varies as the indentation depth changes. The total length of dislocations located in the plastic zone is measured in the simulated samples and compared to that of the corresponding theoretical models. The results obtained from the atomistic simulation verify the theoretical predictions of the dislocation length. Next, the variation of hardness obtained directly from the molecular dynamics outputs, which is the indentation force over the contact area, is studied. In the case of conical indenter, the theoretical predictions of hardness have been verified using both experiments and simulations, and the current simulation shows the same trend, i.e. the hardness decreases as the indentation depth increases. However, in the cases of flat indenters, the theoretical models have not been validated using any experiments or simulations. Here, in the cases of flat indenters, the simulation results verify the theoretical predictions of hardness. They show that the hardness increases as the indentation depth increases. The variation of dislocation density as a function of indentation depth is then studied. In the case of nanoindentation experiment, the validity of Taylor hardening model, i.e. the relation between the hardening and dislocation density, which has not been previously studied with full atomistic details, is investigated. Accordingly, the hardness obtained
directly from the simulation is compared with the one calculated from the dislocation density and theoretical size effects models.

In Chapter 4, the large scale atomistic simulation is incorporated to investigate the size effects in a nanoscale single crystal Ni thin film during indentation. The results show that the hardness decreases as the dislocation density increases, and the forest hardening model cannot capture the strength size effects during nanoindentation at small length scales. It is observed that the size effects is initially controlled by dislocation nucleation and source exhaustion. As the indentation depth increases, the dislocation length and density increase. Consequently, the number of dislocation sources and their characteristic length increase which decreases the material strength. Finally, increasing the dislocation length and density, the dislocation interaction mechanism also becomes important.

In Chapter 5, the effects of grain boundary (GB) on the sources of size effects are investigated. Up to now, several studies have been conducted to address the role of GBs in size effects from the atomistic point of view. However, a study which addresses the effects of GB on different governing mechanisms of size effects as the sample length scale varies has not been presented yet. Here, samples with different length scales are studied to capture the role of GB in size effects as the grain size changes. The response of single and bi-crystal Ni thin films with two different sizes are studied during nanoindentation experiment using large scale atomistic simulation. Various symmetric and asymmetric tilt GBs are incorporated to study the effects of GB geometry on the response of samples during nanoindentation. The sources of size effects are analyzed in each sample using the atomistic information obtained from the simulations. The results show that the size effects mechanism influenced by GBs changes from dislocation nucleation and source exhaustion to the forest hardening mechanism as the grain size increases. In the case of small bi-crystal samples, dislocation nucleation and source exhaustion govern the size effects. The GB contributes to the dislocation nucleation beneath the indenter which reduces the strength of sample by providing required dislocations to
sustain the imposed plastic deformation. Also, the GB itself is the source of defects which can affect the sample strength depending on the indentation depth at which the dislocation is nucleated from the grain boundary. Increasing the indentation depth, some of the dislocations are blocked by the GB. However, there is no noticeable additional hardness due to the dislocations blockage by GB. Furthermore, the results show that the coherent twin boundaries shows the best performance during the nanoindentation. In the case of large bi-crystal samples, the GB does not influence the size effects at lower indentation depths where the source exhaustion is the controlling mechanism of size effects. However, at higher indentation depths, the dislocation interaction with GB contributes to the forest hardening mechanism and induces some hardening. It is observed that the dislocations are firstly absorbed by the GB. Increasing the indentation depth, some dislocations start dissociating into the next grain. However, it is observed that more dislocations are nucleated in the upper grain. The results show that the main role of GB at larger length scale is to change the pattern of dislocation structure in a way that the dislocations are piled up near the GB which increases the hardness.

In Chapter 6, different mechanisms of size effects in fcc metallic samples of confined volumes during high rate compression tests are investigated using large scale atomistic simulation. Different mechanisms of size effects, including the dislocation starvation, source exhaustion, and dislocation source length effect are investigated for pillars with different sizes. The results show that the controlling mechanisms of size effects depend only on the pillar size and not on the value of applied strain. Dislocation starvation is the governing mechanism for very small pillars, i.e. pillars with diameters less than 30 nm. Increasing the pillar size, the dislocation exhaustion mechanism becomes active and there is no more source-limited activations. Next, the average dislocation source length is obtained and compared for pillars with different sizes. The results show that in the case of high rate deformations, the source length does not depend on the sample size, and the related size effects mechanisms are not active anymore. Also, in the case of high rate deformations, there are no size effects for pristine pillars with the diameters larger than 135 nm. In other words, increasing the strain...
rate decreases the pillar size at which there is no more size effects in the absence of strain gradient. The governing mechanisms of plastic deformation at high strain rate experiments are also different from those of the quasi-static tests. First, the diameter in which the dislocation nucleation at the free surface becomes the dominant mechanism changes from around 200 nm to 30 nm. Next, in the case of the pillars with larger diameters, the plastic deformation is governed by the cross-slip instead of the operation of truncated dislocation sources, which is dominant at slower rates of deformation. In order to study the effects of pillar initial structure on the controlling mechanism of size effects, an initial loading and unloading procedure is conducted on some samples prior to the compression test. In the case of the nanopillars with the height smaller than 45 nm, the results show that the pre-straining does not change the controlling size effects mechanism except for the initial phase of dislocation nucleation. In the case of the pillar with the height of 0.3 μm and diameter of 0.15 μm, however, increasing the initial dislocation density leads to the activation of the forest hardening mechanism. In other words, as the strain increases, the dislocation density increases, which activates the mechanism of dislocation interaction with each other and increases the pillar strength.

In Chapter 7, the relation between the size effects in metallic samples of confined volumes and characteristics of the dislocation network are studied using large scale atomistic simulation. There are only few studies that quantitatively tried to relate the dislocation network properties to the sample size effects. Here, the dislocation length distribution in pillars with different sizes during compression test with different strain rates is studied using large scale atomistic simulation. The size and strain rate effects are then investigated using the observed dislocation length distribution.

In Chapter 8, the hardening mechanism in fcc metallic structures during high rate deformations is investigated by incorporating the dislocation network properties. The large scale atomistic simulation is used to study the variations of dislocation length distribution and its characteristic lengths as the strain rate changes. First, the dislocation length distributions at different strain rates are studied to qualitatively capture the relation between the material strength and
applied strain rate. It is observed that increasing the strain rate decreases the dislocation network lengths. Accordingly, the required stress to activate the dislocation sources increases. Since dislocation movements sustain the imposed plastic flow, higher activation stress leads to material hardening. Furthermore, the results show that the properties of dislocation length distribution at high deformation rates are different from those of lower strain rates due to the activation of cross-slip mechanism at high strain rates. In order to quantitatively describe the relation between the material strength and dislocation network properties as the strain rate varies, the variations of average and maximum dislocation length are investigated in the cases of different applied strain rates. The relation between the material strength and average length of mobile dislocations is captured using an inverse linear equation which shows a good agreement with the atomistic simulation results.
CHAPTER 2
EFFECT OF BOUNDARY CONDITIONS ON THE MD SIMULATION OF NANOINDENTATION

2.1. Introduction

Nanoindentation is one of the most popular experiments to investigate the behavior of materials at the micro and nano scales. During nanoindentation, a small hard indenter is pressed into a sample, and the load versus the penetration depth is recorded. The nanoindentation results show that, unlike the traditional indentation test, the hardness does not have a constant value, and it decreases with increase in the indentation depth (Nix and Gao, 1998; Voyiadjis and Abu Al-Rub, 2005). This phenomenon is termed size effect, and it is usually attributed to the role of the geometrically necessary dislocations that cause the enhanced hardening (Nix and Gao, 1998; Voyiadjis and Abu Al-Rub, 2005). To model the size effect in thin films during nanoindentation, the nature and physical properties of the dislocations should be comprehensively investigated (Nair et al., 2008). Corcoran et al. (1997) studied the effects of defect nucleation on the load-displacement curve of Au single crystal using nanoindentation experiments. Suresh et al. (1999) investigated the film thickness effects on the nanoindentation response and defect nucleation processes of polycrystalline Cu thin films. The effects of grain boundaries in metals can also be investigated using nanoindentation experiments. Conducting nanoindentation tests on a Fe-14 wt. %Si alloy bicrystal, Soer and De Hosson (2005) studied the effects of grain boundary on dislocation nucleation. They found that the nanoindentation near a grain boundary leads to the dislocations pile-up and subsequent propagation across the boundary. Almasri and Voyiadjis (2010) showed that the presence of grain boundary can influence the nanoindentation response in thin films.

One approach to investigate the dislocations behavior of a thin film during nanoindentation is to model the film as a cluster of atoms using molecular dynamics (MD). The MD has been widely used to simulate the nanoindentation experiment for various metals. Using MD simulations, Kelchner et al. (1998) studied the dislocation nucleation of Au under the nanoindentation test.
Zimmerman et al. (2001) investigated the effects of surface step on the nanoindentation test of Au using MD simulations. Li et al. (2002) conducted atomistic simulation of nanoindentation on Cu and Al to study the nucleation and subsequent evolution of defects. Lee et al. (2005) studied the defects nucleation and evolution in Al nanoindentation tests using atomistic simulations. Conducting MD simulations of nanoindentation on bicrystal Ni with a Σ=5 (210) [001] grain boundary, Jang and Farkas (2007) studied the interaction of dislocations with a grain boundary. Nair et al. (2008) investigated the size effect in single crystal and bicrystal Ni using atomistic simulations. Incorporating MD, Peng et al. (2010) simulated the effects of Si substrate on the nanoindentation response of Al. Sun et al. (2014) utilized atomistic simulations to analyze the effects of residual stress on incipient plastic deformation of Cu thin films.

In the MD simulation of nanoindentation, the thin film response is influenced by some factors including the film thickness, crystal orientation, grain size and boundaries, atomic potentials, boundary conditions, temperature, type and size of indenter, and rate of indentation. Comparing the Ni thin films responses of different thicknesses during the nanoindentation, Nair et al. (2008) explored the effects of film thickness using the MD simulation. Komanduri et al. (2000) captured the hardness anisotropy using atomistic simulation of nanoindentation on Al thin films with different orientations. Conducting both MD simulations and experimental tests, Salehinia et al. (2013) studied the effects of indenter size and crystallographic orientation on the nanoindentation response of face-centered cubic metals. Liu et al. (2013) performed atomistic simulation of nanoindentation on nanocrystalline Ni and analyzed the effects of grain size. Nair et al. (2009) investigated the effects of indenter size, atomic potential, in-plane expansion of substrate, and indentation rate on the MD simulation of nanoindentation on Ni thin films. The effect of temperature on the nanoindentation simulation of Cu using MD was explored by Fang et al. (2003).

The boundary conditions effects on the MD simulation of nanoindentation have not been comprehensively addressed in previous research. Parts of the reported results are influenced by
the effect of the boundary conditions which is not considered in the MD simulations. Up to now, various boundary conditions have been incorporated in MD simulations of nanoindentation that may be summarized in four main different categories:

- Fixed boundary conditions at the bottom in the direction of indentation by freezing some atomic layers, free boundary conditions at the top, and periodic boundary conditions in the directions perpendicular to the indentation (Nair et al., 2008; Kelchner et al., 1998; Zimmerman et al., 2001; Nair et al., 2009).
- Fixed boundary conditions in the directions perpendicular to the indentation by freezing some atomic layers and free boundary conditions at the top and bottom surfaces in the direction of indentation (Medyanik et al., 2009; Shao et al., 2010).
- Free surfaces at the top and bottom in the direction of indentation, periodic boundary conditions in the directions that are perpendicular to the indentation, and using a substrate under the thin film (Peng et al., 2010).
- Free surfaces at the top and bottom in the direction of indentation, periodic boundary conditions at the surroundings, and adding the required forces to maintain equilibrium (Li et al., 2002; Lee et al., 2005).

In this chapter, the effect of boundary conditions on the MD simulations of a Ni single crystal thin film subjected to nanoindentation is investigated. Four different types of boundary conditions available in the literature are imposed on the thin films with different thicknesses. Spherical indenters of different radii are used to study the coupling effects of the indenter radius and boundary conditions. The substrate, which is incorporated as one of the boundary conditions, is composed of Si atoms. Three different atomic potentials including embedded-atom method (EAM), Tersoff, and Lennard–Jones (LJ) are used to simulate the atomic interactions of Ni-Ni, Si-Si, and Ni-Si, respectively. The elastic behavior is first investigated in nanoindentation of thin films with different boundary conditions and thicknesses using different indenter radii. In the next step,
the nucleation and early evolution of dislocations in the simulated thin films is studied, and the mechanisms responsible for each type of defect structure are discussed. Finally, the mean contact pressure of simulated samples at the onset of plasticity is investigated.

2.2. Simulation details and methodology

To study the boundary conditions effects on the MD simulation of nanoindentation, Ni thin films with various thicknesses of $t_f = 6$ nm, 8 nm, 10 nm, and 12 nm are generated along the [1 1 1] direction. The remaining dimensions are chosen large enough to avoid finite size effects in these directions (Nair et al., 2008). The dimensions of 27.5 nm and 27.5 nm are chosen along $[\bar{1} 1 0]$ and $[1 1 \bar{2}]$ directions (Fig. 2.1). Four different types of boundary conditions are investigated which are denoted by BC1, BC2, BC3, and BC4 (Fig. 2.2). In the case of BC1, the free boundary conditions are used along the [1 1 1] direction with three fixed atomic layers at the bottom representing a hard substrate. Along the $[\bar{1} 1 0]$ and $[1 1 \bar{2}]$ directions, the periodic boundary conditions are incorporated. In the case of BC2, three atomic layers are fixed on all surfaces along $[\bar{1} 1 0]$ and $[1 1 \bar{2}]$ directions. Free boundary conditions are imposed along the [1 1 1] direction. It is worth mentioning that the fixed atomic layers are not considered as part of the Ni thin films. In the cases of BC3 and BC4, the free boundary conditions are imposed on the surfaces along the [1 1 1] direction. The periodic boundary conditions are applied to the remaining surfaces along $[\bar{1} 1 0]$ and $[1 1 \bar{2}]$ directions. Using BC3 and BC4, the thin film will translate along the indentation force. In the case of BC3, to prevent the whole domain from translational movement, the method introduced by Li et al. (2002) is used. In this method, at each step, the total force is divided by the number of atoms and then applied in the opposite direction to all atoms. In the case of BC4, the thin film is placed on the Si substrate with fixed atomic layers at the bottom. The total thickness of Si substrate including the fixed atomic layers and the space between the thin film and the substrate is 6.5 nm.
Fig. 2.1. Geometry and orientation of the simulated thin films.

Fig. 2.2. Boundary conditions of thin films (a) BC1 (b) BC2 (c) BC3 (d) BC4.
The classical molecular dynamics (MD) is incorporated to simulate the nanoindentation test. The velocity Verlet algorithm with a time step of 1 fs is used for the time integration of Newton's equations of motion. The simulations are performed using the parallel MD code LAMMPS (Plimpton, 1995). At first, all samples are relaxed for 100 ps with the temperature increasing from 0 K to 300 K. In the next step, they are relaxed for 100 ps at 300 K. Finally, the relaxed samples are indented along the [1 1 1] direction with the velocity of 10 m/s, which is widely used in previous simulations (Nair et al., 2008; Peng et al., 2010; Nair et al., 2009), at 300 K. All simulations are conducted using NVT ensemble as implemented in LAMMPS (Plimpton, 1995).

Three different atomic potentials including embedded-atom method (EAM), Tersoff, and Lennard–Jones (LJ) are incorporated to simulate the atomic interactions of Ni-Ni, Si-Si, and Ni-Si, respectively. EAM is a well-established method used in MD to simulate metallic systems (Daw and Baskes, 1984). The EAM interatomic potential $E^{\text{EAM}}$ is defined as follows

$$E^{\text{EAM}}(r_{ij}) = \frac{1}{2} \sum_{i,j} V(r_{ij}) + \sum_i F(\rho_i), \quad \rho_i = \sum_{i \neq j} \varphi(r_{ij})$$

(2.1)

where $V(r_{ij})$ and $F(\rho_i)$ represent the pair interaction and embedding potentials, respectively, and $\varphi(r_{ij})$ is a function of the electron density. The nickel EAM potential developed by Mishin et al. (1999) is incorporated in order to simulate the Ni-Ni interaction. A three-body Tersoff potential (1988) is selected to model Si-Si interaction, and the related Si parameters are presented in Table 2.1. Finally, the Ni-Si interaction is modeled using the LJ potential $E^{\text{LJ}}$ which is defined below:

$$E^{\text{LJ}}(r_{ij}) = 4\varepsilon \left[ \left( \frac{\sigma}{r_{ij}} \right)^{12} - \left( \frac{\sigma}{r_{ij}} \right)^6 \right]$$

(2.2)

where $\sigma$ is the collision diameter at which $E^{\text{LJ}} = 0$ and $\varepsilon$ is the depth of the potential well (see, e.g., Liu et al., 2006). The LJ parameters used to model the Ni-Si interaction ($\varepsilon_{\text{Ni-Si}}$ and $\sigma_{\text{Ni-Si}}$) are obtained using the following equations (Peng et al., 2010):

$$\varepsilon_{\text{Ni-Si}} = \sqrt{\varepsilon_{\text{Ni}} \varepsilon_{\text{Si}}}$$

(2.3)
\[ \sigma_{\text{Ni-Si}} = \frac{\sigma_{\text{Ni}} + \sigma_{\text{Si}}}{2} \]

where \( \varepsilon_{\text{Ni}} \) and \( \sigma_{\text{Ni}} \) are the Ni-Ni and \( \varepsilon_{\text{Si}} \) and \( \sigma_{\text{Si}} \) are the Si-Si LJ parameters. Table 2.2 shows the LJ parameters of Ni –Ni, Si-Si, and Ni-Si interactions. The LJ potential cutoff distance is chosen as 2.5\( \sigma \).

The distance between the thin film and substrate is set equal to the collision diameter of the Ni-Si LJ potential (Peng et al., 2010).

Table 2.1. Tersoff potential parameters of Si-Si.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>3264.7 eV</td>
</tr>
<tr>
<td>B</td>
<td>95.373 eV</td>
</tr>
<tr>
<td>( \lambda_1 )</td>
<td>3.2394 Å(^{-1} )</td>
</tr>
<tr>
<td>( \lambda_2 )</td>
<td>1.3258 Å(^{-1} )</td>
</tr>
<tr>
<td>( \alpha )</td>
<td>0</td>
</tr>
<tr>
<td>( \beta )</td>
<td>0.33675</td>
</tr>
<tr>
<td>( \gamma )</td>
<td>22.956</td>
</tr>
<tr>
<td>( c )</td>
<td>4.8381</td>
</tr>
<tr>
<td>( d )</td>
<td>2.0417</td>
</tr>
<tr>
<td>( \lambda_3 )</td>
<td>( \lambda_2 )</td>
</tr>
<tr>
<td>( R )</td>
<td>3.0 Å</td>
</tr>
<tr>
<td>( D )</td>
<td>0.2 Å</td>
</tr>
</tbody>
</table>

Table 2.2. LJ potential parameters of Ni-Ni, Si-Si, and Ni-Si.

<table>
<thead>
<tr>
<th>Interaction</th>
<th>( \varepsilon ) (J)</th>
<th>( \sigma ) (Å)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni-Ni</td>
<td>8.3134e-20</td>
<td>2.2808</td>
</tr>
<tr>
<td>Si-Si</td>
<td>2.7904e-21</td>
<td>3.8260</td>
</tr>
<tr>
<td>Ni-Si</td>
<td>1.5231e-20</td>
<td>3.0534</td>
</tr>
</tbody>
</table>

The spherical indenter is modeled as a rigid system applying a repulsive force on each atom which is defined as follows (Plimpton, 1995):

\[
F(r) = \begin{cases} 
-Ke(r-R)^2 & \text{if } r < R \\
0 & \text{if } r \geq R
\end{cases}
\]

where \( K \) is the specified force constant which is chosen as 10 eV/Å\(^3 \) (Lilleoddena et al., 2003), \( r \) is the distance of atom to the indenter center, and \( R \) is the indenter radius. In order to investigate the effect of indenter radius on the nanoindentation results, two different radii of 3nm and 10nm are
selected. The method used by Saraev and Miller (2006) is incorporated here to calculate the contact area $A$. In this method, first, atoms located in direct contact with the indenter are identified. Next, a triangular 2D-mesh is created from the projections of those atoms onto the indentation surface. The contact area is the summation of all triangle areas.

It should be noted that the true indentation depth $h$ is different from the displacement of the indenter tip $d$. Generally, the indentation depth can be obtained from the tip displacement by excluding the thin film deflection due to the concentrated load. Here, the penetration depth is calculated by incorporating the contact area. In the case of a spherical indenter, the indentation depth can be expressed as follows

$$h = R - \sqrt{R^2 - a_c^2} \quad (2.6)$$

where $a_c = \sqrt{A/\pi}$ is the contact radius.

The centrosymmetry parameter ($CSP$) introduced by Kelchner et al. (1998) is incorporated to visualize the dislocations which are defined as follows:

$$CSP = \sum_{i=1}^{N_p} |\mathbf{R}_i + \mathbf{R}_{i+N_p}|^2 \quad (2.7)$$

where $\mathbf{R}_i$ and $\mathbf{R}_{i+N_p}$ are positions of the $i^{th}$ pair of neighbors relative to the considered atom, and $N_p$ is equal to 6 for fcc materials. $CSP$ will vanish in the case of elastic deformations. The software VMD is used to visualize the defects (Humphrey et al., 1996). Avoiding the display of local atomic vibration as an atomic defect, the cutoff value should be used for the centrosymmetry parameter ($CSP_{\text{cutoff}}$), i.e. if $CSP_i < CSP_{\text{cutoff}}$, the $i^{th}$ atom should not be visualized (Lee et al., 2005). Furthermore, in order to capture clear shots of partial dislocations and stacking faults, point defects should be excluded. Balatov and Cai (2006) recommended that $1 \text{ Å}^2 < CSP < 3 \text{ Å}^2$ correspond to the cores of the partial dislocations and $CSP \geq 3 \text{ Å}^2$ represents the stacking faults. Various values of $0.25 \text{ Å}^2$, $0.5 \text{ Å}^2$, $1 \text{ Å}^2$, and $1.5 \text{ Å}^2$ are used as $CSP_{\text{cutoff}}$. It is observed that the partial dislocations and
stacking faults are visualized in all cases without loss of any details. However, nearly all point defects are vanished in the case of \( CSP_{\text{cutoff}} = 1.5 \text{ Å}^2 \). Hence, the value of 1.5 \( \text{Å}^2 \) is chosen for the \( CSP_{\text{cutoff}} \).

**2.3. Results and Discussions**

2.3.1. Elastic response

Samples with different boundary conditions and various thicknesses of \( t_f = 6 \text{ nm}, 8 \text{ nm}, 10 \text{ nm}, \) and 12 nm are indented using the indenter with a radius of \( R = 10 \text{ nm} \). Figs. 2.3 and 2.4 represent the variation of the indentation load \( P \) as a function of the penetration depth \( h \). Simulating thin films with BC1 and different thicknesses, Nair et al. (2008) showed that the elastic modulus of thin films increases as the thickness decreases. However, they incorporated the tip displacement instead of the true penetration depth. Fig. 2.4 (a) shows that the elastic modulus of BC1 samples is independent of the sample thickness. In the cases of BC2, BC3, and BC4, on the other hand, the elastic modulus is a function of the film thickness, even when the penetration depth is properly calculated. Fig. 2.3 shows that that the elastic response of samples with BC1 is always an upper bound. The difference between the elastic responses of the other boundary condition types with that of BC1 decreases as the film thickness increases.

The Hertzian theory is incorporated to further investigate the elastic response, in which, the indentation force of a spherical indenter is defined as follows (Lilleoddena et al., 2003):

\[
P = \frac{4}{3} E (R)^{1/2} (h)^{3/2}
\]

where \( E \) is the reduced elastic modulus. In the case of a rigid indenter, \( E \) becomes the plane strain elastic modulus of the sample. The elastic modulus of each sample is that of the best-fitting Hertzian solution obtained by the least squares method. The elastic modulus of samples with different boundary conditions is normalized using that of BC1. Fig. 2.5 shows the variation of the normalized elastic modulus versus the sample thickness.

28
The results show that BC2 has the smallest and BC1 has the largest elastic modulus. The elastic modulus of BC3 is smaller than that of BC1 but larger than that of BC2. In the case of BC4, i.e. using the Si substrate, the elastic modulus is smaller than that of BC1 but larger than that of BC3. Considering the simulated thin films with various thicknesses, the smallest differences between the elastic response of BC1 with those of BC2, BC3 and BC4 occur in the case of 12 nm thickness samples. However, the largest differences are obtained in the case of the 6 nm thickness sample.

Fig. 2.3. Nanoindentation responses ($R = 10$ nm) of thin films with different boundary conditions and various thicknesses of (a) 6 nm (b) 8 nm (c) 10 nm (d) 12 nm.
Generally, it is observed that as the film thickness increases, the effect of the boundary conditions on the elastic response becomes less significant.

Fig. 2.4. Nanoindentation responses ($R = 10$ nm) of thin films with different thicknesses and boundary conditions types of (a) BC1 (b) BC2 (c) BC3 (d) BC4.

In order to study the indenter radius effect on the elastic response of thin films, samples with different boundary conditions and varying thicknesses of $t_f = 6$ nm, 8 nm, 10 nm, and 12 nm are indented using an indenter with the radius of $R = 3$ nm. Fig. 2.6 shows the $P - h$ curves for all thin films. It can be observed that the elastic responses of all boundary conditions types are quite similar. The results show that the effect of the boundary conditions is a function of $R / t_f$. It means
that as the thickness increases and the indenter radius decreases, the boundary conditions effect on the elastic response of thin films becomes less significant.

Fig. 2.5. Normalized elastic modulus versus the sample thickness ($R = 10$ nm).

2.3.2. Defect nucleation and evolution

The atomistic visualization of defects is incorporated here in order to study the boundary conditions effect on the MD simulation of thin films subjected to nanoindentation. Early-stage defects of thin films with different boundary conditions types and thicknesses indented by the indenter with $R = 3$ nm and 10 nm are studied. Considering the location of the defects nucleation at the onset of plasticity and the slip planes on which they are grown, three different patterns of early-stage defects are observed (Fig. 2.7):
- **Type I**: The initial dislocations are nucleated beneath the indenter. Two faces of embryonic dislocation loops evolve on (1 1 1) and (1 1 1) planes. Eventually, it is transformed to the tetrahedral sessile lock.

- **Type II**: The dislocations are firstly nucleated beneath the indenter and evolve on the plane parallel to the indentation plane, i.e. (1 1 1). Increasing the indentation depth, some dislocations are gradually nucleated and grown with the pattern similar to Type I.

- **Type III**: The initial dislocation nucleation occurs at the bottom of the sample. Dislocations then evolve towards the top surface on {1 1 1} planes.

Fig. 2.6. Nanoindentation responses ($R = 3$ nm) of thin films with different boundary conditions and various thicknesses of (a) 6 nm (b) 8 nm (c) 10 nm (d) 12 nm.
Fig. 2.7. Defect nucleation and evolution of (a) Type I (b) Type II (c) Type III.
In all three cases, the Shockley partial dislocations bounding the stacking fault domains are nucleated and evolved on \{111\} planes which commonly occur in the case of fcc (111) nanoindentation tests (see, e.g., Lee et al., 2005).

Two mechanisms of indentation and bending control the pattern of dislocation nucleation and growth. In the case of BC1, since the bottom of the sample is fixed, there is no bending. Hence, the defect nucleation and evolution of Type I always occurs which is governed by the indentation mechanism. In the case of BC2 and BC3, for large values of $R / t_f$, the bending mechanism is dominant and defects pattern of Type III is observed. Decreasing $R / t_f$, the indentation mechanism becomes significant and defect nucleation and evolution of Type II occurs which is controlled by both the bending and indentation mechanisms. If the value of $R / t_f$ is further decreased, the indentation will be the dominant mechanism (Type I). In the case of BC4, for large values of $R / t_f$, both bending and indentation mechanisms are important (Type II). Decreasing $R / t_f$, accordingly, bending becomes less significant, and the indentation mechanism governs the dislocation nucleation and evolution pattern (Type I).

The effect of dislocation nucleation and evolution on the nanoindentation response of a thin film is also studied here. Fig. 2.8 shows the nanoindentation response of samples visualized in Fig. 2.7. In the case of the sample with dislocations pattern of Type I, Fig. 2.8 (a) indicates that the first load relaxation occurs when the first dislocations are nucleated beneath the indenter tip. Two faces of embryonic dislocation loops evolve on (111) and (1 1 ̅1) planes. In the next step, another load relaxation occurs and the shape is transformed to the tetrahedral sessile lock. After this stage, there are some local glides of partial dislocation loops emanating from the faces of the tetrahedron which are leading to the overall increase of the indentation load. A similar behavior has been observed by Lee et al. (2005). Fig 8 (b) shows the nanoindentation response of the sample with dislocation nucleation and evolution pattern of Type II. The first load relaxation is coincident with the initial dislocation nucleation beneath the indenter. The dislocations evolve on the plane parallel to the
indentation plane, i.e. (1 1 1). Some dislocations also start nucleating with a similar pattern of Type I. At this stage, the total dislocation nucleation pattern becomes complex and the relation between the load-displacement curve, and dislocations pattern is very complicated. In the case of the sample with dislocations pattern of Type III, Fig. 2.8 (c) shows that the first load relaxation occurs when the dislocations are nucleated at the sample bottom due to the bending effect. In the next stage, the nanoindentation response is oscillatory due to the complex motions of dislocations, but it still results in overall increase of the indentation load.

Among the three observed patterns, only the dislocation nucleation and evolution of Type I have been previously reported in the MD simulation of fcc (111) nanoindentation tests (see, e.g., Lee et al., 2005). It is possible that the dislocation patterns of Type II and Type III are artificially produced due to the unrealistic rate of indentation, size of the samples, or the type of ensemble used in MD simulation. Some of the simulated samples are indented with much lower rates, for example 1 m/s, and the same dislocation patterns are observed. Molecular static method is also incorporated to verify the dislocation patterns. Again, similar dislocation patterns are produced even when the indentation is static. To verify that the dislocation patterns description of Type II and Type III are valid in the case of thin films of larger length scales during the nanoindentation, additional BC2 samples with the dimensions of 100 nm and 100 nm along [1 1 0] and [1 1 2] directions and thicknesses of \( t_f = 20 \) nm and 40 nm are simulated. The total numbers of atoms in these samples are 19,923,555 and 39,691,240 respectively. The samples are indented using the indenter of \( R = 35 \) nm. Fig. 2.9 presents the patterns of dislocation nucleation and evolution during the nanoindentation. It can be seen that the dislocation patterns of Type III and Type II are formed in the samples with \( t_f = 20 \) nm and 40 nm, respectively. In the case of ensemble type used in MD simulation, three BC3 samples with different thicknesses of \( t_f = 6 \) nm, 10 nm, and 12 nm are simulated by incorporating NPT scheme with lateral pressure equal to zero. The first two samples (i.e., \( t_f = 6 \) nm and 10 nm) are indented using the indenter of \( R = 10 \) nm, and the last one (i.e., \( t_f = 35 \) nm)
12 nm) is indented using the indenter of $R = 3$ nm. Fig. 2.10 presents the pattern of dislocation nucleation and evolution in all samples. It is clear that the samples with $t_f = 6$ nm, 10 nm, and 12 nm have the dislocation nucleation patterns of Type III, Type II, and Type I, respectively.

2.3.3. Incipient plasticity

The indenter mean contact pressure ($p_m$), which is defined by $p_m = P/A$, is usually incorporated in order to study the plasticity initiation in thin films. Simulating thin films with BC1 and different thicknesses, Nair et al. (2008) showed that the mean contact pressure related to the plasticity initiation ($p_m^\gamma$) is mostly independent of the film thickness. However, they stated that as the film thickness increases, the plasticity initiation occurs at larger indentation depths. In order to investigate the effect of film thickness on $p_m^\gamma$, the $p_m - h$ curves of simulated samples with different boundary conditions types and varying thicknesses indented by $R = 10$ nm indenter are shown in Fig. 2.11. In the case of BC1, the results show the same trend as Nair et al. (2008) and the $p_m^\gamma$ does not depend on the film thickness. However, the indentation depth in which the first dislocation is emitted is also independent of the film thickness. The contradiction between the observed results and those of Nair et al. (2008) is due to the fact that they used tip displacement instead of true penetration depth. In the cases of BC2 and BC3 and in the range of the simulated thicknesses, it is obvious that $p_m^\gamma$ increases as the thickness increases. In the case of BC4, there is an increase in $p_m^\gamma$ from 6 nm thickness sample to 8 nm thickness sample; however, $p_m^\gamma$ is nearly independent of the film thickness for thicknesses greater than 8 nm.

To investigate the effect of the boundary conditions on the mean contact pressure at the onset of plasticity, the variation of $p_m$ as a function of $h$ for thin films of 6 nm and 12 nm thicknesses and various boundary conditions are shown in Fig. 2.12 ($R = 10$ nm). In the case of 6 nm thickness film, the boundary conditions type with stiffer elastic response has larger $p_m^\gamma$ values. In the case of 12 nm thickness film, at the onset of plasticity, all types of boundary conditions have nearly the same mean contact pressure.
Fig. 2.8. Nanoindentation responses of thin films with different dislocation nucleation patterns of (a) Type I (b) Type II (c) Type III.
It is apparent that as the thickness increases, the effect of the boundary conditions on $p_m^y$ becomes less significant. In the case of $R = 3$ nm (Fig. 2.13), all samples with different types of boundary conditions and thicknesses have nearly the same value of $p_m^y$. In other words, the effect of the boundary conditions on $p_m^y$ disappears.

The effect of the boundary conditions on the contact pressure at the onset of plasticity can be explained by studying the early-stage defect nucleation and evolution pattern. All the thin films with BC1 and different thicknesses have dislocation pattern of Type I, and Fig. 2.11 (a) shows that the $p_m^y$ value of these samples does not depend on the film thickness. Furthermore, samples indented by the indenter of $R = 3$ nm have the dislocation pattern of Type I. Again, $p_m^y$ is independent of the boundary conditions or thickness (Fig. 2.13). The results show that as far as the
dislocation pattern of Type I occurs, $p_m^X$ is independent of the film thickness or boundary conditions. In the case of dislocation patterns of Type II and Type III, $p_m^X$ increases as the thickness increases (Fig. 2.11). Finally, simulating the samples with different boundary conditions and thicknesses indented with the indenter of the same size shows that the dislocation patterns of Type I and Type III have the largest and smallest $p_m^X$ values, respectively.

![Fig. 2.10. Defect nucleation and evolution of BC3 samples simulated using the NPT ensemble with the thicknesses of (a) 6 nm (b) 10 nm (c) 12 nm.](image-url)
Fig. 2.11. Mean contact pressure versus the indentation depth of simulated samples ($R = 10 \text{ nm}$) with different thicknesses and boundary conditions types of (a) BC1 (b) BC2 (c) BC3 (d) BC4.

Fig. 2.12. Mean contact pressure versus the indentation depth of simulated samples ($R = 10 \text{ nm}$) with different boundary conditions and thicknesses of (a) 6 nm (b) 12 nm.
2.4. Conclusions

The current study addresses the effect of boundary conditions on MD simulations of nanoindentation tests. Ni thin films of different boundary conditions and thicknesses indented with indenters of different radii are simulated using MD. The indentation is performed along the [1 1 1] direction with a velocity of 10 m/s at 300 K. Silicon substrate is selected as one of the boundary conditions. Three different atomic potentials of embedded-atom method, Tersoff, and Lennard–Jones are used to model the atomic interactions of Ni-Ni, Si-Si, and Ni-Si, respectively.

First, the effects of the boundary conditions, film thickness $t_f$, and indenter radius $R$ on the elastic response of Ni thin film are investigated. The elastic modulus is obtained by fitting the Hertzian solution to the elastic part of the indentation response using the least squares method. Thin films with different thicknesses and various types of boundary conditions are indented using the indenter of $R = 10$ nm. The smallest and largest differences between the elastic responses of samples with different boundary conditions types are observed in the case of 12 nm and 6 nm thicknesses thin films, respectively. On the other hand, in the case of the indenter with $R = 3$ nm and in the range of the simulated thicknesses, the effect of the boundary conditions on the elastic responses of thin films is negligible. Generally, the effect of the boundary conditions on the film elastic response is a function of $R / t_f$. In other words, as the thickness increases and the indenter radius decreases, the boundary conditions effect on the elastic response becomes less significant.

In the next step, the centrosymmetry parameter is incorporated to visualize the dislocations. The typical observed defects are the Shockley partial dislocations bounding the stacking fault domains which nucleate and evolve on {1 1 1} planes. Two mechanisms of indentation and bending govern three patterns of the initial dislocation nucleation and the slip planes as follows:
- **Type I**: If the indentation mechanism is dominant, the initial dislocation nucleation will occur beneath the indenter. Two faces of embryonic dislocation loops will be grown on \((1 \bar{1} 1)\) and \((1 1 \bar{1})\) planes and then transformed to the tetrahedral sessile lock.

- **Type II**: In the case of coupling effects of indentation and bending mechanisms, the dislocations are firstly nucleated beneath the indenter and evolve on the \((1 1 1)\) plane. Increasing the indentation depth, some dislocations are gradually nucleated and grown with the pattern similar to **Type I**.

- **Type III**: If bending becomes the dominant mechanism, the initial dislocation nucleation will be located at the bottom of the sample. The dislocations will grow and move towards the top surface on \(\{1 1 1\}\) planes.

To verify that the validity of dislocation patterns description is not limited to the small samples, thin films with larger length scales are simulated during the nanoindentation. Again, the same dislocation patterns are observed.

![Dislocation Patterns](image)

**Fig. 2.13.** Mean contact pressure versus the indentation depth of simulated samples \((R = 3 \text{ nm})\) with different boundary conditions and thicknesses of (a) 6 nm (b) 12 nm.
Finally, the effects of boundary conditions and film thickness on the mean contact pressure at the onset of plasticity are investigated. It is observed that the pattern of dislocation nucleation governs the effects of boundary conditions and film thickness on the contact pressure of plasticity initiation. In the case of dislocation pattern of Type I, the pressure at incipient plasticity is independent of the film thickness or the boundary conditions. In the case of dislocation patterns of Type II and Type III, however, pressure at the onset of plasticity increases as the thickness increases. Furthermore, simulating the samples with different boundary conditions and thicknesses indented with the indenter of same size shows that the dislocation patterns of Type I and Type III have the largest and smallest contact pressure at the onset of plasticity, respectively.
CHAPTER 3
LARGE SCALE ATOMISTIC SIMULATION OF SIZE EFFECTS DURING NANOINDENTATION: DISLOCATION LENGTH AND HARDNESS

3.1. Introduction

Nowadays, metallic devices of micro and nano scales play a key role in many modern technologies. The main focuses are thin films and nanolayer systems which have significantly impacted the fast-developing fields of information technologies, bridge and highway monitoring and assessment, biomedicine, and intelligent control and automation of many different industries. When it comes to the smaller scales of micro and nano, the mechanical behavior of metals strongly depends on the material length scales which is commonly termed as size effects (Nix and Gao, 1998). Size effects in metals are generally attributed to the dislocations (Voyiadjis and Abu Al-Rub, 2005). The behavior of dislocations and their interactions with other types of defects such as grain boundaries govern the size effects (Nair et al., 2008). Size effects have been reported in different experiments such as microtorsion, microbending, and nanoindentation. Fleck et al. (1994) conducted microtorsion experiments on the polycrystalline copper wires and showed that a wire with smaller diameter has a greater torsional strength. Stolken and Evans (1998) performed microbending test on Ni thin foils. Again, a thinner foil exhibited a greater flexural strength.

In the case of nanoindentation, the results show that, unlike the traditional indentation test, the hardness does not have a constant value, and it should be described as a function of indentation depth (Nix and Gao, 1998; Voyiadjis and Abu Al-Rub, 2005; Swadener et al., 2002). Nix and Gao (1998) presented a simple model of geometrically necessary dislocations to capture the size effect in crystalline materials during the nanoindentation with a conical indenter. Swadener et al. (2002) extended the Nix and Gao model to indenters of various shapes and compared the model with the experimental results. Following that, Pugno (2007) modified the previous models to remove the limitations for small indentation depths and derived a general scaling law for size effect in
nanoindentation based on the surface-to-volume ratio of the domain. In the case of conical and Berkovich indenters, the theoretical models state that the hardness decreases as the indentation depth decreases which has been observed during several experiments (Nix and Gao, 1998; Voyiadjis and Abu Al-Rub, 2005; Swadener et al., 2002; Pugno, 2007). In the cases of flat indenters, theoretical models predict that the hardness increases as the indentation depth increases (Swadener et al., 2002; Pugno, 2007). However, the models, to the authors' knowledge, have not been verified by any experiments or simulations. Grain boundaries in metals can also influence the size effect during nanoindentation. Soer and De Hosson (2005) studied the effects of grain boundary on dislocation nucleation during the nanoindentation tests on a Fe-14 wt. %Si alloy bicrystal. They found that the nanoindentation near a grain boundary leads to the dislocations pile-up and subsequent propagation across the boundary. Almasri and Voyiadjis (2010) investigated the effect of grain boundary on the nanoindentation response of thin films. They showed that besides the general size effect during the nanoindentation test, interaction of dislocations with grain boundaries may locally increase the hardness.

The nature and physical properties of the dislocations should be fully investigated to model the size effect in metallic thin films during nanoindentation. Molecular dynamics (MD) is a powerful tool to simulate the nanoindentation test with all the atomistic details. The nanoindentation experiment of various metals has been simulated using MD. Kelchner et al. (1998) conducted atomistic simulation of nanoindentation to study the dislocation nucleation of Au. Using MD simulations, Zimmerman et al. (2001) investigated the effects of surface step on the nanoindentation test of Au. Li et al (2002) studied the dislocation nucleation and evolution of Cu and Al under the nanoindentation test using MD simulations. Lee et al. (2005) simulated the defects nucleation and evolution of Al during the nanoindentation test using MD. Jang and Farkas (2007) performed MD simulations of nanoindentation on bicrystal Ni with a Σ=5 (210) [001] grain boundary to study the interaction of dislocations with a grain boundary. Using atomistic simulation,
Nair et al. (2008) addressed the size effect in single crystal and bicrystal Ni. Peng et al. (2010) conducted MD simulation of nanoindentation to study the effects of Si substrate on the response of Al thin film. Sun et al. (2014) analyzed the effects of residual stress on incipient plastic deformation of Cu thin films using atomistic simulations. Gao et al. (2014) simulated indentation and scratching using MD to investigate the dislocations nucleation, evolution, and reaction. Yaghoobi and Voyiadjis (2014) studied the effect of boundary conditions on the MD simulation of nanoindentation by incorporating various boundary conditions and thicknesses. Several indenter tip geometries have been incorporated in the previous MD simulations of nanoindentation. However, none of the previous MD simulations have studied the dislocation nucleation and evolution during nanoindentation using flat indenters.

As explained by Durst et al. (2005), the size effect in metals are mainly controlled by dislocation density which is obtained from two factors of plastic zone size and the total length of dislocations located in the plastic zone beneath the indenter. Nix and Gao (1998) assumed that the plastic zone is a hemisphere with the radius equal to the contact radius. However, Feng and Nix (2004) showed that the plastic zone radius is much bigger in the case of MgO. Durst et al. (2005) stated that the radius of the plastic zone is equal to the contact radius multiplied by a constant factor which is in the range of 0-3.5. They incorporated the results of a finite element simulation of the indentation process to determine the size of the plastic zone. Recently, Gao et al. (2014) incorporated the assumption stated by Durst et al. (2005) for the plastic zone size and obtained the dislocation length during the nanoindentation using atomistic simulation. However, the dislocation length obtained from the MD simulation does not agree well with that of the theoretical prediction.

In this chapter, the size effect in a Ni single crystal thin film during nanoindentation experiment is studied using large scale atomistic simulation. The main focus of this paper is to evaluate the presented size effects theories of nanoindentation experiment using atomistic simulation. In addition to the conical indenter, flat indenters with geometries of cylindrical and
right square prismatic are incorporated. First, the dislocation nucleation and evolution are discussed during the nanoindentation. In the next step, the plastic zone size is obtained during the nanoindentation for several indentation depths using the dislocation visualization results. Total length of dislocations located in the plastic zone is measured during the nanoindentation and compared with the available theoretical models. Next, the variation of hardness obtained directly from the MD simulation, which is the indentation force over the contact area, is studied. The variation of dislocation density with the indentation depth is investigated. Finally, the validity of Taylor hardening model for nanoindentation experiment, which has not been previously studied with full atomistic detail, is investigated. The hardness obtained directly from the MD simulation is compared with the one which is determined using the dislocation density and theoretical models.

3.2. Simulation details and methodology

To model the size effect in thin films during nanoindentation, a Ni thin film with the dimensions of 1200 Å, 1200 Å, and 600 Å along [1 1 0], [1 1 2], and [1 1 1] directions is generated and simulated using the classical molecular dynamics (MD). The total number of atoms in the samples is 79,473,309. Three different indenter shapes of cylindrical, right square prismatic, and conical are incorporated. The cylindrical indenter has a radius of \( r_1 = 4.8 \) nm. In the case of right square prismatic indenter, the indentation surface is a 7.5 nm \( \times \) 7.5 nm square. The blunt conical indenter has the smaller radius of \( r_2 = 0.3 \) nm and the cone semi-angle is selected as \( \theta = 56.31^\circ \). The boundary conditions introduced by Li et al. (2002) are selected in which the periodic boundary conditions are applied to the surfaces along [1 1 0] and [1 1 2] directions. The free boundary conditions are selected at the bottom of the sample which ensures that the dislocations do not bounce back from the sample bottom. To prevent the whole domain from translational movement, at each step, the total force is divided by the number of atoms and then applied in the opposite direction to all atoms. Yaghoobi and Voyiadjis (2014) have shown that the effect of the chosen boundary conditions on the dislocation pattern, hardness, and indentation force is a function of the
sample thickness. Four different types of boundary conditions were investigated including the one which is selected in the current paper. The results showed that as the thickness increases, the boundary condition effects becomes less significant and at some points it becomes negligible.

The reason that the authors did not incorporate the boundary conditions with fixed atomic layers at the bottom is due to the fact that this boundary conditions type blocks the dislocations at the sample bottom and the nucleated dislocation loops will bounce back from the bottom surface which can produce artificial hardness and increase the dislocation length in the plastic zone (Lee et al., 2005). In the present paper, the effect of the selected boundary conditions on the nanoindentation response of the simulated sample is negligible which is demonstrated in Appendix A.

To integrate Newton’s equations of motion, the velocity Verlet algorithm with a time step of 2 fs is incorporated. The parallel MD code LAMMPS is used (Plimpton, 1995). All samples are relaxed for 100 ps with the temperature increasing from 1 K to 300 K. In the next step, they are relaxed for 100 ps at 300 K. Finally, the relaxed samples are indented along the [1 1 1] direction with the velocity of 10 m/s, which is widely used in previous simulations (Nair et al., 2008; Peng et al., 2010; Gao et al., 2014; Yaghoobi et al., 2014; Remington et al., 2014), at 300 K. All simulations are performed using NPT ensemble. The nickel embedded-atom method (EAM) potential developed by Mishin et al. (1999) is incorporated to simulate the atomic interaction of Ni-Ni. EAM is a common method to simulate metallic systems using MD (Daw and Baskes, 1984). The EAM interatomic potential $E_{EAM}$ is defined as follows:

$$E_{EAM}(r_{ij}) = \frac{1}{2} \sum_{i,j} V(r_{ij}) + \sum_{l} F(\rho_{l}), \quad \rho_{l} = \sum_{i \neq j} \varphi(r_{ij})$$

(3.1)

where $V(r_{ij})$ and $F(\rho_{l})$ represent the pair interaction and embedding potentials, respectively, and $\varphi(r_{ij})$ is a function of the electron density.
A repulsive potential is incorporated here to describe the interaction between the indenter and Ni atom, which is described as follows (Plimpton, 1995):

\[ E^{\text{ind}}(r) = \varepsilon (r - r_c)^2 \quad r < r_c \]  

(3.2)

where \( \varepsilon \) is the specified force constant which is chosen as 1 eV/\( \text{Å}^2 \), \( r \) is the distance from particle to the indenter surface, and \( r_c \) is the cutoff distance which is selected as 0.3 nm. To calculate the contact area \( A \), first, atoms located in direct contact with the indenter are identified. Next, a triangular 2D-mesh is created from the projections of those atoms onto the indentation surface. The contact area is the summation of all triangle areas. The method is explained in more detail by Saraev and Miller (2006).

In the case of conical indenter, true indentation depth \( h \), which is slightly different from the displacement of the indenter tip \( d \), can be obtained from geometrical relations. Here, the indentation depth of a conical indenter is calculated by incorporating the contact area as follows

\[ h = \frac{(a_c - a_0)}{\tan \theta} \]  

(3.3)

where \( a_c = \sqrt{A/\pi} \) is the contact radius, and \( a_0 = r_2 + r_c(1/\cos \theta - \tan \theta) \).

There are no geometrical relations to find the indentation depth of cylindrical and right square prismatic indenters, and the tip displacement \( d \) is incorporated instead of \( h \).

The Crystal Analysis Tool developed by Stukowski and his co-workers (Stukowski and Albe, 2010; Stukowski et al, 2012; Stukowski, 2012; Stukowski, 2014) is incorporated to visualize the dislocations and provide additional information such as dislocation length and Burgers vector. The Crystal Analysis Tool, which is a more advanced version of the Dislocation Extraction Algorithm (DXA) tool, is developed based on the common-neighbor analysis method (Faken and Jonsson, 1994). First, atomic arrangements and structures are identified. In the next step, a pattern matching algorithm groups atoms into so-called clusters. A Delaunay mesh is generated that connects all atoms and the corresponding elastic deformation gradient tensor is computed for the constructed
mesh. If a dislocation intersects a tessellation element, the elastic deformation gradient is multi-valued. Next, trial circuits are built on the interface mesh to define the dislocation lines. Finally, the code provides a one-dimensional line representation of the dislocation segments. Furthermore, the stacking faults, which are identified using the Crystal Analysis Tool, are also visualized. The software OVITO (Stukowski, 2010) and Paraview (Henderson, 2007) are used to visualize the defects and measure the dislocation length.

3.3. Results and Discussions

3.3.1. Dislocation nucleation and evolution

The variation of indentation load $P$ as a function of penetration depth $h$ is depicted in Fig. 3.1. In the case of samples indented by flat indenters, Swadener et al. (2002) and Pugno (2007) predicted that the hardness increases as the indentation depth increases. Considering the fact that the projected contact area of the cylindrical and right square prismatic indenters are nearly constant throughout the indentation process, the theoretical models state that the indentation force of cylindrical and right square prismatic indenters should increase as the indentation depth increases due to the increase in hardness. Fig. 3.1 shows that, after an initial drop, as the indentation depth increases, the indentation forces of the cylindrical and right square prismatic indenters increase. Fig. 3.2 presents the dislocation nucleation and evolution of a sample indented by the cylindrical indenter. The first dislocation is Shockley partial dislocation with the Burgers vector of $1/6 [1 2 1]$ nucleated at $d \approx 0.7$ nm and evolved on $(1 \bar{1} 1)$ plane. Fig. 3.2 shows that the initial dislocation nucleation occurs at the periphery of the indenter base. Unlike the samples indented by the spherical indenter (Yaghoobi and Voyiadjis, 2014), there is no sharp load relaxation immediately after the generation of the first dislocation. In the next step, two more dislocations are nucleated at the periphery of the indenter base. The load drop occurs prior to the formation of the first large shear loop at $d \approx 0.96$ nm. Gradually, other Shockley partial dislocations are nucleated with the Burgers vector of $1/6 (1 1 2)$ and evolved on $\{1 1 1\}$ planes. Furthermore, a few perfect
dislocations, Hirth partial dislocations, and stair-rod partial dislocations with Burgers vectors of $1/2\langle 1\ 1\ 0 \rangle$, $1/3\langle 0\ 0\ 1\rangle$, and $1/6\langle 0\ 1\ 1\rangle$, respectively, are produced. In Figs. 3.2, 3.5, and 3.7, the color of Shockley, Hirth, and stair-rod partial dislocations and perfect dislocations are green, yellow, blue, and red, respectively. Hirth and stair-rod partial dislocations are formed due to intersection of two partial dislocations as follows

\begin{align}
\text{Hirth partial dislocations} & : \frac{1}{6}[\overline{1}\ 2\ 1] + \frac{1}{6}[1\ \overline{2}\ 1] \rightarrow \frac{1}{3}[0\ 0\ 1] \tag{3.4} \\
\text{Stair-rod partial dislocations} & : \frac{1}{6}[\overline{1}\ 2\ \overline{1}] + \frac{1}{6}[1\ \overline{1}\ 2] \rightarrow \frac{1}{6}[0\ 1\ 1] \tag{3.5}
\end{align}

First prismatic loop is released at $d \approx 1.05$ nm and glide along $[0\ \overline{1}\ \overline{1}]$ direction downward leading to the load relaxation. The next load relaxation coincides with the glide of the second prismatic loop along $[\overline{1}\ \overline{1}\ 0]$ direction at $d \approx 1.12$ nm. The dislocations loop emission during the nanoindentation is presented in Fig. 3.3 showing that the glide occurs along three directions of $[0\ \overline{1}\ \overline{1}]$, $[\overline{1}\ \overline{1}\ 0]$, and $[\overline{1}\ 0\ \overline{1}]$. The prismatic loops pass the bottom surface and leave parallelogram surface steps. Fig. 3.4 shows the contact surface and impression of nanoindentation at $d \approx 3.68$ nm. It can be observed that the cylindrical indenter is in full contact with the surface. Comparing the shape of the prismatic loops presented by Lee et al. (2005) in the case of sample indented by the spherical indenter and Fig. 3.2, it can be concluded that the general shape of the prismatic loop are independent of the indenter geometry. However, in the simulated sample, it is sometimes observed that instead of two Shockley partial dislocations, there is a perfect dislocation on part of the surrounding edges (Fig. 3.2). Moreover, one may observe that there are some Hirth and stair-rod partial dislocations in addition to the Shockley partial dislocation in the side of the prismatic loops which makes it slightly nonuniform (Fig. 3.2).

In the case of the right square prismatic indenter, the first dislocation is Shockley partial dislocation with the Burgers vector of $1/6[1\ 1\ 2]$ nucleated at $d \approx 0.62$ nm and evolved on $(1\ 1\ \overline{1})$ plane (Fig. 3.5). Similar to the sample indented by the cylindrical indenter, the first nucleation
occurs at the periphery of the indenter base without any sudden drop in the indentation force. Again, the first large load relaxation occurs prior to the formation of the first large shear loop at $d \approx 0.76$ nm. The rest of the dislocation nucleation and evolution pattern is similar to the sample indented by the cylindrical indenter. Fig. 3.6 illustrates the contact surface and indentation impression at $d \approx 3.68$ nm. It shows that the indenter is in full contact with the surface.

![Graphs](image)

Fig. 3.1. Nanoindentation responses of thin films indented by the (a) cylindrical indenter (b) right square prismatic indenter (c) conical indenter.
Fig. 3.2. Defect nucleation and evolution of Ni thin film indented by the cylindrical indenter at (a) $d \approx 0.70$ nm (b) $d \approx 0.86$ nm (c) $d \approx 0.96$ nm (d) $d \approx 1.02$ nm (e) $d \approx 1.05$ nm (f) $d \approx 1.12$ nm.
Fig. 3.3. Prismatic loops forming and movement in Ni thin film indented by the cylindrical indenter during nanoindentation (a) side view (b) top view.

Fig. 3.4. The visualization of sample top surface under nanoindentation using the cylindrical indenter at $d \approx 3.68$ nm (a) contact surface (b) indentation impression.
Fig. 3.5. Defect nucleation and evolution of Ni thin film indented by the right square prismatic indenter at (a) $d \approx 0.62$ nm (b) $d \approx 0.76$ nm (c) $d \approx 2.56$ nm.

Fig. 3.6. The visualization of sample top surface under nanoindentation using the right square prismatic indenter at $d \approx 3.68$ nm (a) contact surface (b) indentation impression.
Dislocation nucleation and evolution of the sample indented by the conical indenter is presented in Fig. 3.7. The first dislocation is nucleated at \( h \approx d = 0.54 \, \text{nm} \) which is a Shockley partial dislocation with the Burgers vector of \( 1/6 \langle 2 \ 1 \ 1 \rangle \) evolved on \( \langle \bar{1} \ 1 \ 1 \rangle \) plane. There is no sharp load relaxation after the generation of first dislocation. After the first dislocation nucleation, another Shockley partial dislocation is nucleated parallel to the first one which is connected to it with two more partial dislocations on \( \langle 1 \ 1 \ 1 \rangle \) plane, and the reaction produces a Hirth partial dislocation. Most of the dislocations are Shockley partial dislocations with the Burgers vector of \( 1/6 \langle 1 \ 1 \ 2 \rangle \). However, there are also a few perfect dislocations, Hirth and stair-rod partial dislocations with the Burgers vectors of \( 1/2 \langle 1 \ 1 \ 0 \rangle \), \( 1/3 \langle 0 \ 0 \ 1 \rangle \), and \( 1/6 \langle 0 \ 1 \ 1 \rangle \), respectively. First prismatic loop is released at \( h \approx 2.8 \, \text{nm} \) \( (d \approx 2.82 \, \text{nm}) \) and glides along \( \langle \bar{1} \ 0 \ 1 \rangle \) direction downward to the bottom surface without any sharp drop in the indentation force. Again, increasing the indentation depth, more prismatic loops are emitted which glide along three directions of \( \langle 0 \ 1 \ 1 \rangle \), \( \langle 1 \ 1 \ 0 \rangle \), and \( \langle 1 \ 0 \ 1 \rangle \). The contact surface and indentation impression are also illustrated at \( h \approx 8.08 \, \text{nm} \) \( (d \approx 8.36 \, \text{nm}) \) in Fig. 3.8. Comparing Figs (2), (5), and (7), one can see that the shape of the prismatic loop is independent of the indenter geometry.

3.3.2. Plastic zone size and total dislocation length

During the nanoindentation, dislocations are stored in a volume which is termed plastic zone. Nix and Gao (1998) assumed that the plastic zone is a hemisphere with the radius equal to the contact radius \( R_{pz} = a_c \). Durst et al. (2005) stated that \( R_{pz} = f a_c \) where \( f \) is a constant in the range of 0-3.5. They selected \( f = 1.9 \) in their simulations. Here, the following assumption is considered in order to measure the plastic zone size directly from the MD simulation:

- The dislocation prismatic loops glide toward the bottom of the sample. Hence, they should not be considered as a measure of the plastic zone size. Otherwise, the size of the plastic zone becomes unreasonably large. In other words, the plastic zone size is determined based
on the furthest dislocation which is attached to the main body of dislocations beneath the indenter.

Considering the above assumption, the reported plastic zone size may have a little variation due to the existence of dislocation loops. For example, Fig. 3.9 shows that the plastic zone sizes determined before and after the prismatic loop emission are not the same. In the first case (i.e. before emission), the plastic size contains the prismatic loop which is still attached to the main

Fig. 3.7. Defect nucleation and evolution of Ni thin film indented by the conical indenter at (a) \( d \approx 0.54 \text{ nm} \) (b) \( d \approx 0.58 \text{ nm} \) (c) \( d \approx 2.82 \text{ nm} \).
body of dislocations. However, the prismatic loop is not part of the plastic zone after its emission. Despite of the stated fact, the obtained results still can give a fair range of the plastic zone size during the indentation. Here, it is assumed that the plastic zone is a hemisphere with a radius of $R_{pz} = f a_c$. In the case of right square prismatic indenter, the equivalent contact radius is incorporated which is equal to $a_c = \sqrt{A/\pi}$. The dislocation length is obtained at several indentation depths using different values of $f = 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, 4.5, \text{ and } 5$. The value of $R_{pz}$ is then defined and confirmed using the visualization results. Moreover, using the visualization of dislocations, the obtained value of $f$ is further refined, and if the plastic zone is not completely contained in the tested ranges, i.e. $f > 5$, the final value of $f$ is obtained using the visualization results.

![Fig. 3.8](a) (b)

Fig. 3.8. The visualization of sample top surface under nanoindentation using the conical indenter at $d \approx 8.36$ nm (a) contact surface (b) indentation impression.

Figs. 3.10 and 3.11 show the variations of $R_{pz}$ and $f$ versus the indentation depth. In the literature, $f$ has been assumed to be a constant value which is independent of indentation depth (see e.g., Durst et al., 2005). However, Fig. 3.11 shows that $f$ is a function of indentation depth. In the case of the sample indented by the cylindrical indenter, $f$ is in the range of 2-9. In the case of the sample indented by the right square prismatic indenter, $f$ varies between 3.0 and 11.6. The lowest change in the value of $f$ belongs to the sample indented by the conical indenter in which $1.5 \leq f \leq 4.5$. 

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5.2. In the case of the sample indented by the right square prismatic indenter, the obtained range of $f$ cannot be compared to the previous works due to the fact that the equivalent contact radius is incorporated which might influence the observed results. In the case of conical indenter, which is a self-similar indenter, the maximum value of $f$ observed during the nanoindentation simulation is 5.2 which is slightly larger than those which have been previously proposed (see Durst et al., 2005). However, in the case of cylindrical indenter, the maximum value of $f$ is 9 which is much larger than that of the self-similar indenter.

Fig. 3.9. Dislocation pattern (a) before the prismatic loop emission (b) after the prismatic loop emission.
Fig. 3.10. Radius of plastic zone \( R_{pz} \) versus the indentation depth of thin films indented by the indenters with different geometries of cylindrical, right square prismatic, and conical.

Fig. 3.11. The parameter \( f \) versus the indentation depth of thin films indented by the indenters with different geometries of cylindrical, right square prismatic, and conical.

Nix and Gao (1998) presented a simple expression of dislocation length during nanoindentation with the conical indenter based on the concept of geometrically necessary dislocations. The dislocation length produced by the conical indenter is expressed as follows (Nix and Gao, 1998; Swadener et al., 2002):
\[ \lambda_{\text{co}} = \frac{\pi a_c h}{b} \quad (3.6) \]

Pugno (2007) approximated the indentation surface using the staircase-like surface and proposed the following equation for the dislocation length during the nanoindentation

\[ \lambda = \frac{S}{b} \quad (3.7) \]

where \( S = \Omega - A \) and \( \Omega \) is the total contact surface (both vertical and horizontal). In the case of cylindrical indenter, the total dislocation length can be determined using Eq. (3.7) as follows

\[ \lambda_{\text{cy}} = \frac{2\pi a_c h}{b} \quad (3.8) \]

Incorporating the same procedure, the total dislocation length during the nanoindentation using the right square prismatic indenter can be obtained as follows

\[ \lambda_{\text{pr}} = \frac{4ch}{b} \quad (3.9) \]

where \( c = \sqrt{A} \). In Eqs. (3.6), (3.8), and (3.9), \( h \) should be modified to remove the initial elastic part of deformation. For example, assuming \( h_p \) is the indentation depth for plasticity initiation, Eq. (3.9) mistakenly shows that there is a dislocation length equal to \( 4ch/b \) even for \( h < h_p \). In other words, part of \( h \), which is very small but not negligible, is due to the elastic deformation which should not be taken into account in dislocation length predictions. Here, the first data is used to modify \( h \) in Eqs. (3.6), (3.8), and (3.9).

Fig. 3.12 compares the dislocation length obtained from MD simulation and theoretical models expressed in Eqs. (3.6), (3.8), and (3.9), which shows a good agreement. In the cases of samples indented by the right square prismatic and conical indenters, all the dislocation length obtained from MD simulation are larger than the theoretical model predictions. However, in the case of the sample indented by the cylindrical indenter, there are few points in which the theoretical predictions of dislocation lengths are slightly larger than those obtained from MD simulation.
Fig. 3.12. Total dislocation length obtained from simulation and theoretical models in samples indented by the (a) cylindrical indenter (b) right square prismatic indenter (c) conical indenter.

The difference between the theoretical models and MD simulation can be explained as follows:

- Theoretical models predict only the length of geometrically necessary dislocations; however, the dislocation length measured from MD simulation includes both statistically
stored and geometrically necessary dislocations. For example, Fig. 3.12 (c) shows that the difference between the values of dislocation length obtained from simulation and theoretical model increases as the indentation depth increases. It can be due to the fact that the length of statistically stored dislocations increases in this case as the indentation depth increases. However, Figs. 3.12 (a) and (b) show that the length of statistically stored dislocations in the samples indented by cylindrical and right square prismatic indenters does not considerably change as indentation depth increases.

- In Eqs. (3.6), (3.8), and (3.9), the Burgers vector of the Shockley partial dislocations is incorporated. Although the majority of dislocations are Shockley partial dislocations, there are also a few Hirth and stair-rod partial and perfect dislocations which have different Burgers vectors.

During the dislocation length measurement, the prismatic loops reaching out of the plastic zone are not considered. It can be a reason of why few theoretical predictions of dislocation length are slightly larger than those obtained from MD simulation.

3.3.3. Hardness and dislocation density

Fig. 3.13 shows the variation of hardness $H$, which is defined by $H = P / A$, as a function of indentation depth $h$. In the cases of samples indented by the cylindrical and right square prismatic indenters, Figs. 3.13 (a) and (b) show that the hardness increases as the indentation depth increases which has been previously predicted by Swadener et al. (2002) and Pugno (2007). However, a drop in hardness occurs at small indentation depth prior to the formation of first large dislocation shear loop. In the case of sample indented by the conical indenter, $H$ increases as $h$ decreases which has been predicted theoretically and shown experimentally (see e.g., Nix and Gao, 1998).
Fig. 3.13. Hardness directly obtained from MD simulation \((H = P/A)\) in samples indented by the (a) cylindrical indenter (b) right square prismatic indenter (c) conical indenter.
Some theoretical models have been proposed based on the concept of geometrically necessary dislocations to capture the size effect during nanoindentation (Nix and Gao, 1998; Swadener et al., 2002; Pugno, 2007). In these models, the dislocation density $\rho$ is related to the shear strength using Taylor hardening model as follows:

$$\tau = \alpha \mu b \sqrt{\rho}$$  \hspace{1cm} (3.10)

where $\mu$ is the shear modulus which is 125 GPa for Ni, and $\alpha$ is a constant. For FCC metals, $\alpha$ is in the range of 0.3-0.6 (Swadener et al., 2002). In this chapter, $\alpha = 0.6$ and 0.4 are selected for the flat and conical indenters, respectively. The dislocation density can be stated as $\rho = \lambda/V$ where $\lambda$ is total dislocation length and $V$ is the plastic zone volume. It is trivial that the Taylor hardening model cannot capture the shear strength before the dislocation nucleation. In the next step, the hardness $H$ can be obtained using the von Mises flow rule and the Tabor factor of 3 as follows:

$$\sigma = \sqrt{3}\tau$$

$$H = 3\sigma = 3\sqrt{3}\alpha \mu b \sqrt{\rho}$$ \hspace{1cm} (3.11)

where $\sigma$ is the flow stress. Eq. (3.11) cannot describe the initial drop in hardness and the variation of hardness prior to the plastic initiation. Moreover, a constant $\overline{H}$ should be added to $H$ in Eq. (3.11) to address the elastic part of hardness which is obtained using the best curve fitting method. Accordingly, Eq. (3.11) is modified as follows:

$$H = \overline{H} + 3\sqrt{3}\alpha \mu b \sqrt{\rho}$$ \hspace{1cm} (3.12)

Here, the validity of Taylor hardening model, i.e. the relation between the hardening and dislocation density, is investigated in the case of nanoindentation experiment. To this end, $H = P/A$ is compared with the hardness calculated using the Taylor hardening model. The dislocation length $\lambda$ and plastic zone volume $V$ are directly obtained from MD simulation. The plastic zone volume can be expressed as $V = (2/3)\pi (f a_c)^3 - V_{\text{indenter}}$, where $V_{\text{indenter}}$ is part of the indenter volume located in the plastic zone. Finally, the hardness can be calculated using Eqs. (3.10)-(3.12). As explained in Durst et al. (2005), the plastic zone size which is defined based on the region that
contains all the plastic deformation does not lead to an accurate prediction of the dislocation density and hardness. As an example, Fig. 3.14 shows the variation of the dislocation density versus indentation depth using the plastic zone size and dislocation length presented in Figs. 3.10-3.12 in the case of the sample indented by the right square prismatic indenter. As indentation depth increases, the dislocation density should increase (see Swadener et al., 2002; Pugno, 2007). However, Fig. 3.14 shows that as \( h \) increases, \( \rho \) first decreases and then becomes constant. Accordingly, the effective plastic zone size is incorporated here based on the best prediction of hardness directly obtained from the MD simulation, which is \( H = P/A \). The parameter \( f \) is selected as 4.5, 4.5, and 1.5 for the samples indented by the cylindrical, right square prismatic, and conical indenters, respectively. Dislocation density is obtained by measuring the length of dislocations located in the effective plastic zone. Fig. 3.15 shows the variation of the dislocation density as a function of the indentation depth. The results show that as \( h \) increases, \( \rho \) increases in the case of samples indented by the cylindrical and right square prismatic indenters. However, in the case of the sample indented by the conical indenter, \( \rho \) decreases as \( h \) increases. The obtained results are in agreement with the theoretical predictions of Swadener et al. (2002) and Pugno (2007). In the next step, using the obtained dislocation density and incorporating Eq. (3.12), the hardness is calculated and compared to the one obtained directly from MD simulation, which is the indentation force divided by the contact area. In the case of samples indented by the cylindrical and right square prismatic indenters, \( \bar{H} \) is selected as 10 GPa. However, in the case of the sample indented by the conical indenter, Eq. (3.11) still gives reasonable values of \( H \). Fig. 3.16 shows that the hardness obtained from dislocation density using the theoretical models and \( H = P/A \) compare quite well.

In the current chapter, the thickness is selected 60 nm to eliminate the boundary conditions effects. To demonstrate this, the sample indented by the conical indenter is selected. Two different boundary conditions types are incorporated:

- Three atomic layers at the bottom are fixed to represent a hard substrate (BC1).
The free boundary conditions are selected at the bottom of the sample. Next, at each step, the total force is divided by the number of atoms and then applied in the opposite direction to all atoms (BC3).

Fig. 3.14. Dislocation density of sample indented by the right square prismatic indenter using the plastic zone size obtained from the dislocation visualization results.

The abbreviations for the boundary conditions types are chosen based on the work of Yaghoobi and Voyiadjis (2014). Fig. 3.17 shows the hardness for samples with boundary conditions types of BC1 and BC3. It shows that the hardness obtained by using both boundary conditions types of BC1 and BC3 are nearly identical. However, after $h \approx 6$ nm, the dislocation loops are reaching the bottom of BC1 sample and eventually getting trapped which induces slight artificial hardness. Accordingly, one can see that the hardness of BC1 sample is slightly larger than that of BC3 sample after $h \approx 6$ nm. Fig. 3.18 shows the dislocation length of BC1 and BC3 samples located in the effective plastic zone, i.e. $f = 1.5$. The results show that the dislocation length for samples with both boundary condition types are nearly the same before $h \approx 6$ nm. However, following that, the dislocation length of BC1 sample becomes larger than that of BC3 sample. It is due to the fact that the dislocation loops bounce back from the bottom surface in the case of BC1. Hence, in the indentation depth region where the dislocation loops do not reach the bottom, the hardness and dislocation length are not affected by the choice of the boundary conditions (Figs. 3.17 and 3.18).
However, after the dislocation loops reach the bottom surface, if the boundary conditions block them, it will induce artificial hardness and dislocation length.

Fig. 3.15. Density of dislocations located in the effective plastic zone for samples indented by the (a) cylindrical indenter (b) right square prismatic indenter (c) conical indenter.
Fig. 3.16. Comparison of hardness directly obtained from MD simulation with the one obtained using dislocation density and theoretical models in samples indented by the (a) cylindrical indenter (b) right square prismatic indenter (c) conical indenter.
Fig. 3.17. Hardness of the samples with different boundary conditions types of BC1 and BC3 indented by the conical indenter.

Fig. 3.18. Dislocation Length located in the effective plastic zone for samples with different boundary conditions types of BC1 and BC3 indented by the conical indenter.

3.4. Conclusions

The size effect in metallic thin films during the nanoindentation is addressed incorporating large scale MD. In the cases of samples indented by the flat indenters, it is observed that the initial dislocation nucleation occurs at the periphery of the indenter base. Moreover, it is observed that in addition to the Shockley partial dislocations with the Burgers vector of \(1/6\ 1\ 1\ 2\), there are also a few perfect, Hirth partial, and stair-rod partial dislocations with the Burgers vectors of \(1/2\ 1\ 1\ 0\),
1/3 (0 0 1), and 1/6 (0 1 1), respectively. The results show that the shape of induced prismatic loops is independent of the indenter geometry.

The plastic zone size is obtained for each sample at several indentation depths incorporating the dislocation visualization results. In the case of conical indenter, the results show that the plastic zone size is slightly larger than the one previously proposed. However, in the cases of flat indenters, the plastic zone size is much larger. Furthermore, unlike the previous assumptions, the value of the plastic zone size divided by the contact radius is not constant, and it is a function of the indentation depth. The total length of dislocations located in the plastic zone is then measured, and the theoretical predictions of dislocation length are evaluated using the obtained results. The simulation results validate the theoretical predictions of dislocation length.

In the cases of samples indented by flat indenters, the theoretical models of Swadener et al. (2002) and Pugno (2007), which predict that the hardness increases as the indentation depth increases, are verified using the obtained results. In the case of the sample indented by the conical indenter, hardness increases as the indentation depth decreases which has been shown in several experiments (see e.g., Nix and Gao, 1998).

Finally, the variation of dislocation density versus the indentation depth is illustrated. It is shown that using the size of plastic zone which contains all plastic deformation does not lead to the accurate prediction of dislocation density and hardness. In the cases of samples indented by the flat indenters, the results show that the dislocation density increases as the indentation depth increases. However, in the case of the sample indented by the conical indenter, as the indentation depth increases, the dislocation density decreases. The validity of the Taylor hardening model is investigated with full atomistic details for nanoindentation experiments. To this end, the hardness obtained directly from the MD simulation is compared with the one calculated from the dislocation density and the Taylor hardening model. The results show that the Taylor hardening model can successfully capture the size effects during nanoindentation.
CHAPTER 4
ATOMISTIC SIMULATION OF SIZE EFFECTS IN SINGLE-CRYSTALLINE METALS OF CONFINED VOLUMES DURING NANOINDENTATION

4.1. Introduction

In bulk metallic systems, size effects are governed by the interaction of dislocations with each other, which is commonly termed forest hardening (Nix and Gao, 1998; Abu Al-Rub and Voyiadjis, 2004; Voyiadjis and Abu Al-Rub, 2005; Greer, 2013). Taylor-like hardening models are commonly incorporated to capture the forest hardening. As an example, the effects of strain gradient on material strength is modeled using the relation between the dislocation density and strength which is commonly described by the Taylor hardening models (Nix and Gao, 1998; Abu Al-Rub and Voyiadjis, 2004; Voyiadjis and Abu Al-Rub, 2005). There are various types of forest hardening models. However, the models generally state that the strength increases as the dislocation density increases (Nix and Gao, 1998; Abu Al-Rub and Voyiadjis, 2004; Voyiadjis and Abu Al-Rub, 2005). In the cases of nanosize samples, however, several experiments have shown that the sources of size effects are different from those of bulk samples (see, e.g., Kraft et al., 2010). Kraft et al. (2010), Greer and De Hosson (2011), and Greer (2013) have reviewed different types of size effects models in metallic samples of confined volumes. Three models of source exhaustion hardening, source truncation, and weakest link theory are usually incorporated to describe these size effects (see, e.g., Greer, 2013). In the region of small length scales, the samples may lose the dislocation sources due to the source shut down, mechanical annealing, or dislocation starvation (Greer et al., 2005; Espinoza et al., 2005; Kiener and Minor, 2011). Consequently, the reduced mobile dislocation density is insufficient and the applied stress should be increased to sustain the plastic deformation which is termed exhaustion hardening (Rao et al., 2008; El-Awady, 2014). Source truncation occurs when the existing double-ended dislocation sources changes into the single-ended ones. Consequently, a new characteristic length of dislocation sources is introduced which increases the strength (Parthasarathy et al.,
Rao et al., 2007). Cui et al. (2015) recently studied the role of single arm sources in microstructural behavior of micropillars using dislocation dynamics. The weakest link theory states that reducing the sample size, the strength of the weakest slip plane present increases which increases the material strength at smaller length scales (Norfleet et al., 2008; El-Awady et al., 2009).

In recent years, new experimental techniques have been developed to measure the density of geometrically necessary dislocations (GNDs) (Kysar and Briant, 2002; Kysar et al., 2007; Zaafarani et al., 2008; Demir et al., 2009; Dahlberg et al., 2014). The experimental methods are developed based on the relation between GND density and spatial gradients of plastic slip (Nye, 1953; Kondo, 1964; Fox, 1966). The experiments have shown new phenomena in metallic samples which cannot be explained using the theories developed for bulk-size material. Demir et al. (2009, 2010) showed that the sources of size effects at small length scales are different from those of the bulk materials during nanoindentation and microbending of metallic samples. Demir et al. (2009) conducted nanoindentation of Cu single crystal and measured the GND densities at various indentation depths. It was observed that as the indentation depth decreases the hardness increases. According to the forest hardening models, the GND density should increase (Nix and Gao, 1998; Abu Al-Rub and Voyiadjis, 2004; Voyiadjis and Abu Al-Rub, 2005). However, the results showed that the GND density decreases as the indentation depth decreases (Demir et al., 2009). It implies that the hardness decreases as the GND density increases. Moreover, only GNDs were measured during the experiments and not all the dislocation content which includes both GNDs and SSDs (statistically stored dislocations). Hence, it may have affected the final conclusion. Demir et al. (2010) also investigated the size effects in microbending experiment on the Cu single crystal. Again, the results showed that the mean-field behavior of the dislocations breaks down at small length scales.

Nowadays, using very powerful supercomputers and efficient and massively parallel codes, the samples in the order of 0.1 μm can be simulated using Molecular dynamics (MD) (Voyiadjis and Yaghoobi, 2015). To study the relation between the dislocation density and hardness using molecular
dynamics, the dislocation information should be extracted from the MD outputs. Various methods of defect visualization has been developed including energy filtering, centrosymmetry parameter analysis, bond order analysis, Voronoi analysis, adaptive common neighbor analysis, and neighbor distance analysis (see Stukowski, 2012). Stukowski (2012) implemented these methods and compared the obtained results. Stukowski and his co-workers (Stukowski and Albe, 2010; Stukowski et al., 2012; Stukowski, 2012; Stukowski, 2014) developed the Crystal Analysis Tool to obtain the dislocation length and Burgers vector from atomistic simulation outputs. Hua and Hartmaier (2010) proposed a method to quantify the Burgers vectors of dislocations from MD results. They calculated densities of GNDs and SSDs using the developed methodology. Begau et al. (2012) simulated the nanoindentation of a copper single crystal indented by a spherical indenter using MD. They qualitatively studied the dislocation density variation as the indentation depth increases. However, they did not compare the results to any theoretical models or experiments. Gao et al. (2014) tried to compare the dislocation length obtained by atomistic simulation of nanoindentation with that of the theoretical models. However, the dislocation length obtained from MD simulation cannot satisfactorily capture the theoretical models for dislocation length. Recently, Gao et al. (2015) studied the plastic zone size of single crystal metals indented by spherical indenter during MD simulation. Voyiadjis and Yaghoobi (2015) studied the relation between the dislocation density and hardness during nanoindentation of metallic samples. However, none of the previous works have studied the sources of size effects in the studied cases.

In the current chapter, the size effects in nanoscale thin films are studied during indentation using large scale atomistic simulation. A Ni single crystal thin film is indented using a conical indenter with a spherical tip. First, a geometric model is derived to predict the GNDs length and density during nanoindentation with a spherical tip. The precise geometry of the indenter is incorporated in the developed model. The obtained results are then compared with the predictions available in the literature. The dislocation length calculated from the theoretical predictions is then compared to the
one obtained from MD simulation during nanoindentation. Next, the dislocation density is obtained for various plastic zone sizes using MD. The final step is to investigate the size effects sources using the microstructural information obtained from MD simulation.

Fig. 4.1. The atomic structure of the Ni thin film.

### 4.2. Simulation details and methodology

To investigate the sources of size effects during nanoindentation, a Ni thin film is simulated using the classical molecular dynamics. The sample dimensions are 120 nm, 120 nm, and 60 nm along [1 1 0], [1 1 2], and [1 1 1] directions, respectively, which consists of 79,473,309 atoms. A part of the generated sample is represented in Fig. 4.1. The indenter geometry is selected according to the nanoindentation experiment performed by Demir et al. (2009) which is a conical indenter with an angle of $\theta = 60^\circ$ and a spherical tip with the radius of $R = 10$ nm (Fig. 4.2). Along [1 1 0] and [1 1 2] directions, the boundary conditions are set periodic. At the sample bottom, the boundary conditions are free. The required force is applied at each step to all atoms to inhibit the translational movement which is described in detail by Yaghoobi and Voyiadjis (2014). The parallel MD code LAMMPS is used to perform the simulations (Plimpton, 1995). The time step of 2 fs is chosen and the integration algorithm is velocity Verlet. The sample is relaxed for 200 ps prior to the indentation process. The relaxed sample is indented with a velocity of 10 m/s along the [1 1 1] direction at 300 K. The velocity has been widely used in previous works. The NPT ensemble is used to simulate the nanoindentation. The EAM interatomic potential $E^{EAM}$ is selected to model the Ni-Ni atomic interaction which is defined as follows.
\[ E^{EAM}(r_{ij}) = \frac{1}{2} \sum_{i,j} V(r_{ij}) + \sum_i F(\rho_i), \quad \rho_i = \sum_{i \neq j} \varphi(r_{ij}) \] (4.1)

where \( V(r_{ij}) \) and \( F(\rho_i) \) represent the pair interaction and embedding potentials, respectively, and \( \varphi(r_{ij}) \) is a function of the electron density. The EAM potential developed by Mishin et al. (1999) is used to model the Ni-Ni atomic interaction (Becker et al., 2013).

Fig. 4.2. The geometry of conical indenter with a spherical tip.

Fig. 4.3. The true indentation depth \( h \) for: (a) spherical part of the indenter (b) conical part of the indenter.
The interaction between the indenter and Ni atoms is described as follows (Plimpton, 1995):

\[ E^{\text{ind}}(r) = \varepsilon (r - r_c)^2 \quad r < r_c \]  

where \( \varepsilon, r, \) and \( r_c \) are the specified force constant, distance from particle to the indenter surface, and cutoff distance, respectively. The parameters \( \varepsilon \) and \( r_c \) are chosen as 1 eV/Å² and 0.3 nm, respectively.

The triangulation method is incorporated to calculate the contact area \( A \) which is described in detail in (Yaghoobi and Voyiadjis, 2014; Voyiadjis and Yaghoobi, 2015). The true indentation depth \( (h) \) of a spherical tip, which is slightly different from the tip displacement \( (d) \), is calculated using the obtained contact area:

\[ h = R - \sqrt{R^2 - a_c^2} \]  

where \( a_c = \sqrt{A/\pi} \) and \( R \) is the radius of the tip (Fig. 4.3). In the case of the conical part of the indenter, \( h \) can be obtained as follows:

\[ h = \frac{(a_c - a_0)}{\tan(\theta/2)} + h_0 \]  

where \( h_0 \) is the indentation depth at which the transition occurs in the indenter geometry from the spherical tip to the conical shape and \( a_0 \) is the contact radius at \( h_0 \) (Fig. 4.3). In order to study the effects of temperature, rate of indentation, and selected boundary conditions, a molecular static study, i.e. zero temperature and static indentation, is presented in Appendix A in which the sample is placed on the Si substrate. The Crystal Analysis Tool is used to extract the dislocation information from MD outputs (Stukowski and Albe, 2010; Stukowski et al., 2012; Stukowski, 2012; Stukowski, 2014). The dislocations are visualized and processed using the software OVITO (Stukowski, 2010) and Paraview (Henderson, 2007).

4.3. Results and Discussions

4.3.1. Indentation response and dislocation nucleation

Fig. 4.4 presents the indentation load \( P \) versus the indentation depth \( h \). The nanoindentation response is initially elastic and follows the Hertzian theory (Yaghoobi and Voyiadjis, 2014). Initial
phases of dislocation nucleation and evolution are in line with the previous works (see Yaghoobi and Voyiadjis, 2014). The first load drop in the $P - h$ curve is related to the first dislocation nucleation beneath the indenter at $h \approx 0.64$ nm. Next, dislocation embryos are grown on $\{111\}$ planes. After this stage and by increasing the indentation depth, prismatic loops are formed and glide along the three directions of $[0 \bar{1} 1], [\bar{1} 0], \text{and} [\bar{1} \ 0 \bar{1}]$, as described in Voyiadjis and Yaghoobi (2015). Fig. 4.5 presents the visualization of dislocations and indentation surface during nanoindentation. Most of the dislocations are Shockley partial dislocations and their Burgers vectors are $1/6 \langle 1 \ 1 \ 2 \rangle$. The Shockley partial dislocations are evolved on $\{111\}$ planes which were previously reported (Yaghoobi and Voyiadjis, 2014; Voyiadjis and Yaghoobi, 2015). There are also a few perfect dislocations, Hirth, stair-rod, and Frank partial dislocations. The Burgers vectors are $1/2 \langle 1 \ 1 \ 0 \rangle$, $1/3 \langle 0 \ 0 \ 1 \rangle$, $1/6 \langle 0 \ 1 \ 1 \rangle$, and $1/3 \langle 1 \ 1 \ 1 \rangle$ for perfect dislocations, Hirth, stair-rod, and Frank partial dislocations, respectively. In Fig. 4.5, the color of perfect dislocations and Hirth, Shockley, Frank, and stair-rod partial dislocations are red, yellow, green, pink, and blue, respectively. Fig. 4.6 presents the variation of the mean contact pressure ($p_m = P/A$), which is equivalent to the hardness $H$ in the plastic region, as a function of indentation depth $h$. In the elastic region, Fig. 4.6 shows that $p_m$ increases as the indentation depth increases. However, after the initiation of plasticity, Fig. 4.6 shows that the mean contact pressure, i.e. hardness, decreases as the indentation depth increases.

Fig. 4.4. Variation of indentation load $P$ as a function of indentation depth $h$. 

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Fig. 4.5. The visualization of dislocations structure during nanoindentation at different indentation depths of (a) $h = 0.9 \text{ nm}$ (b) $h = 4 \text{ nm}$ (c) $h = 11 \text{ nm}$. 
4.3.2. Dislocation length and density

The dislocation length obtained from the MD simulation is compared with the theoretical predictions of GNDs length. In the case of a spherical tip, Swadener et al. (2002) approximated the indenter geometry with a parabola and obtained the GNDs length as follows:

\[
\lambda_{sp} \approx \frac{2\pi}{3} \frac{a_c^3}{bR} \quad (4.5)
\]

where \(b\) is the magnitude of the Burgers vector. However, Eq. (4.4.5) is reliable only for small indentation depths (Swadener et al., 2002). Hence, the exact geometry of the indenter should be incorporated to calculate the dislocation length for deeper indentations.

The total GNDs length can be obtained as follows (Swadener et al., 2002; Pugno, 2007):

\[
\lambda_{sp} = \int_0^{a_c} \frac{2\pi r}{b} \left( \frac{dh}{dr} \right) dr = \frac{2\pi}{b} \int_0^{a_c} \left( \frac{r^2}{\sqrt{R^2 - r^2}} \right) dr = \frac{2\pi}{b} \left[ \frac{R^2}{2} \sin^{-1} \left( \frac{a_c}{R} \right) - \frac{1}{2} \left( a_c \sqrt{R^2 - a_c^2} \right) \right] \quad (4.6)
\]

In the case of the conical part of the indenter, the GNDs length can be written as follows (Nix and Gao, 1998):

\[
\lambda_{co} = \lambda_{sp} \big|_{h=h_0} + \int_{a_0}^{a_c} \frac{2\pi r}{b \tan \left( \frac{\theta}{2} \right)} dr = \lambda_{sp} \big|_{h=h_0} + \frac{\pi (a_c^2 - a_0^2)}{b \tan \left( \frac{\theta}{2} \right)} \quad (4.7)
\]

Fig. 4.7 shows the variation of the theoretical GNDs length obtained from the approximate (Swadener et al., 2002) and exact (see Eq. (4.6)) geometries of the indenter versus the normalized...
contact radius $a_c/R$. It shows that the approximation is satisfactory only for the shallow indentations. For example, at $a_c/R \approx 0.2$, the dislocation length obtained from Eq. (4.5) is 12% smaller than the one calculated from Eq. (4.6). Generally, the approximate geometry gives smaller dislocation length compared to the exact one during nanoindentation (Fig. 4.7). Fig. 4.8 shows the variation of dislocation length obtained from MD simulation and approximate and exact theoretical predictions of Eqs. (4.5-4.7) as a function of indentation depth $h$. The results show that the theoretical dislocation length is a lower bound for that of the MD simulation. It is due to the fact that Eqs. (4.5-4.7) only predict the GNDs and not SSDs.

In the current chapter, the plastic zone is defined as a hemisphere. The radius of hemisphere is $R_{pz} = f a_c$ in which $f$ is a constant. Durst et al. (2005) stated that $f$ is smaller than 3.5. They selected $f = 1.9$ in their simulations. In the case of the theoretical predictions, the plastic zone with the radius of $1.9 a_c$ is selected. The dislocation density can be stated as follows:

$$\rho = \frac{\lambda}{V} \quad (4.8)$$

where $V$ is the volume of plastic zone and $\lambda$ is the dislocation length. Fig. 4.9 shows the variation of the theoretical GNDs density obtained from the approximate (Swadener et al., 2002) and exact (see Eq. (4.6)) geometries of the indenter versus the normalized contact radius $a_c/R$. Swadener et al. (2002) neglected the indenter volume in their calculations. Similar assumption is incorporated here in the case of the theoretical predictions to make the results comparable to those of Swadener et al. (2002). As Swadener et al. (2002) explained, using Eq. (4.5) to calculate the dislocation length leads to a constant GNDs density during nanoindentation with a spherical indenter. However, using the precise spherical geometry, Fig. 4.9 shows that the GNDs density is not constant anymore and increases as the indentation depth increases. One should note that the observed trend does not change by selecting different values of $f$ because $f$ is a constant. Moreover, it is usually assumed that the SSDs density does not change. Hence, the variation of total dislocation density versus the indentation depth is similar to Fig. 4.9.
Fig. 4.7. Theoretical GNDs length obtained from the approximate (Swadener et al., 2002) and exact (see Eq. (4.6)) geometries of the indenter versus the normalized contact radius $a_c/R$.

Fig. 4.8. Comparison between the dislocation lengths obtained from theoretical models and MD simulation during nanoindentation.

In the case of MD simulation, the dislocation density is measured using the dislocation length located in the plastic zone divided by the volume of the plastic zone. Here, five different values of $f = 1.5, 2.0, 2.5, 3.0, \text{ and } 3.5$ are chosen to investigate the effect of plastic zone size on the dislocation density during nanoindentation. In the case of MD simulation, the plastic zone volume can be precisely obtained as follows:

$$V = (2/3)\pi (f a_c)^3 - V_{\text{indenter}}$$  \hfill (4.9)
where $V_{\text{indenter}}$ is the part of the plastic zone occupied by the indenter. The dislocation density $\rho$ versus the indentation depth $h$ is plotted in Fig 7. The results indicate that as the indentation depth increases, the dislocation density also increases for different values of $f$. Comparing Figs. 4.9 and 4.10, the results show that the trend of dislocation density variation obtained from MD simulation versus the indentation depth follows the one calculated from the theoretical prediction of Eqs. (4.6) and (4.7) in which the exact geometry of the indenter is incorporated.

![Graph showing dislocation density vs normalized contact radius](image)

Fig. 4.9. Theoretical GNDs density obtained from the approximate (Swadener et al., 2002) and exact (see Eq. (4.6)) geometries of the indenter versus the normalized contact radius $a_c/R$.

4.3.3. Sources of size effects

In a bulk metallic sample, the strength is controlled by the so-called forest hardening mechanism in which the strength increases as dislocation density increases. The Taylor hardening model is one of the widely used models to capture the forest hardening by linking the shear strength to the dislocation density as follows:

$$\tau = \alpha \mu b \sqrt{\rho}$$  \hspace{1cm} (4.10)

where $\mu$ is the shear modulus, and $\alpha$ is a constant. The hardness $H$ is obtained by incorporating the von Mises flow rule as described below:
\[ \sigma = \sqrt{3} \tau \]
\[ H = 3\sigma = 3\sqrt{3}a\mu b\sqrt{\rho} \quad (4.11) \]

where Tabor factor is selected as 3 and \( \sigma \) is the flow stress. There are few modifications for the Taylor hardening model such as using different Burgers vectors for GNDs and SSDs or modifications in exponents, which can be expressed as follows:

\[ H = 3\sqrt{3}a_S \mu b_S \sqrt{\rho} \]
\[ \rho = \left[ \rho_G^{\beta/2} + (a_G^2 b_G^2 \rho_G / a_S^2 b_S^2)^{\beta/2} \right]^{2/\beta} \quad (4.12) \]

where the indices \( G \) and \( S \) designate GNDs and SSDs parameters, respectively. However, all equations derived from the Taylor hardening model have the same trend in which the hardness increases as the dislocation density increases. Based on the Taylor hardening model, since the dislocation density increases as the indentation depth increases (Fig. 4.10), the hardness should also increase. However, Fig. 4.6 shows that as the indentation depth increases, the hardness decreases. In other words, the results show that the forest hardening model cannot capture the size effect in the case of the simulated sample.

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**Fig. 4.10.** Dislocation density obtained from MD simulation for different values of \( f \) during nanoindentation.
Fig. 4.11. Dislocation nucleation and evolution at small tip displacements: (a) initial homogeneous dislocation nucleation beneath the indenter which has a Burgers vector of $1/6 \{\bar{2} 1 \bar{1}\}$ (Shockley partial dislocation); (b-j) cross slip of screw components which produces new pinning points; (k-l) first loop is released by pinching off action.
Similar to the compression and tension experiments on micropillars of small length scales, the observed results show that the forest hardening mechanism does not govern size effects in the nanoscale samples during nanoindentation. To unravel the sources of size effects, the initial phases of indentation should be studied. First, the nanoindentation is elastic which follows the Hertzian theory. The first dislocation homogeneously nucleates beneath the indenter which has the Burgers vector of $1/6 [\bar{2} 1 \bar{1}]$ (Fig. 4.11 (a)). The critical resolved shear stress of nucleation is close to the theoretical strength of perfect crystals. Increasing the indentation depth, the rest of dislocation nucleation process is heterogeneous. Fig. 4.11 illustrates the initial phases of dislocation nucleation. It shows that the cross-slip is responsible for dislocation multiplication in which the number of pinning points are increased by cross-slip mechanism. It has been previously shown that the cross-slip is an important dislocation multiplication mechanism in FCC metallic samples of confined volumes (Zhou et al., 2011; Cui et al., 2013). The required dislocation length is provided by the elongation of the dislocations which are pinned at their ends. Dislocation loops are then nucleated by cross-slip and pinching off of screw dislocations which glide along the three directions of $[0 \bar{1} \bar{1}]$, $[\bar{1} \bar{1} 0]$, and $[\bar{1} 0 \bar{1}]$. The procedure is depicted in Fig. 4.11 (i)-(l). However, the resulting mobile dislocation density is insufficient and the applied stress should be increased to sustain the plastic deformation. Hence, source exhaustion hardening is the governing mechanism of size effects. By increasing the indentation depth, the dislocation length and density increases as well, which provides more dislocation sources. Also, the source lengths increase which reduces the critical resolved shear stress. Consequently, the applied stress required to sustain flow during nanoindentation decreases, i.e. hardness decreases as indentation depth increases. At higher indentation depths, the dislocation density and length are large enough in a way that the forest hardening is also activated (Fig. 4.12). However, since the dislocation density tends to a constant value at high indentation depths, the hardness becomes nearly constant.
In the current chapter, the temperature of 300 K and velocity of 10 m/s are selected. Moreover, at the sample bottom, the boundary conditions are free. In the final part of this chapter, the effects of temperature and loading rate are investigated using the molecular static simulation of similar indentation process using slightly smaller sample, due to the computational limitations. In the case of molecular static, there is no temperature, and indentation is performed static. Furthermore, the Si substrate is used here to investigate the effects of the chosen boundary conditions. The dimensions of the Ni thin film are 100 nm, 100 nm, and 50 nm along [1 1 0], [1 1 2], and [1 1 1] directions, respectively. The total thickness of Si substrate is 6.5 nm. The EAM potential presented by Mishin et al. (1999) is used to describe the Ni-Ni interaction. In order to model the Si-Si interaction, a three-body Tersoff potential (1988) is selected. The Si parameters for Tersoff potential are similar to those incorporated in Yaghoobi and Voyiadjis (2014). The Lennard–Jones (LJ) potential is incorporated to simulate the Ni-Si interaction as follows:

\[
E_{\text{LJ}}(r_{ij}) = 4\varepsilon \left[ \left( \frac{\sigma}{r_{ij}} \right)^{12} - \left( \frac{\sigma}{r_{ij}} \right)^{6} \right]
\]

where \( \sigma \) and \( \varepsilon \) are the collision diameter and the depth of the potential well, respectively. The LJ parameters are obtained as follows:

\[
\varepsilon_{\text{Ni-Si}} = \sqrt{\varepsilon_{\text{Ni-Ni}} \varepsilon_{\text{Si-Si}}}
\]

\[
\sigma_{\text{Ni-Si}} = \frac{\sigma_{\text{Ni}} + \sigma_{\text{Si}}}{2}
\]

where \( \varepsilon_{\text{Ni}} \) and \( \sigma_{\text{Ni}} \) are the Ni-Ni and \( \varepsilon_{\text{Si}} \) and \( \sigma_{\text{Si}} \) are the Si-Si LJ parameters. The LJ parameters are similar to those incorporated in Yaghoobi and Voyiadjis (2014). The cutoff distance of the LJ potential is set as 2.5\( \sigma \). First, the sample is relaxed using molecular static. The indenter is lowered by 0.2 Å at each step and the minimum energy configuration is obtained using the conjugate gradient method (Plimpton, 1995).

The variation of indentation load \( P \) as a function of indentation depth \( h \) is presented in Fig. 4.13. Again, the nanoindentation response is initially elastic which follows the Hertzian theory. The
variation of mean contact pressure $p_m$ as a function of indentation depth $h$ is shown in Fig. 4.14. Similar to the molecular dynamics simulation, after the initial elastic part, the mean contact pressure, i.e. hardness, decreases as the indentation depth increases. The dislocation density is measured using the dislocation length located in the plastic zone divided by the volume of the plastic zone. The plastic zone is a hemisphere with a radius of $R_{pz} = f a_c$. Again, five different values of $f = 1.5, 2.0, 2.5, 3.0, \text{and} 3.5$ are chosen. Fig. 4.15 shows the dislocation density $\rho$ versus the indentation depth $h$. The results verified the observed results in molecular dynamics simulation and the dislocation density increases as the indentation depth increases. The results show that the observed sources of size effects are not artificially produced due to the temperature, indentation rate, or boundary conditions. The dislocation nucleation and evolution is illustrated in Fig. 4.16. It shows that the cross-slip is responsible for dislocation multiplication which is similar to the results observed in MD simulation. Again, dislocation loops are nucleated by cross-slipping and pinching off of screw dislocations which glide along three directions of $[0 \bar{1} \bar{1}], [\bar{1} \bar{1} 0], \text{and} [\bar{1} 0 \bar{1}]$.

4.4. Conclusions

The present chapter investigates the size effects sources at small length scales during nanoindentation using large scale atomistic simulation. A Ni single crystal thin film is indented using a conical nanoindenter with a spherical tip. A geometric model is developed to predict the dislocation length and density during nanoindentation for the spherical tip. The precise tip geometry is incorporated in the model. The model is then compared to the previously developed one in which the spherical tip geometry is approximated using a parabola. The results show that the approximate model underestimates the dislocation length and gives reasonable results only at shallow indentation depths.

Next, the dislocation length is measured using MD during nanoindentation. The results show that the developed model and MD results have better correlation compared to the approximate model. Also, the results show that the theoretical dislocation length is a lower bound for that of the MD simulation.
The fact that the theoretical prediction includes only GNDs and not SSDs justifies the obtained results.

Using various sizes of plastic zone, the dislocation density is calculated during nanoindentation.

Fig. 4.12. Dislocation nucleation and evolution at large tip displacements of (a) $d = 4.8$ nm (b) $d = 8.05$ nm (c) $d = 13.05$ nm (d) $d = 13.3$ nm.
Fig. 4.13. Variation of indentation load $P$ as a function of indentation depth $h$.

Fig. 4.14. Variation of mean contact pressure $p_m$ as a function of indentation depth $h$. 
Fig. 4.15. Dislocation density obtained from molecular static simulation for different values of $f$ during nanoindentation.

The approximate model predicts that the dislocation density is constant. The model with precise geometry and MD simulation, however, show that the dislocation density increases as the indentation depth increases. The results show that the derived theoretical prediction is able to capture the variations of dislocation length and density versus the indentation depth even for nanoscale samples. It is due to the fact that the theoretical prediction is developed based on the geometric relations.

To investigate the sources of size effects, the variation of hardness obtained from MD simulation should be studied. Since the dislocation density increases as the indentation depth increases, the hardness should also increase if the forest hardening is the governing mechanism. However, at low indentation depths, the results show that the hardness decreases as the dislocation density increases which implies that the dislocation interactions with each other is not the source of size effects.
On the other hand, at low indentation depths, it is observed that the source exhaustion hardening is the controlling mechanism of size effects. In other words, the available mobile
dislocation density is insufficient and the applied stress should be increased to sustain the plastic deformation. First, a dislocation is homogenously nucleated beneath the indenter at the stress close to the theoretical strength. Then, the dislocations are multiplied using the cross-slip mechanism. The prismatic loops are released by cross-slip and pinching off of screw dislocations. Increasing the indentation depth, the dislocation density and length increase which provides more dislocation sources and increases the source lengths. Consequently, the critical resolved shear stress decreases. At higher indentation depths, the forest hardening mechanism also becomes important. However, since the dislocation density tends to a constant value, the hardness becomes constant.
CHAPTER 5
EFFECTS OF GRAIN BOUNDARY ON THE SOURCES OF SIZE EFFECTS

5.1. Introduction

The mechanical properties of crystalline metals are usually governed by grain size and grain boundary (GB) properties (Meyers et al., 2006; Koch et al., 2007; Zhu et al., 2008). The GB effects on the strength depend on several factors including the grain size, GBs geometry, mechanical and crystallographic structure of metal, strain rate, and temperature (Meyers et al., 2006; Koch et al., 2007; Zhu et al., 2008). These factors define the deformation mechanisms in crystalline metals. Several numerical and experimental studies have been conducted to unravel the role of GBs in crystalline metals. In the case of large grains, i.e. grain sizes of more than 1 μm, the strength increases as the grain size decreases which is commonly described by the Hall-Petch relation. The enhancement in strength is justified using the dislocation pile-up mechanism. By decreasing the grain size, the pile-up model and consequently the Hall-Petch relationship break down at some grain size which is of the order of nanometers (Nieh and Wadsworth, 1991). Other mechanisms such as GB sliding, GB rotation, and GB dislocation creation and annihilation have been proposed to describe the grain size dependency of strength in nanocrystalline metals (Meyers et al., 2006; Koch et al., 2007; Zhu et al., 2008).

Molecular dynamics (MD) simulation is a powerful tool to model the effects of grain size and GB properties on the strength of nanocrystalline metals from the atomistic point of view. Van Swygenhoven et al. (1999,2001) incorporated the atomistic simulation and showed that below a critical grain size, the GB sliding is a dominant mechanism using molecular dynamics. In the cases of grains larger than 10-12 nm, they observed some intra-grain deformation mechanism such as partial dislocations. Yamakov and coworkers (Yamakov et al., 2002; Yamakov et al., 2003a; Yamakov et al., 2003b; Wolf wt al., 2005) also conducted several atomistic simulation to study the effect of grain size on the deformation mechanisms of nanocrystalline fcc metals. It was observed that as the grain size...
decreases, the deformation process changes from a dislocation-based to a grain-boundary-based mechanism. Derlet et al. (2003) summarized the limitation of MD in the case of nanocrystalline metals simulation. They stated that the atomic deformation mechanisms can be successfully extracted from the MD simulation. However, when it comes to the extrapolation of the obtained MD results to the experimental observations, one should consider the temperature, size, and rate at which the simulation is conducted.

The interaction of the dislocations with GBs has been investigated using atomistic simulation to understand the underlying deformation process. De Koning et al. (2003) performed 2D and 3D MD simulations to study the various mechanisms of dislocation absorption, transmission, and reflection by incorporating both symmetric and asymmetric Σ11 tilt GBs. Hasnaoui et al. (2004) simulated the nanoindentation experiment using MD simulation to study the interaction between dislocations and GBs. It was observed that the GBs can act as a sink for dislocations, reflect or emit dislocations. Jang and Farkas (2007) incorporated MD simulation and studied a bi-crystal nickel thin film with \( \sum 5 (210) [001] \) GB during the nanoindentation. It was shown that the GB induced some resistance to the indentation due to the stacking fault expansion. Kulkarni et al. (2009) compared the response of coherent twin boundary (CTB) with that of \( \sum 9 (221) \) tilt GB during nanoindentation using atomistic simulation. They stated that unlike the \( \sum 9 (221) \) GB, the CTB does not considerably reduce the strength of the sample. However, unlike Jang and Farkas (2007), they did not observe noticeable enhancement in strength. Tsuru et al. (2010) studied the effects of coincidence site lattice (CSL) grain-boundaries on the incipient plasticity using MD. They changed the distance of indenter with respect to the GB and monitored the incipient plasticity. They concluded that the GBs with lower energies have higher mean pressures at the onset of plasticity. Stukowski et al. (2010) compared the uniaxial response of nanotwinned Cu and Pd nanocrystalline samples with those without twin boundaries using large scale atomistic simulation. They showed that the effects of twin boundaries depend on the unstable stacking fault and twin boundary migration energies, and it can increase or
decrease the strength of the material. Sangid et al. (2011) measured the energy barriers of CSL grain boundaries of slip–GB interactions using MD. It was observed that the energy barrier has an inverse relationship with GB energy in a way that the GB with lower energy has the higher energy barrier. Zhang et al. (2014) incorporated the atomistic simulation to investigate the response of symmetric and asymmetric $\Sigma 5$ GBs during uniaxial tension experiment. They studied the effect of inclination angles on the mechanical response of copper bi-crystal. Sainath and Choudhary (2015) simulated the Cu nanopillars with and without twin boundaries under uniaxial tension using MD. It was shown that the GB changes the deformation mechanism and increase the strength of the simulated nanopillar.

The interaction of dislocations with each other and with GBs governs the size effects in bulk metals which is termed as the forest hardening (Voyiadjis and Abu Al-Rub, 2005; Greer, 2013). Decreasing the sample size to the order of nanometers, however, the forest hardening model is not the governing mechanism. The size effects in nanoscale metallic samples are described using three models of source exhaustion hardening, weakest link theory, and source truncation (Greer, 2013; Kraft et al., 2010; Greer and De Hosson, 2011). Dislocation starvation, mechanical annealing, and source shut down reduce the number of sources in nanoscale samples in a way that the reduced mobile dislocation density is insufficient to handle the imposed deformation. Consequently, the applied stress should be increased to sustain the plastic deformation. The induced hardening due to the lack of sources is termed exhaustion hardening. In the case of metallic sample of confined volumes, since the double-ended dislocation sources are close to the free surfaces, they transform into the single-ended sources. Consequently, the strength is increased due to the reduction in characteristic length of dislocation sources which is commonly termed as source truncation. The last mechanism is the weakest link theory in which the strength of the weakest slip plane increases by reducing the sample size which enhances the strength. Tucker et al. (2013) investigated the effects of GBs on the size effects of nanowires under uniaxial compression using MD. It was shown that both GB and free surfaces can be the favorable dislocation nucleation sites. Recently, Yaghoobi and
Voyiadjis (2016a) studied the sources of size effects in nanosize single crystal Ni thin films during nanoindentation using large scale atomistic simulation. They showed that the dislocation nucleation and source exhaustion are mainly responsible for size effects during the nanoindentation of Ni samples of confined volumes at lower indentation depths.

In the current chapter, the effects of GBs on the sources of size effects are investigated. The nanoindentation response of single and bi-crystal Ni thin films are investigated by incorporating large scale atomistic simulation. Here, samples with two different sizes and various symmetric and asymmetric tilt GBs are studied to capture the role of GB in size effects as the grain size changes. Furthermore, the sources of size effects are analyzed in each sample using the microstructural information obtained from the simulations. In order to study the size effects controlling mechanisms, the total dislocation length of each sample is studied during the nanoindentation. The obtained results are then compared to the variation of hardness versus the indentation depth to unravel the contribution of GBs to the sources of size effects as the sample length scale changes. The dislocation visualization is also incorporated to study the dislocation nucleation and evolution patterns for bi-crystal and single crystal samples of different sizes.

5.2. Simulation details and methodology

To investigate the effects of grain size on the sources of size effects, two different sizes of Ni thin films with the dimensions of 24 nm × 24 nm × 12 nm (S1) and 120 nm × 120 nm × 60 nm (S2) are generated and simulated using the classical molecular dynamics. The total number of atoms in the samples are around 650,000 for S1 and 79,000,000 for S2. The periodic boundary conditions are applied to the surrounding surfaces. The free boundary conditions are selected at the bottom of the sample. The selected boundary conditions ensure that the dislocations do not bounce back from the sample bottom. To prevent the whole domain from translational movement, at each step, the total force is divided by the number of atoms and then applied in the opposite direction to all atoms. Voyiadjis and his coworkers (Yaghoobi and Voyiadjis, 2014; Voyiadjis and Yaghoobi, 2015; Yaghoobi
and Voyiadjis, 2016a) has previously shown that the chosen boundary conditions type can successfully capture the nanoindentation response and microstructural information such as dislocation structure and length. The velocity Verlet algorithm with a time step of 2 fs is selected. The parallel MD code LAMMPS is used. Seven different types of GB including four symmetric tilt boundaries of $\Sigma 3 \ (1 \ 1 \ 1) \ [1 \ 1 \ 0] \ (\theta = 109.5^\circ)$, $\Sigma 3 \ (1 \ 1 \ 2) \ [1 \ 1 \ 0] \ (\theta = 70.5^\circ)$, and $\Sigma 11 \ (1 \ 1 \ 3) \ [1 \ 1 \ 0] \ (\theta = 50.5^\circ)$, $\Sigma 11 \ (3 \ 3 \ 2) \ [1 \ 1 \ 0] \ (\theta = 129.5^\circ)$, and three asymmetric tilt boundaries of $\Sigma 11 \ (2 \ 2 \ 5)/(4 \ 4 \ 1) \ (\varphi = 54.74^\circ)$, $\Sigma 3 \ (1 \ 1 \ 2)/(\bar{5} \ 5 \ 2) \ (\varphi = 19.47^\circ)$, and $\Sigma 3 \ (1 \ 1 \ 4)/(1 \ 1 \ 0) \ (\varphi = 35.26^\circ)$ are selected to study the effects of GB properties on the size effects, where $\theta$ and $\varphi$ are the interface misorientation and inclination angles, respectively. The GBs are located at a third of the thickness from the top surface. For each bi-crystal sample, one single crystal sample is also generated which has the atomic structure similar to that of the upper grain. Two spherical indenters with the radius of $R_1 = 10$ nm and $R_2 = 15$ nm are selected to perform indentation on S1 and S2 samples, respectively. The CSL grain boundaries are created by a symmetric tilt rotation $\theta$ of opposing lattice regions around a misorientation axis $[1 \ 1 \ 0]$. Asymmetric tilt GBs are obtained from symmetric tilt boundaries of the same misorientation angle by incorporating the inclination angle $\varphi$, which is the deviation of the GB plane from the selected symmetrical position. The final configuration is obtained using molecular statics. Energy minimization is attained using the conjugate gradient algorithm. In order to reach the minimum energy configuration of GBs, several in-plane rigid body translations are conducted in which the top and bottom grains are rigidly translated with respect to each other. Next, the samples are relaxed for 100 ps with the temperature increasing from 1 K to 300 K. Finally, they are relaxed for 100 ps at 300 K. The GBs equilibrium structures are presented in Fig. 5.1 using the Centrosymmetry parameter. The software OVITO (Stukowski, 2010) is used to visualize the equilibrium structures. In Fig. 5.1, the vertical direction of the upper grain is normal to the upper free surface of each sample.
Finally, the relaxed samples are indented along the normal to the upper free surface with the velocity of 10 m/s, which has been widely used in previous simulations, at 300 K. The simulation is performed using NPT ensemble. The nickel embedded-atom method (EAM) potential developed by Mishin et al. (1999) is incorporated to simulate the atomic interaction of Ni-Ni. A repulsive potential is selected here to describe the interaction between the indenter and Ni atoms, which is described as follows:

$$E_{\text{ind}}(r) = \varepsilon (r - r_c)^2 \quad r < r_c \quad (5.1)$$

where $\varepsilon$ is the specified force constant which is chosen as 1 eV/Å$^2$, $r$ is the distance from particle to the indenter surface, and $r_c$ is the cutoff distance which is selected as 0.3 nm. To calculate the contact area $A$, first, atoms located in direct contact with the indenter are identified. Next, a triangular 2D-mesh is created from the projections of those atoms onto the indentation surface. The contact area is the summation of all triangle areas. The mean contact pressure $p_m$ can be obtained by dividing the indentation load $P$ by contact area $A$. In the case of the spherical tip, true indentation depth $h$, which is different from the displacement of the indenter tip $d$, can be obtained from geometrical relations.
Here, the indentation depth of a spherical tip is calculated by incorporating the contact area as follows:

\[ h = R - \sqrt{R^2 - a_c^2} \]  

(5.2)

where \( a_c = \sqrt{A/\pi} \) is the contact radius and \( R \) is the spherical tip radius.

The Crystal Analysis Tool developed by Stukowski and his co-workers (Stukowski and Albe, 2010; Stukowski et al., 2012; Stukowski, 2012; Stukowski, 2014) is incorporated to visualize the dislocations and provide additional information such as dislocation length and Burgers vector. One can obtain all the required dislocation properties from the MD simulation output using the Crystal Analysis Tool which, to the best of authors’ knowledge, is the only open source post processing tool providing such information. First, atomic arrangements and structures are identified. Next, a pattern matching algorithm groups atoms into so-called clusters. A Delaunay mesh is generated that connects all atoms and the corresponding elastic deformation gradient tensor is computed for the constructed mesh. If a dislocation intersects a tessellation element, the elastic deformation gradient is multi-valued. In the next step, trial circuits are built on the interface mesh to define the dislocation lines. Finally, the code provides a one-dimensional line representation of the dislocation segments. The software OVITO (Stukowski, 2010) and Paraview (Henderson, 2007) are used to visualize the dislocations and process the microstructural information such as dislocation length and density. As described by Stukowski et al. (2010), GBs may be detected as small angle GBs. However, the immobile GB dislocations should be excluded from the total length of lattice dislocations. Accordingly, the dislocations located in a 1 nm wide region on each side of the GB planes are not considered.

5.3. Results and Discussions

Fig. 5.2 presents the variation of the mean contact pressure \( (p_m = P/A) \), which is equivalent to the hardness \( H \) in the plastic region, as a function of indentation depth \( h \) for S1 samples. In the cases of S1 samples, the results show that the GB mostly reduces the strength. However, in the case
of $\Sigma 3 \{1 \bar{1} 1\} [1 \bar{1} 0]$, i.e. coherent twin boundary (CTB), the GB slightly increases the strength at some ranges of indentation depth. Furthermore, the CTB has the least reduction in the strength compared to those of the other GBs. Same results has been reported by Kulkarni et al. (2009) during nanoindentation using MD simulation. In order to unravel the governing mechanisms of strength reduction in S1 samples, the variations of dislocation length $\lambda$ and mean contact pressure $p_m$ versus the indentation depth $h$ should be studied. As an example, Fig. 5.3 illustrates the variation of $p_m$ and $\lambda$ versus the $h$ in the case of $\Sigma 3 \{1 \bar{1} 1\} [1 \bar{1} 0]$ GB. The size effects can be studied in five different regions of indentation depths:

- **Region I**: Here, the only defect existing in bi-crystal sample is the $\Sigma 3 \{1 \bar{1} 1\} [1 \bar{1} 0]$ GB. In the case of single crystal, the sample is defect-free. The response in this region is elastic for both bi-crystal and single crystal samples. There is no difference between the single crystal and bi-crystal samples responses in this region.

- **Region II**: In the case of bi-crystal sample, the initial dislocation nucleation occurred beneath the indenter at lower indentation depth compared to the single crystal sample, which was comprehensively studied by Tsuru et al. (2010). The size effects in the bi-crystal sample is initially controlled by dislocation nucleation and source exhaustion. Here, the resulting mobile dislocation density is increased by increasing the total dislocation length. Consequently, the required applied stress to sustain the plastic deformation decreases. In other words, the dislocations created in the bicrystal sample reduce the strength. In the case of single crystal, the sample is still defect-free and the response remains elastic.

- **Region III**: In the case of single crystal sample, the first dislocation is nucleated beneath the indenter which induces a large drop in hardness. Again, dislocation nucleation and source exhaustion mechanisms initially govern the size effects. In the case of the bi-crystal sample, the dislocation length remains nearly constant. According to the source exhaustion mechanism, the applied stress should increase to sustain the plastic flow which increases the
strength. The dislocations are stopped by the GB from moving downward. However, it does not lead to any additional strength since the number of dislocations blocked by the GB is not noticeable. The fact that the dislocation blockage mechanism is available in the next regions but does not lead to any significant size effects supports this argument.

- **Region IV:** According to the source exhaustion mechanism, increasing the mobile dislocation density reduces the strength which justifies the strength reduction in both single crystal and bi-crystal samples. In Regions III and IV, the dominancy of source exhaustion mechanism decreases by increasing the total dislocation length in the case of single crystal sample. Consequently, the slope of hardness reduction decreases as the total dislocation length increases which is obvious in Region IV. The same trend can be observed for the bi-crystal sample in Regions IV and V. The GB still blocks the dislocations moving downward. Eventually, the dislocations start dissociating into the next grain.

- **Region V:** Increasing the indentation depth, the dislocation interaction with each other becomes important which activates the forest hardening mechanism. Also, the available dislocation length is enough to sustain the applied plastic flow. Consequently, the source exhaustion mechanism is not active anymore. As described by Swadener et al. (2002) and Yaghoobi and Voyiadjis (2016a), in this region, the hardness should become nearly constant. The GB still blocks some part of dislocations. However, it does not influence the size effects, and single crystal and bi-crystal samples reach the same hardness.

Fig. 5.4 presents the dislocation structure in different regions for both single crystal and bi-crystal samples in the case of \( \Sigma 3 \ (1 \ 1 \ 1) [1 \ 1 \ 0] \) GB. Fig. 5.4 (a) shows that the single crystal sample in Region II is defect-free. The dislocation structure of the bi-crystal sample in Region II \( (h \approx 0.6 \text{ nm}) \) is depicted in Fig. 5.4 (b) in which the dislocations are nucleated beneath the indenter. Fig. 5.4 (b) shows that the dislocation multiplication mechanism is cross-slip which increases the number of pinning points. The dislocations which are pinned at their ends are elongated and provide the
required mobile dislocation to sustain the plastic flow. Fig. 5.4 (c) illustrates the cross-slip and dislocation elongation in the case of single crystal sample in Region III \( h \approx 0.9 \text{ nm} \). The dislocations blockage by GB are depicted in Fig. 5.4 (d). Figs. 5.4 (e) and (f) demonstrates the dislocation structure of single crystal and bi-crystal samples, respectively, in Region IV. First dislocation which is emitted into the next grain is a Shockley partial dislocation with the Burgers vector of \( \frac{1}{6} \overline{1} \overline{2} \overline{1} \) which is shown in Fig. 5.4 (f). Figs 4 (g) and (h) show the dislocation network beneath the indenter in single crystal and bi-crystal samples, respectively, in which there is enough dislocation content to sustain the applied plastic flow, and the dislocation interaction with each other becomes important (Region V).
Fig. 5.2. Variation of mean contact pressure $p_m$ as a function of indentation depth $h$ for S1 single crystal and their related bi-crystal samples with the grain boundaries of (a) $\Sigma 3$ (1 1 1) (b) $\Sigma 11$ (1 1 3) (c) $\Sigma 3$ (1 1 2) (d) $\Sigma 11$ (3 3 2) (e) $\Sigma 11$ (2 2 5)/(4 4 1) (f) $\Sigma 3$ (1 1 2)/(5 5 2) (g) $\Sigma 3$ (3 1 4)/(1 1 0).

Fig. 5.3. Variation of mean contact pressure $p_m$ and dislocation length $\lambda$ as a function of indentation depth $h$ for S1 bicrystal sample with $\Sigma 3$ (1 1 1) GB and its related single crystal sample.
(a) single crystal sample, $h \approx 0.6$ nm

(b) bi-crystal sample, $h \approx 0.6$ nm

(c) single crystal sample, $h \approx 0.9$ nm

(d) bi-crystal sample, $h \approx 0.9$ nm

(e) single crystal sample, $h \approx 1.1$ nm

(f) bi-crystal sample, $h \approx 1.1$ nm
The variation of $p_m$ and $\lambda$ versus the indentation depth for all bi-crystal and their related single crystal samples, except the sample with $\Sigma 3 (1 1 1) [1 \bar{1} 0]$ GB, are presented in Fig. 5.5. The results show that, in contrast to the $\Sigma 3 (1 1 1) [1 \bar{1} 0]$ bicrystal and related single crystal samples, the sudden drop in strength does not simultaneously occur with the nucleation of the first dislocation. In the case of single crystal samples, Fig. 5.5 shows that the large drop in strength occurs when a sudden jump happens in the total dislocation length. In the case of bi-crystal samples, since the dislocations interact with the GB, the nature of sudden strength reduction becomes complicated. Moreover, Fig. 5.5 shows that the drop in hardness occurs at lower indentation depths in the cases of bi-crystal samples compared to that observed in the single crystal samples. At the moment of sudden drop in hardness of bi-crystal samples, they have larger total dislocation lengths compared to those of the single crystal samples which is reasonable based on the source exhaustion mechanism. Also, the grain boundary itself can be a source of dislocation nucleation in the initial phases of indentation. Although all GBs become the source of defects, it happens at different stages of indentation which leads to the different effects. As an example, Fig. 5.6 shows the initial dislocation nucleation in bi-
crystal samples with GBs of $\Sigma 3 (1 1 2)/(\bar{5} 5 2)$ and $\Sigma 11 (2 2 5)/(4 4 1)$. The results show that the initial dislocations are nucleated from the GB which are the Shockley partial dislocations with the Burgers vectors of $\frac{1}{6}[1 1 2]$ and $\frac{1}{6}[1 1 2]$ in the cases of $\Sigma 3 (1 1 2)/(\bar{5} 5 2)$ and $\Sigma 11 (2 2 5)/(4 4 1)$ GBs, respectively. In other words, the initial dislocation nucleation does not occur beneath the indenter. Since the dislocation nucleation from the $\Sigma 3 (1 1 2)/(\bar{5} 5 2)$ and $\Sigma 11 (2 2 5)/(4 4 1)$ GBs occurs at the very initial phases of defect nucleation, it severely affects the strength and eliminates the local hardening occurred in the other bi-crystal samples after the sudden drop in hardness, as shown in Figs. 5.5 (d) and (e). One can unravel the variation of hardness versus the indentation depth for all samples using the microstructural information including the total dislocation length and dislocation visualization. Generally, the results show that the size effects is initially controlled by the source exhaustion mechanism which is affected by GB in the cases of S1 samples. Eventually, the total dislocation length required to sustain the plastic flow is provided, and the source exhaustion mechanism becomes inactive. On the other hand, the GB does not have any considerable effect on the forest hardening mechanism. In other words, the interaction between dislocation and GB does not induce any noticeable hardening at this length scale for any of the simulated S1 samples.

Fig. 5.7 presents the variation of the mean contact pressure ($p_m = P/A$), which is equivalent to the hardness $H$ in the plastic region, as a function of indentation depth $h$ for the S2 samples. The results show that the nanoindentation responses of the bi-crystal and their related single crystal samples are nearly similar at the lower indentation depths. However, the bi-crystal samples show larger hardness at higher indentation depths. The variation of $p_m$ and $\lambda$ versus $h$ should be investigated to understand the mechanisms leading to the observed behavior. As an example, Fig. 5.8 illustrates the variation of $p_m$ and $\lambda$ versus $h$ for $\Sigma 3 (1 1 1) [1 \bar{1} 0]$ bi-crystal and its related single crystal S2 samples. The results show that the size effects can be divided in three regions as follows:

- **Region I:** The response in this region is elastic for both bi-crystal and single crystal samples.

Besides the GB, there are no other defects in both bi-crystal and single crystal samples.
Fig. 5.5. Variation of mean contact pressure $p_m$ and dislocation length $\lambda$ as a function of indentation depth $h$ for S1 bicrystal and their related single crystal samples with grain boundaries of: (a) $\Sigma 11 (1 1 3)$ (b) $\Sigma 3 (1 1 2)$ (c) $\Sigma 11 (3 3 2)$ (d) $\Sigma 11 (2 2 5)/(4 4 1)$ (e) $\Sigma 3 (1 1 2)/(5 5 2)$ (f) $\Sigma 3 (1 1 4)/(1 1 0)$. 
Fig. 5.6. Dislocation nucleation from the GB: (a) bi-crystal sample with \( \Sigma 3 \) (1 1 2)/(5 5 2) GB, \( h \approx 0.37 \) nm; (b) bi-crystal sample with \( \Sigma 11 \) (2 2 5)/(4 4 1) GB, \( h \approx 0.43 \) nm.

- **Region II**: The initial dislocation nucleation occurred beneath the indenter. A large drop in hardness occurs immediately after the first dislocation nucleation for both bi-crystal and single crystal samples. The size effects in bi-crystal and single crystal samples are controlled by dislocation nucleation and source exhaustion. As the total dislocation density increases, the resulting mobile dislocation density also increases which leads to the lower hardness according to the source exhaustion mechanism. In **Region II**, the difference between the total dislocation length of the bi-crystal and single crystal samples is small which leads to the
nearly similar hardness for the bi-crystal and single crystal samples according to the source 
exhaustion mechanism. Increasing the indentation depth, the GB starts blocking the 
movement of dislocations. However, the source exhaustion mechanism is still the controlling 
mechanism of size effects and the hardness decreases as the total dislocation length 
increases. The slope of hardness reduction decreases by increasing the indentation depth 
which means that the dominancy of source exhaustion mechanism decreases.

- **Region III**: As the total dislocation length increases, the source exhaustion mechanism 
becomes inactive in this region due to the fact that enough dislocation length is available to 
sustain the applied plastic flow. On the other hand, the forest hardening mechanism gradually 
becomes activated as the total dislocation length increases. The total dislocation length of the 
bi-crystal sample starts deviating from that of the single crystal sample, and it has larger 
values. Furthermore, the total length of dislocation blocked by the GB becomes noticeable. In 
Region III, the hardness in the bi-crystal samples becomes larger than that of the single crystal 

Fig. 5.9 illustrates the dislocation nucleation and evolution for $\Sigma 3 (1 1 1) [1 \bar{1} 0]$ bi-crystal 
and its related single crystal S2 samples. Figs. 5.9 (a) and (b) show that the initial dislocation 
nucleation process is homogeneous which is similar for both single crystal and bi-crystal samples. 
The initial dislocation is a Shockley partial dislocation with the Burgers vector of $\frac{1}{6}[\bar{2} 1 \bar{1}]$. Figs. 5.9 
(c) and (d) show that the dislocations are multiplied using the cross slip mechanism, and the 
dislocations pinned at their ends are elongated to sustain the applied plastic flow. Again, the 
dislocation structure for both single crystal and bi-crystal looks similar at this stage. Increasing the 
indentation depth, the dislocation loops are emitted by cross-slipping and pinching off of screw 
dislocations. In the case of the single crystal sample, Fig. 5.9 (e) shows that the dislocation loop is 
moving downward and reaches the sample bottom. In the case of the bi-crystal sample, however, Fig. 
5.9 (f) shows that the dislocation loops are blocked by the GB. Eventually, the dislocations are emitted
to the lower grain. The first emitted dislocation into the next grain is a Shockley partial dislocation with the Burgers vector of $\frac{1}{6}[1 \bar{2} 1]$ which is depicted in Fig. 5.9 (h). The dislocation structure of the single crystal and bi-crystal samples at higher indentation depths ($h \approx 11.5$ nm) are presented in Figs. 5.9 (i) and (j), respectively. The results show that in the case of bi-crystal, although part of the dislocation are emitted to the next grain, most of the dislocations are located in the upper grain which are blocked by the GB. In the case of single crystal sample, however, there is no obstacle for the dislocation movement and they are freely moving downward.
Fig. 5.7. Variation of mean contact pressure $p_m$ as a function of indentation depth $h$ for S2 single crystal and their related bi-crystal samples with the grain boundaries of (a) $\Sigma 3 (111)$ (b) $\Sigma 11 (113)$ (c) $\Sigma 3 (112)$ (d) $\Sigma 11 (332)$ (e) $\Sigma 11 (225)/(441)$ (f) $\Sigma 3 (112)/(552)$ (g) $\Sigma 3 (114)/(110)$.

Fig. 5.10 shows the variation of $p_m$ and $\lambda$ versus $h$ for all bi-crystal and their related single crystal samples except the sample with $\Sigma 3 (111) [1\bar{1}0]$ GB. The mechanisms described for $\Sigma 3 (111) [1\bar{1}0]$ GB are applicable to all cases except the samples with the GBs of $\Sigma 11 (332)$ and $\Sigma 11 (225)/(441)$ in which the total dislocation lengths of bicrystal samples are nearly similar to those of the related single crystal samples. However, the hardness of bi-crystal samples are larger at higher indentation depths. In the case of source hardening mechanism, the total dislocation length of the thin film is an appropriate representative parameter. However, in the case of forest hardening mechanism, the dislocation density around the indenter is a controlling factor and not the total dislocation length. Here, the plastic zone is assumed to be confined in the upper grain. In other words,
the dislocations which are effective in the forest hardening are the ones located in the plastic zone which can be approximately considered as upper grain. Hence, in order to study the size effects due to the forest hardening, the total dislocation length located in the upper grain $\lambda_{\text{upper}}$ of the bi-crystal sample is more appropriate. In the case of single crystal samples, the dislocations located in the upper one third of the thickness, which is similar to the location of the upper grain, are considered. Fig. 5.11 shows the variation of $p_m$ and $\lambda_{\text{upper}}$ versus $h$ for all bi-crystal and their related single crystal samples. It shows that, the $\lambda_{\text{upper}}$ is larger for all bi-crystal samples at higher indentation depths compared to those of the single crystal samples which leads to the larger hardness according to the forest hardening mechanism. Although some of the dislocations start dissociating to the next grain, the results show that more dislocations are nucleated in the upper grain. Figs. 5.10 and 5.11 show that the GBs main role is to change the pattern of dislocation structure in a way that the dislocations are piled up near the GB which increases the hardness.

Fig. 5.8. Variation of mean contact pressure $p_m$ and dislocation length $\lambda$ as a function of indentation depth $h$ for S2 bicrystal sample with $\Sigma 3 \,(1 \,1 \,1)$ GB and its related single crystal sample.
(a) single crystal sample, \( h \approx 0.88 \) nm

(b) bi-crystal sample, \( h \approx 0.88 \) nm

(c) single crystal sample, \( h \approx 1.15 \) nm

(d) bi-crystal sample, \( h \approx 1.15 \) nm

(e) single crystal sample, \( h \approx 1.44 \) nm

(f) bi-crystal sample, \( h \approx 1.44 \) nm
Fig. 5.9. Dislocation nucleation and evolution: (a) single crystal sample, $h \approx 0.88$ nm; (b) bi-crystal sample, $h \approx 0.88$ nm; (c) single crystal sample, $h \approx 1.15$ nm; (d) bi-crystal sample, $h \approx 1.15$ nm; (e) single crystal sample, $h \approx 1.44$ nm; (f) bi-crystal sample, $h \approx 1.44$ nm; (g) single crystal sample, $h \approx 2.03$ nm; (h) bi-crystal sample, $h \approx 2.03$ nm; (i) single crystal sample, $h \approx 11.5$ nm; (j) bi-crystal sample, $h \approx 11.5$ nm.
Fig. 5.10. Variation of mean contact pressure $p_m$ and dislocation length $\lambda$ as a function of indentation depth $h$ for S2 bicrystal and their related single crystal samples with grain boundaries of: (a) $\Sigma 11 (1 1 3)$ (b) $\Sigma 3 (1 1 2)$ (c) $\Sigma 11 (3 3 2)$ (d) $\Sigma 11 (2 2 5)/(4 4 1)$ (e) $\Sigma 3 (1 1 2)/(5 5 2)$ (f) $\Sigma 3 (1 1 4)/(1 1 0)$. 

- $p_m$ (Single Crystal) 
- $p_m$ (Bi-Crystal) 
- $\lambda_{upper}$ (Single Crystal) 
- $\lambda_{upper}$ (Bi-Crystal)
Fig. 5.11. Variation of mean contact pressure $p_m$ and total dislocation length located in the upper grain $\lambda_{upper}$ as a function of indentation depth $h$ for S2 bicrystal and their related single crystal samples with grain boundaries of (a) $\Sigma 3 (1 1 1)$ (b) $\Sigma 11 (1 1 3)$ (c) $\Sigma 3 (1 1 2)$ (d) $\Sigma 11 (3 3 2)$ (e) $\Sigma 11 (2 2 5)/(4 4 1)$ (f) $\Sigma 3 (1 1 2)/(5 5 2)$ (g) $\Sigma 3 (1 1 4)/(1 1 0)$. 
5.4. Conclusions

The present chapter investigates the coupling effects of sample length scale and grain boundary on the sources of size effects using large scale atomistic simulation. Two different sizes of Ni thin films are incorporated to study the effects of sample length scale. Seven different types of symmetric and asymmetric tilt boundaries are selected to investigate the effects of GB geometry on the size effects. In the case of single crystal samples, the size effects are initially governed by dislocation nucleation and source exhaustion mechanisms. Therefore, increasing the total content of dislocations leads to the hardness reduction because more dislocations are available to sustain the plastic flow. However, the dominancy of source exhaustion hardening decreases as the indentation depth and consequently the total dislocation length increase. Hence, the slope of hardness reduction decreases as the indentation depths increases. Eventually, the source exhaustion mechanism becomes inactive when enough dislocation content is available to sustain the applied plastic flow. On the other hand, the forest hardening mechanism, which is originated from dislocation interactions with each other, becomes more dominant as the total dislocation length increases. Finally, at higher indentation depths, the forest hardening is the only governing mechanism.

The effects of GB on the sources of size effects depend on the sample length scale. GB does not change the general pattern of size effects; however, it can contribute to some specific mechanisms depending on the sample size. The size effects is initially controlled by source exhaustion mechanism during the nanoindentation which is influenced by GB in the cases of small samples. The initial strength drop occurs earlier in small bi-crystal samples compared to their related single crystal samples. It may be originated from the fact the total dislocation lengths of bi-crystal samples are larger than those of the related single crystals at the moment of strength drop which ease the sudden hardness reduction according to the source exhaustion mechanism. Also, the grain boundary itself is a source of dislocation nucleation. However, the effects of a GB, as a nucleation source, on the hardness of a thin film depend on the depth at which the dislocations start nucleating from GB. If the
dislocation nucleation occurs at the very initial phases of indentation, it can severely affect the strength. The forest hardening mechanism becomes dominant at larger indentation depths. In the cases of small samples, however, the responses of bi-crystal and single-crystal samples are similar in this region. It shows that the GB does not change the nanoindentation response at larger indentation depths when the source exhaustion mechanism becomes inactive and forest hardening is dominant. It is worth mentioning that the dislocations movement are blocked by GBs during nanoindentation in small bi-crystal samples. However, the density of piled-up dislocation is not enough to change the nanoindentation response. In the cases of small samples, the best performance between all GBs is demonstrated by coherent twin boundary which has the least strength reduction. Also, it shows small enhancement in hardness at some ranges of indentation depth.

In the cases of large samples, the total dislocation content is much greater than that of the small samples. Hence, the interaction of dislocations and GBs plays a key role. The initial nanoindentation responses of bi-crystal and single crystal samples are similar in the cases of large samples. It is due to the fact that the total dislocation length of the bi-crystal sample is close to that of the single crystal sample. It means that the GB does not have any noticeable effect on the source exhaustion mechanism. The dislocations moving downward are blocked by the GB. At higher indentation depths, the strength of bi-crystal samples becomes larger than that of the single crystal samples. The total dislocation lengths are large at higher indentation depths, and consequently the density of dislocations piled-up behind the GB becomes noticeable. Hence, the increase in bicrystal samples can be justified according to the forest hardening mechanism. Moreover, the results show that the total dislocation length in the upper grain is a better representative factor to investigate the strength size effects. In other words, according to the forest hardening mechanism, the dislocations in the plastic zone which are closer to the indenter are the effective ones. Hence, in the cases of large samples, the most important role of GBs is to change the pattern of dislocation structure by blocking their movement which increases the resulting hardness at higher indentation depths.
CHAPTER 6
SIZE EFFECTS IN FCC CRYSTALS DURING THE HIGH RATE COMPRESSION TEST

6.1. Introduction

The response of materials during high strain rate experiments is crucial for many applications including armor design, automobile collisions, and projectile impact. The dislocation mechanics in high rate deformation is different from those with slower rates. In the case of quasi-static deformations, the shear strain rate can be described using the usual Orowan equation (Armstrong and Li, 2015; Meyers et al., 2009). In the case of high strain rate deformations, however, the shear strain rate should be calculated from the modified Orowan equation considering the rate of dislocation nucleation (Armstrong and Li, 2015; Meyers et al., 2009). In other words, the dislocation mechanics in high strain rate deformations are governed by dislocation generation at the propagating shock front. The material response to high-rate deformations has been investigated using various experiments including split-Hopkinson pressure bar (SHPB), pulsed laser loading, and high intensity laser facilities coupled with X-ray diffraction techniques (Armstrong and Li, 2015; Meyers et al., 2009). Various models have been proposed to capture the governing mechanisms of deformation observed at high strain rate experiments (Armstrong and Li, 2015; Meyers et al., 2009; Armstrong and Walley, 2008; Armstrong et al., 2007; Gurrutxaga-Lerma et al., 2015; Meyers, 1994; Meyers et al., 2003). However, the focus have been more on the deformation mechanisms, and the size effects of materials have not been thoroughly studied in the case of high strain rate deformations.

Size effects in metallic samples have gained a lot of interest between the researchers. The size effects in metallic samples have been initially attributed to the strain gradient available in some types of loading such as microtorsion and nanoindentation (Nix and Gao, 1998; Abu Al-Rub and Voyiadjis, 2004; Voyiadjis and Abu Al-Rub, 2005). It has been observed that the strain gradient increases the dislocation density which enhances the material strength according to the forest hardening mechanism (Nix and Gao, 1998; Abu Al-Rub and Voyiadjis, 2004; Voyiadjis and Abu Al-Rub, 2005).
However, Uchic et al. (2003, 2004) introduced a micropillar compression test without any strain gradients using the focused ion beam (FIB) machining and observed that the material still shows strong size effects. Source exhaustion hardening, dislocation starvation, source truncation, and weakest link theory have been introduced as the controlling mechanisms of size effects in the absence of strain gradient (Uchic, et al., 2009; Kraft et al., 2010). Source exhaustion hardening occurs when the available mobile dislocation density is insufficient to sustain the plastic flow. Consequently, the applied stress should be increased, which is termed as exhaustion hardening. In the case of the micropillar compression experiment, if the dislocation density is small, the mobile dislocations may leave the sample through the free surfaces leading to a dislocation-starved sample. Consequently, the deformation mechanism, which is commonly termed as dislocation starvation hardening, is governed by source-limited activations. It is worth mentioning that the dislocation starvation can also be considered as a special case of source exhaustion. However, in the current chapter, it is considered as a separate mechanism (see, e.g., Sansoz, 2011). Decreasing the sample size can also increase the sample strength by transforming double-ended dislocation sources into the single-ended ones. The resulting hardening mechanism is termed as source truncation. Finally, the strength of the weakest slip plane present in the sample is increased by decreasing the sample size leading to the sample strength enhancement, which is termed as the weakest link theory. El-Awady (2014) studied the effects of sample size and dislocation density on the controlling mechanism of size effects. It was observed that the dislocation starvation governs the size effects for very small dislocation densities. As the dislocation density increases in sample with a specific size, the single-source strengthening governs the size effects. Exhaustion hardening becomes the controlling mechanism of size effects by further increasing the dislocation density. Finally, the forest hardening mechanism controls the sample strength for high enough dislocation contents and the Taylor hardening model can capture the size effects (El-Awady, 2014). As the sample size increases, the dislocation density at which the governing mechanism of size effects changes decreases (El-Awady, 2014). As an example, the
transition from single-source strengthening to dislocation starvation occurs at lower dislocation densities by increasing the sample size.

Discrete dislocation dynamics (DDD) is a very common tool to study the micromechanics of the size effects governing mechanisms in the metallic samples of confined volumes. Benzerga and Shaver (2006) studied the size effects during compression test using 2-D DDD. Benzerga (2008) incorporated the planar DDD and showed that the exhaustion hardening is originated from intermittent elastic deformation and discrete multiplication events. The same method was incorporated by Benzerga (2009) to investigate the effects of initial dislocation density and sample size on the size effects mechanisms during the micropillar compression test. It was concluded that in the cases of lower initial densities, the exhaustion hardening is the governing mechanism of size effects. Increasing the initial dislocation density, the size effects are controlled by forest hardening mechanism (Benzerga, 2009). Although 2-D DDD has significantly contributed to the investigation of size effects, it cannot capture the dislocation curvature. Also, 2-D DDD ignores or simplifies the 3-D mechanisms. Tang et al. (2007) investigates the athermal size effects in micropillar compression experiment using 3-D DDD. They observed that the dislocations escape from the free surfaces is the controlling mechanism of size effects. Rao and his coworkers (Rao et al., 2007; Parthasarathy et al., 2007; Rao et al., 2008) studied the athermal mechanisms of the size effects in the sample with different initial dislocation densities and sample sizes using 3-D DDD. It was observed that the exhaustion hardening controls the size effects for the sample with the length scale smaller than a specific limit. Increasing the sample size, it was concluded that the source truncation should also be taken into account to reasonably capture the size effects (Rao et al., 2008). The cross-slip of dislocation has not been considered in the simulations conducted by Tang et al. (2007) and Rao and his coworkers (Rao et al., 2007; Parthasarathy et al., 2007; Rao et al., 2008). El-Awady et al. (2009) incorporated the cross-slip in 3-D DDD and studied the size effects of Ni micropillars. They stated that the size effects can be interpreted by the weakest-link activation mechanism which states that
the weakest-link activation stress decreases as the sample size increases. Motz et al. (2009) generated the more realistic initial dislocation network and studied the effects of initial dislocation structure on the micropillar mechanical response. They studied the effect of cross-slip on the variation of dislocation density and tensile strength. Zhou et al. (2010) also included the cross-slip in their 3-D DDD model and studied the size effects of metallic samples at small length scales. The initial structures were obtained by cutting the pillars from the bulk sample, and the variation of dislocation density was investigated throughout the simulation. It was concluded that as the sample size increases, the average of effective source length increases which decreases the strength of the dislocation sources. The plastic deformation mechanisms of metallic samples were further investigated by Zhou et al. (2011) using 3-D DDD. Two plasticity mechanisms of surface dislocations nucleation and multiplication of internal dislocations were introduced which are governing for very small and very large length scales, respectively. In the case of intermediate length scales, both mechanisms should be considered. Cui et al. (2014) investigated the single arm mechanism incorporating the 3-D DDD. Next, a theoretical model was developed by including this mechanism in the strain hardening model. Ryu et al. (2015) studied the size effects in fcc pillars using 3-D DDD and showed that the governing mechanism of deformation varies from truncated dislocation sources operations to dislocation nucleation from the free surfaces as the pillar diameter decreases.

Molecular dynamics (MD) is another common method to capture the controlling mechanism of size effects. Compared to the DDD, MD simulation can precisely model the cross-slip, which is an important deformation mechanism in the metallic samples of confined volumes. Also, the free surface can be precisely captured in atomistic simulations. Bringa et al. (2006) incorporated the large scale atomistic simulation to study the deformation mechanisms of fcc metallic samples at high strain rate compression test. They were able to capture the experimentally observed deformation mechanisms of copper during the high strain rate experiments by incorporating a very large sample which contains 352 million atoms. Diao et al. (2006) investigated the variation of nanowire yield strength
with its size using atomistic simulation. It was concluded that the yield strength of the sample with very small length scales is mainly governed by the surface stress. Weinberger and Cai (2008) introduced a dislocation multiplication mechanism for BCC pillars in which the dislocation multiplies itself when it is leaving the pillar due to the free surface stress. Sansoz (2011) comprehensively simulated the size effects of nanopillars with diameters in the range of 10.8 nm to 72.3 nm with the periodic height of 30 nm. Instead of initial defect free structure, the new algorithm for building sample with the appropriate initial dislocation density was proposed. Sansoz (2011) concluded that all nanopillars experience both mechanisms of dislocation exhaustion and source-limited activation. It was stated that the strain value at which the transition between these two size effects mechanisms occurs depends on the pillar size. Weinberger et al. (2012) incorporated the transition state theory to exclude the effect of high strain rates from the MD results. They focused on the dislocation nucleation stress and failure mechanisms for nanowires with a 5 nm nominal size and 15 nm length. Weinberger and Tucker (2012) investigated the stability of single arm sources in nanopillars using MD simulation. It was shown that these sources are not stable enough to create static pining points. Next, they induced some artificially pining points to study the yield strength of nanopillars with various sizes in the presence of stable single arm sources, which shows the strength decreases as the pillar diameter increases. Tucker et al. (2013) studied the effects of grain boundary (GB) on the deformation mechanisms of nanopillars with the diameters of 30 nm using large scale atomistic simulation. The results showed that the GB itself may become a source of dislocation nucleation. Xu et al. (2013) incorporated the atomistic simulation to study the effects of crystallographic orientation and sample size on the Al nanopillar compressive response. Yaghoobi and Voyiadjis (2016a) studied the size effects mechanisms of metallic samples during nanoindentation using atomistic simulation. The variations of dislocation density and hardness versus the indentation depth were incorporated to investigate the mechanisms which controls the size effects. Voyiadjis and Yaghoobi (2016) incorporated large scale MD to model the nanoindentation of bicrystalline thin films and studied the
effects of various grain boundaries on the controlling mechanisms of size effects as the sample length scale increases. Although MD simulation can precisely capture the surface effects and atomistic deformation mechanisms, it can reach a very limited span of time and length scales. In the case of size effects, the sample length scale is very important in a way that some mechanisms cannot be captured using very small samples. However, nowadays, using very efficient parallel MD codes and powerful high performance computing resources, the metallic samples up to 0.3 μm can be simulated. Furthermore, in the case of high rate compression tests, the time span which is required to be captured is reachable using MD simulation. Hence, MD simulation is a very powerful tool to study the size effects in metallic samples of confined volumes during the high strain rate experiments.

In the current study, the size effects in metallic pillars of confined volumes are investigated during high strain rate compression tests. The large scale atomistic simulation is incorporated to study the pillars with different sizes. The dislocation starvation, source exhaustion, and effect of source length are considered as the governing mechanisms of size effects. The size effects mechanisms are then evaluated using the variation of true stress, dislocation density, average dislocation source length, and immobile dislocation density versus the applied strain. Finally, the effects of pre-straining on the governing mechanisms of size effects are investigated in the cases of pillars with various sizes.

6.2. Simulation details and methodology

The parallel code LAMMPS (Plimpton, 1995) is incorporated to conduct the MD simulation of Ni pillars. The embedded-atom method (EAM) potential obtained by Mishin et al. (1999) is incorporated to capture the Ni-Ni atomic interaction. The wide range of sample sizes should be generated to study the controlling mechanism of size effects. Here, the pillars sizes are changed in two different ways. First, the pillar diameter is changed while the height is similar for all pillars. Samples with height of $H=45$ nm and different diameters of $D=22.5$, $45$, $90$, and $135$ nm are generated. Next, the pillar height is varied while the diameter is kept fixed for all pillars. The pillars with the
diameters of 22.5 nm and 135 nm and heights of 30, 45, and 75 nm are generated to study the effects of pillar height on the controlling mechanisms of size effects. Finally, the response of a very large sample with the height of 0.3 μm and diameter of 0.15 μm is generated. The largest sample generated contains around 487 million atoms. The pillars are circular cross sections with the axis along [1 1 1] direction.

The pillar boundary conditions should be carefully selected to accurately capture the dislocation density during the simulation. The boundary conditions at the surroundings, along [1 1 0] and [1 1 2] directions, are set free. Along the loading direction, however, four different types of boundary conditions can be used:

- The periodic boundary conditions are used along the loading direction. The pillar loading procedure is conducted by shrinking the simulation box along the loading direction. Using these boundary conditions, a dislocation which leaves the bottom surface will enter from the upper one.

- The free surface is incorporated for the top, and some atomic layers at the bottom are fixed to simulate the substrate. The pillar can be uniaxially compressed using a large flat indenter. The dislocations cannot pass the bottom surface due to the fixed atomic layers which leads to a slight overestimation of dislocation density.

- The third type of boundary conditions is to model the pillar with its substrate, which is from the same material. Again, the displacement is imposed on the pillar using a flat indenter. This boundary conditions type is able to precisely capture the dislocation content. However, the substrate itself may have some plastic deformation, which is not easy to predict or exclude from the total deformation of the pillar. The substrate deformation is not uniform, and it is a function of pillar dimensions and applied strain. Hence, the pillar strain cannot be easily interpreted.
The boundary conditions for top and bottom surfaces are set free. The substrate is simulated using a prescribed potential wall. The compressive displacement is then applied using a large flat indenter.

The authors tested all four types of boundary conditions and observed that the last one is the most appropriate choice to study the size effects of pillar in which the dislocation density should be precisely captured during the simulation. The interaction between the indenter and Ni atoms is modeled using the repulsive potential as follows:

\[ E^{\text{ind}}(r) = \bar{\varepsilon}_{\text{ind}}(r - r_c)^2 \quad r < r_c \]  

(6.1)

where \( \bar{\varepsilon}_{\text{ind}}, r, \) and \( r_c \) are the specified force constant, distance from particle to the indenter surface, and cutoff distance, respectively. The parameters \( \bar{\varepsilon}_{\text{ind}} \) and \( r_c \) are chosen as 1 eV/Å\(^2\) and 0.3 nm, respectively.

The indenter applies the displacement-controlled compressive load in a way to induce the strain rate of 3.33e8 s\(^{-1}\), which is in the common range of applied strain rate of the previous works (Tucker et al., 2013; Xu et al., 2013). The Lennard–Jones (LJ) potential is incorporated to model the interaction between the substrate and Ni atoms which is defined as follows:

\[ E^{\text{LJ}}(r_{ij}) = 4\varepsilon \left[ \left( \frac{\sigma}{r_{ij}} \right)^{12} - \left( \frac{\sigma}{r_{ij}} \right)^{6} \right] \]  

(6.2)

where \( \sigma \) is the collision diameter at which \( E^{\text{LJ}} = 0 \), and \( \varepsilon \) is the depth of the potential well. The LJ parameters are selected to model the Si substrate, which are \( \varepsilon_{\text{Ni-Si}} = 1.5231 \times 10^{-20} \text{ J} \) and \( \sigma_{\text{Ni-Si}} = 3.0534 \text{ Å} \). The LJ potential cutoff distance is chosen as 2.5\( \sigma \). The velocity Verlet algorithm with the time step of 5 fs is used to numerically integrate the equations of motion. The NVT ensemble is selected to simulate the compression test. The precise cross section should be calculated during the simulation to obtain the true stress. Here, the contact area is obtained using the triangulation method described in Yaghoobi and Voyiadjis (2014) and Voyiadjis and Yaghoobi (2015).
In order to study the effects of pillar initial structure on the governing mechanisms of size effects, two different types of samples are generated as follows:

- First, the pillars are initially defect free and have perfect lattice. The samples are relaxed for 100 ps with the temperature increasing from 1 K to 300 K. The samples are then relaxed for 100 ps at 300 K.
- Second, the pillars are pre-strained. The initial relaxation procedure is similar to the defect free samples. Next, the pillars are uniaxially loaded up to the strain equal to 0.12. Then the samples are unloaded and relaxed for 100 ps at 300 K.

The dislocation structures are extracted at each step using the Crystal Analysis Tool developed by Stukowski and his co-workers (Stukowski and Albe, 2010; Stukowski et al., 2012; Stukowski, 2012; Stukowski, 2014). The dislocation visualization and their information processing, such as the total dislocation length and density calculations, are conducted using the software OVITO (Stukowski, 2010) and Paraview (Henderson, 2007). In order to study the effects of mean source length $\bar{\lambda}$ on the size effects, which is proposed by Parthasarathy et al. (2007), $\bar{\lambda}$ is calculated as follows:

$$\bar{\lambda} = \frac{\lambda}{N}$$  \hspace{1cm} (6.3)

where $\lambda$ is the total dislocation length and $N$ is the total number of dislocation lines.

6.3. Results and Discussions

Fig. 6.1 presents the variation of true stress ($\sigma$) versus the strain ($\varepsilon$) in the cases of pillars with the height of 45 nm and several diameters of 22.5, 45, 90, and 135 nm. In order to study the size effects, the initial dislocation nucleation stress is not considered as the strength of the sample. It is due to the fact that this stress is not realistic and only available in perfect MD samples, which does not have any kind of defect. Instead, the stress after the initial nucleation is incorporated to study the size effects. The pillars compressive responses are initially elastic and independent of pillar diameter.
After the elastic region, however, the stress shows a strong size effects. Fig. 6.1 shows that in the case of $D$ from 22.5 nm to 90 nm, as the diameter decreases the strength increases. However, there is nearly no size effects by increasing the pillar diameter from 90 nm to 135 nm. The variation of dislocation density should be investigated to unravel the controlling mechanisms of observed size effects. Fig. 6.2 shows the variations of dislocation density and true stress versus the strain. Fig. 6.2 (a) shows that the size effects in the pillar with the diameter of 22.5 nm are controlled by the source-limited activation, i.e. dislocation starvation. In other words, each time, the mobile dislocations are driven out of the sample, which locally increases the stress until another dislocation nucleation and evolution occurs leading to a stress relaxation. In the case of the pillar with the diameter of $D=45$ nm, Fig. 6.2 (b) shows that the exhaustion hardening mechanism controls the size effects in a way that as the dislocation density decreases, the stress increases. Some small local jumps are observed in true stress which occur because some dislocations leave the pillar from the free surfaces. However, the peak of these local changes are smaller than those observed in the pillar with the diameter of $D=22.5$ nm. After the elastic response and following stress relaxation, Figs. 6.2 (c) and (d) show that there is no jump in the stress-strain curves in the case of the pillars with the diameters of 90 nm and 135 nm, which is due to the smooth variation of dislocation density. The observed responses are different from experimental results and DDD simulations, which are conducted at much slower rates. The smooth variation of dislocation density is due to the high dislocation density induced to sustain the imposed high rate deformations. The results show that both pillars eventually reach a steady response which is not size dependent. However, they reach the steady states response throughout the different procedures in a way that the response of the pillar with larger diameter, i.e. $D=135$ nm, becomes steady at smaller strains.

Two pillars with the diameters of 90 nm and 135 nm eventually tend to the same stress. It means that if the sample height is kept fixed, there is a limit that increasing the sample diameter will not further change the response. Figs. 6.2 (c) and (d) show that increasing the pillar diameter from
90 nm to 135 nm does not change the dislocation density after the dislocation density reaches its steady state. Fig. 6.3 depicts the variation of mean source length $\bar{\lambda}$ in the cases of pillars with the diameters of 90 nm and 135 nm and height of 45 nm. The results show that the mean source length of the pillar with $D=135$ nm is very close to that of the pillar with $D=90$ nm. It is due to the high dislocation density induced by the imposed high strain rates. El-Awady (2014) studied the variation of effective source length versus the dislocation density. It was shown that for high dislocation densities, the effective source length is not a function of sample size and has an inverse relation with the dislocation density. Consequently, the resulting strength for both samples are similar. The obtained results show that in the region of high strain rates, $\bar{\lambda}$ is not a function of sample size and the size effects mechanisms which depend on $\bar{\lambda}$, such as source truncation, are not active anymore.

![Graph showing the variation of true stress versus the strain in the cases of pillars with the height of $H=45$ nm and different diameters of $D=22.5$, 45, 90, and 135 nm.](image)

**Fig. 6.1.** Variation of true stress versus the strain in the cases of pillars with the height of $H=45$ nm and different diameters of $D=22.5$, 45, 90, and 135 nm.
Fig. 6.2. The compressive responses of pillars with the height of 45 nm and different diameters of: (a) $D=22.5$ nm (b) $D=45$ nm (c) $D=90$ nm (d) $D=135$ nm.

Fig. 6.3. Variation of mean source length versus the strain in the cases of pillars with the height of 45 nm and diameters of 90 and 135 nm.
The responses of pillars with three heights of 30, 45, and 75 nm and two diameters of 22.5 and 135 nm are compared during the compression test to study the effects of pillar height on the controlling mechanisms of size effects. Figs. 6.4 (a) and (b) present the variations of true stress and dislocation density versus the strain, respectively, in the cases of samples with the diameter of 22.5 nm and different heights of 30, 45, and 75 nm. The results show that the size effects governing mechanism is dislocation starvation for all samples. In other words, when the dislocation starvation is the dominant mechanism of size effects, changing the sample height does not considerably alter the pillar response. However, the values of local stress peak are slightly different based on the density of dislocations remains in the sample during the starvation step. For example, the maximum stress peak happens in the case of the pillar with the height of 75 nm at $\varepsilon \approx 0.18$ which is related to the minimum dislocation density occurring slightly prior to the same strain. Fig. 6.5 depicts the response of pillars with the diameter of 135 nm and different heights of 30, 45, and 75 nm during the compression test. Fig. 6.5 (a) shows that as the pillar height decreases, the strength of the pillar increases. As shown in Fig. 6.5 (b), the dislocation starvation does not occur, and the source-limited activation is not the governing mechanism of size effects. Comparing the responses of pillars with the height of 45 nm and diameters of 90 nm and 135 nm in Figs. 6.2 and 6.3, it is shown that the variation of source length is also not responsible for the observed size effects. In the cases of samples with heights of 30 nm and 45 nm, Fig. 6.5 (b) shows that the pillars have nearly the similar dislocation density as the strain varies. However, the required stress to maintain the observed dislocation density increases as the pillar height decreases. It is due to the fact that as the pillar height decreases, dislocations reach the sample top and bottom surfaces faster and they are not able to sustain more deformation. Accordingly, more dislocations should be nucleated to maintain the plastic flow which increases the required stress. Lee et al. (2009) studied the pillars with low height to diameter ratios during the compression experiment and observed that the slip on \{1 1 1\} planes could be inhibited at the top and bottom surfaces. They also observed that the strong adhesion between pillar and
substrate induces a multi-axial stress state. In the case of the current simulation, however, the potential selected to model the substrate does not induce adhesion. In the case of the pillar with the height of 75 nm, due to the similar procedure, the sample strength is less than those of the pillars with the heights of 30 nm and 40 nm. Also, the dislocation density of sample with the height of 75 nm is higher than those of the shorter pillars which provides more dislocations to sustain the plastic flow and decreases the required stress.

![Graphs showing true stress-strain and dislocation density-strain for pillars of different heights.](image)

Fig. 6.4. Compressive response of pillars with the diameter of 22.5 and different heights of 30, 45, and 75 nm: (a) True stress-strain (b) Dislocation density-strain.
In the case of the pillar with the height of 75 nm and after the initial dislocation nucleation phase, the results show that the dislocation density increases as the strain increases. If all the dislocations are mobile, the required stress to sustain the plastic flow should decrease as the dislocation density increases. However, the stress reaches a steady state with a constant value. It is due to the fact that the dislocation density, which is presented in Fig. 6.5, is the total dislocation density including both the mobile and immobile dislocations. In other words, in the case of sample with the height of 75 nm, the increase in total dislocation density as the strain increases is mainly due to the increases in the immobile dislocation density. Hence, the stress does not decreases because what releases the stress is an increase in mobile dislocation density and not the total one. In order to investigate the proposed explanation, the variation of immobile dislocation density verses the strain should be studied. In the case of fcc metals, the mobile dislocations include the majority of Shockley partial dislocations with the Burgers vector of $1/6 \langle 1\bar{1}\bar{2} \rangle$ and perfect dislocations with the Burgers vector of $1/2 \langle 1\bar{1}0 \rangle$. On the other hand, the immobile dislocations include the Hirth, stair-rod, and Frank partial dislocations with the Burgers vectors of $1/3 \langle 001 \rangle$, $1/6 \langle 011 \rangle$, and $1/3 \langle 111 \rangle$, respectively, and some of the Shockley partial dislocations. Here, the density of immobile dislocations $\rho_{\text{immobile}}$ is approximated by the total dislocation density excluding the perfect and Shockley partial dislocations, which is denoted by $\rho'_{\text{immobile}}$. Fig. 6.6 presents the variation of $\rho'_{\text{immobile}}$ versus the strain in the cases of pillars with the diameter of 135 nm and heights of 30, 45, and 75 nm. In order to have the exact value of immobile dislocation density, the immobile Shockley partial dislocations should also be considered. However, there is no applicable way to distinguish between the mobile and immobile Shockley partial dislocations. In the case of the pillar with the height of 75 nm and after the initial dislocation nucleation phase, Fig. 6.6 shows that the $\rho'_{\text{immobile}}$ increases as the strain increases. The results show that, however, the $\rho'_{\text{immobile}}$ does not vary in the cases of smaller pillars.

It is worth mentioning that the immobile dislocations trap other mobile dislocations and locally
immobilized them. Hence, larger $\rho'_{\text{imobile}}$ leads to the larger fraction of immobile Shockley partial dislocations.

![Graphs showing True stress-strain and Dislocation density-strain for pillars of different heights.](image)

**Fig. 6.5.** Compressive response of pillars with the diameter of 135 and different heights of 30, 45, and 75 nm: (a) True stress-strain (b) Dislocation density-strain.

In the final step, the size effects mechanisms of a pillar with the height of 0.3 $\mu$m and diameter of 0.15 $\mu$m ($S1$) are studied. Fig. 6.7 compares the compressive response of the $S1$ sample to the pillar with $D=135$ nm and $H=75$ nm ($S2$). The results show that although the dislocation density of $S1$ is larger than that of the $S2$, besides the initial dislocation nucleation stress, there is no size effects in strength. In other words, in the absence of strain gradient and in the high rate deformations, there is...
no more size effect in the case of pristine pillars larger than the one with $D=135$ nm and $H=75$ nm. It is worth mentioning that part of the differences in dislocation density of the two samples is due to their different aspect ratios. However, as the results indicate, it does not lead to any size effects. Compared to the experimental results obtained at quasi-static rates, such as Greer et al. (2005) and Espinosa et al. (2005), the results show that increasing the strain-rate decreases the size of pillars at which there is no more size effects. It is due to the fact that at high strain rate deformations, the dislocation source size does not govern the size effects. Accordingly, increasing the sample size from $S2$ to $S1$ does not lead to any size effects.

![Graph](image)

Fig. 6.6. Variation of $\rho_{immobile}^{'}$ versus the strain in the cases of pillars with the diameter of 135 nm and different heights of 30, 45, and 75 nm.
Fig. 6.7. Compressive response of pillars with the heights of 75 nm and 0.3 μm and diameters of 135 nm and 0.15 μm, respectively.

Fig. 6.8. Compressive response of pillar with the diameter of 15 nm and height of 30 nm.

In order to study the microstructure of different size effects governing mechanisms, the dislocation visualization is incorporated here for the largest and smallest simulated pillar heights,
which are $H=30$ nm and $H=0.3$ μm, with the height to diameter ratio of 2:1. Fig. 6.8 presents the compressive response of the pillar with the heights of 30 nm and diameter of 15 nm. The dislocation nucleation and evolution patterns at various strains are shown in Fig. 6.9. The color of Shockley, Hirth, and stair-rod partial dislocations and perfect dislocations are green, yellow, blue, and red, respectively. The initial dislocation nucleation occurs at the free surfaces. The results show that the dominant dislocation multiplication mechanism is the dislocation nucleation from the free surfaces which follows by cross-slip, which is termed as surface cross-slip (Rao et al., 2013). Hussein et al. (2015) observed a similar deformation mechanism during 3-D DDD simulation of fcc crystals. However, since the pillar diameter is so small, cross-slip cannot create many pining points before the dislocations leave the sample from another free surface. The results show that each time, the dislocation starvation occurs, the strength required to sustain the plastic flow starts increasing. New dislocations are then nucleated and elongated which releases the stress as shown in Figs. 6.9 (b), (c), (e), (g), and (i). Dislocations eventually leave the pillar which leads to another starvation, as shown in Figs. 6.9 (d), (f), and (h). Fig. 6.10 illustrates the dislocation nucleation and evolution of the pillar with the height of 0.3 μm and diameter of 0.15 μm. Fig. 6.10 (b) shows that the initial dislocation nucleation occurs from the free surfaces which follows by the cross-slip. However, as shown in Fig. 6.10 (c), the dislocation starvation does not occur in the pillar. In order to investigate the dislocation multiplication mechanism, Fig. 6.11 shows the dislocations in a small block of pillar at $\varepsilon = 0.1416$, which is presented in Fig. 6.10 (c). Fig. 6.11 shows several cross-slips which increase the number of pinning points. Next, the elongation of the dislocations which are pinned at their ends provides the dislocation length required to sustain the imposed plastic flow. The results show that if the dislocations do not immediately leave the sample, the dominant dislocation multiplication mechanism is cross-slip. It is worth mentioning that in the case of quasi-static experiment, if the sample is large enough that the dislocation starvation does not occur, the plastic deformation
mechanism is governed by truncated dislocation sources operations (Rao et al., 2007). The results show that this mechanism is not available anymore at high rate deformations.
Sansoz (2011) presented a model which stated that all nanopillars experience both deformation mechanisms of source-limited activation, i.e. dislocation starvation, and mobile dislocation exhaustion, i.e. exhaustion hardening. It was demonstrated that as the strain increases, the governing mechanism of deformation will change from the dislocation exhaustion to the source-limited activation. It was also stated that all samples will experience the phase which the dislocation exhaustion is the dominant mechanism of deformation. However, Figs. 6.1 and 6.2 show that in the case of sample with very small diameter, such as D=22.5 nm, the governing mechanism of size effects is solely dislocation starvation. Also, the results show that the dislocation exhaustion is the only governing mechanism for pillars with a large diameter, such as D=135 nm. Sansoz (2011) incorporated the pillars which contain the initial dislocations. In order to study the effect of pillar initial structure on the governing mechanisms of size effects, the responses of pre-strained pillars with the height of 45 nm and diameters of 22.5 nm and 135 nm, which have been initially loaded and unloaded, are studied during the compression test.
Fig. 6.10. The visualization of dislocations structure during compression test in the case of pillar with the diameter of 0.15 μm and height of 0.3 μm at different strains of: (a) \( \varepsilon = 0.0233 \) (b) \( \varepsilon = 0.0433 \) (c) \( \varepsilon = 0.1416 \).

Figs. 6.12 (a) and (b) present the variations of stress and dislocation density versus the strain, respectively, during the compression test for both pristine and pre-strained pillars. In the case of larger sample, i.e. pillar with D=135 nm and H=45 nm, the pre-straining only alters the initial part of the response, and both stress and dislocation density become similar for the pristine and pre-strained pillars after the initial phase of dislocation nucleation. In the cases of smaller sample, i.e. pillars with D=22.5 nm and H=45 nm, besides the initial phase of dislocation nucleation, the pre-straining slightly shift the location of stress peaks. However, the size effects is still governed by the source-limited activation. The results show that the controlling mechanisms of size effects are independent of sample initial structure in the cases of pillars with diameters of 22.5 nm and 135 nm and height of 45 nm. Fig. 6.13 compares the response of the pre-strained sample with height of 0.3
\( \mu m \) and diameter of 0.15 \( \mu m \) to that of the pristine pillar to investigate the effect of initial structure in the case of larger pillars. The results show that the pre-straining increases the dislocation density which triggers the forest hardening mechanism. In other words, the strength increases as the strain increases due to the increase in dislocation density which activates the mechanism of dislocation interaction with each other.

\[ \varepsilon = 0.1416 \]

![Fig. 6.11. A selected block of dislocations in the case of pillar with the diameter of 0.15 \( \mu m \) and height of 0.3 \( \mu m \) at \( \varepsilon = 0.1416 \).](image)

### 6.4. Conclusions

The present chapter investigates the governing mechanisms of size effects in fcc metallic samples of confined volume under high strain rate loading using large scale MD simulation. Samples with different aspect ratios and sizes are simulated to cover the wide range of pillar length scales. The smallest pillar has the diameter of 15 nm and height of 30 nm, and the largest pillar has the diameter of 0.15 \( \mu m \) and height of 0.3 \( \mu m \). In the case of quasi-static loading, the size effects are controlled by the dislocation starvation, source exhaustion, and effects of dislocation source length. In the case of high rate deformation, however, the results show that only the first two mechanisms are active. The mean source length is not a function of sample size anymore, which is due to the high dislocation density induced by high rate deformations. Also, the results show that increasing the strain rate decreases the pillar size at which the size effects governing mechanisms changes from
source exhaustion to dislocation starvation. It is shown that the controlling mechanism of deformation is independent of the strain value and solely depends on the pillar size.

Fig. 6.12. Compressive response of pristine and pre-strained pillars with the height of 45 nm and different diameters of 22.5 nm and 135 nm: (a) True stress-strain (b) Dislocation density-strain.
In the case of quasi-static loading, the plastic deformation is governed by two mechanisms of dislocation nucleation at the free surface and truncated dislocation sources operations which the former mechanism is dominant in the case of the pillars with diameter smaller than 200 nm (Ryu et al., 2015). However, the results show that in the case of high rate deformations and large samples, the mechanism of truncated dislocation sources is not active anymore. Instead, the cross-slip becomes the dominant mechanism of plastic deformation at the larger samples which increases the number of pinning points. Furthermore, in the case of high strain rate deformations, the dislocation nucleation at the free surface becomes dominant for pillars with the diameters smaller than 45 nm, which is smaller than the one in the quasi-static loading, i.e. 200 nm. In other words, increasing the strain rate decreases the limit for changing the deformation mechanisms.

Finally, the effects of pillar initial structure are studied by comparing the responses of pre-strained pillars with those of the pristine pillars. In the cases of samples with the height of 45 nm and diameters of 22.5 nm and 135 nm, the pre-straining only alters the initial part of the response.
including the elastic part and initial dislocation nucleation. Following the initial part of response, however, both true stress and dislocation density are not considerably influenced by pre-straining. In the case of the pillar with the height of 0.3 \( \mu \text{m} \) and diameter of 0.15 \( \mu \text{m} \), the dislocation density increases due to the pre-straining which triggers the forest hardening mechanism. In other words, as the strain increases, the strength increases due to the increase in dislocation density, which activates the mechanism of dislocation interaction with each other.
CHAPTER 7
SIZE AND STRAIN RATE EFFECTS IN METALLIC SAMPLES OF CONFINED VOLUMES: DISLOCATION LENGTH DISTRIBUTION

7.1. Introduction

The most common experiment to investigate the size effects in samples of confined volumes is the micropillar compression test, which was introduced by Uchic et al. (2003,2004). During the micropillar compression test, the sample is subjected to the uniform loading and in the absence of any strain gradients, the size effects are solely originated from the size of the tested pillars. Size effects in metallic samples of confined volume have been attributed to the different mechanisms of dislocation starvation, source exhaustion, and the effect of dislocation source length (Uchic et al., 2009; Kraft et al., 2010). In the case of the latter mechanism, the strength of the sample is controlled by the longest dislocation source, which is the first dislocation activated by the applied stress. Parthasarathy et al. (2007) presented an equation to describe the probability for the maximum distance from the pin to the free surface that leads to the longest dislocation arm. They also proposed an equation to incorporate the dislocation source length into the calculation of critical resolved shear strength (CRSS). Rao et al. (2008) incorporated 3-D discrete dislocation dynamics (DDD) and investigated the athermal mechanisms of size effects during the micropillar compression test. They reported the values of largest source length for 26 different samples with the same size and initial dislocation density. El-Awady et al. (2009) investigated the size effects using 3-D DDD and observed that the mean dislocation length governs the size effects. They incorporated a random dislocation length distribution that follows the two-parameter Weibull distribution. El-Awady et al. (2007) observed that to reproduce the experimental results obtained by Dimiduk et al. (2005) and Frick et al. (2008), the mean length of dislocation should be chosen equal to the $D/25$ which $D$ is the pillar diameter. They also observed that the dislocation lengths close to the pillar diameter are rare. Cui et al. (2014) studied the dislocation source as the governing mechanism of size effects using 3-D DDD.
They measured the stable source lengths for samples with various sizes and observed that the average stable source lengths is a function of sample diameter (Cui et al, 2014). El-Awady (2014) conducted 3-D DDD simulations of pillars with various sizes and initial dislocation densities and captured the variation of mean dislocation length using the sample size and initial dislocation density. The dislocation length distribution has been extensively studied for bulk materials (El-Awady, 2009; Lin et al., 1989; Mughrabi et al., 1976; Ardell and Lee, 1986). However, it has not been investigated for samples of confined volumes. In the current chapter, the size and strain rate effects are presented during the micropillar compression test. The results show that increasing the strain rate decreases the size effects. Next, it is shown that the dislocation density is not the appropriate parameter for studying size effects in samples of confined volumes. Finally, the dislocation length distribution is investigated for pillars of different sizes subjected to the compression loading of various strain rates. The size and strain rate effects are captured using the observed dislocation network properties. It is shown that in the cases of the studied samples, one should incorporate the largest dislocation length and not the mean dislocation length to capture the size and strain rate effects.

7.2. Simulation details and methodology

The first mathematical prediction of effective dislocation source length was proposed by Parthasarathy et al. (2007). They introduced the probability of a cylindrical sample with \( n \) pins to have the maximum distance from the free surface equal to \( \lambda_{\text{max}} \) as follows:

\[
p(\lambda_{\text{max}})d\lambda_{\text{max}} = \left[ 1 - \frac{\pi (R - \lambda_{\text{max}})(b - \lambda_{\text{max}})}{\pi R b} \right]^{n-1} \left( \frac{\pi [(R - \lambda_{\text{max}}) + (b - \lambda_{\text{max}})]}{\pi R b} \right)^n d\lambda_{\text{max}} \quad (7.1)
\]

where \( R \) is the specimen radius, \( b = R / \cos \beta \) is the major axis of the glide plane, and \( \beta \) is the angle between the primary slip plane and the loading axis. Using the probability function defined in Eq. (7.1), the mean effective source length \( \bar{\lambda}_{\text{max}} \) can be obtained as below:
\[ \bar{\lambda}_{\text{max}} = \int_{0}^{R} \lambda_{\text{max}} \rho(\lambda_{\text{max}}) d\lambda_{\text{max}} \]

\[ = \int_{0}^{R} \left[ 1 - \frac{\pi(R - \lambda_{\text{max}})(b - \lambda_{\text{max}})}{\pi R b} \right]^{n-1} \left( \frac{\pi(R - \lambda_{\text{max}}) + (b - \lambda_{\text{max}})}{\pi R b} \right) n\lambda_{\text{max}} d\lambda_{\text{max}} \]

(7.2)

In the next step, the CRSS was related to \( \bar{\lambda}_{\text{max}} \) as follows:

\[ \text{CRSS} = \frac{\alpha G b}{\bar{\lambda}_{\text{max}}} + \tau_{0} + 0.5 G b \sqrt{\rho} \]

(7.3)

where \( \alpha \) is a constant, \( G \) is the shear modulus, \( b \) is the Burgers vector, \( \tau_{0} \) is the friction stress, and \( \rho \) is the dislocation density. The number of pins, \( n \), is also predicted as follows:

\[ n = \text{Integer} \left[ \frac{L_{\text{mobile}}}{L_{\text{ave}}} \right] \]

(7.4)

where \( L_{\text{ave}} \) is the average length of dislocation segments, \( L_{\text{mobile}} = \rho n R^{2} h / s \) is the total length of mobile dislocations, \( h \) is the height of the pillar, and \( s \) is the number of slip systems. Basically, Parthasarathy et al. (2007) and later Rao et al. (2008) related the size effects to the average largest source length \( \bar{\lambda}_{\text{max}} \). El-Awady and his coworkers (El-Awady et al., 2008; El-Awady et al., 2009; El-Awady, 2014) related the size effects to the mean dislocation length \( L_{\text{ave}} \). El-Awady (2014) proposed an equation to obtain \( L_{\text{ave}} \) using the pillar diameter and initial dislocation density \( \rho_{0} \) as follows:

\[ L_{\text{ave}} = \frac{b D \sqrt{\rho_{0}}}{\beta} \quad \text{for} \ \rho_{0} < \rho_{0}^{c} \]

(7.5a)

\[ L_{\text{ave}} = \frac{1}{\alpha \sqrt{\rho_{0}}} \quad \text{for} \ \rho_{0} \geq \rho_{0}^{c} \]

(7.5b)

where \( \rho_{0}^{c} \) is the critical initial dislocation density at which \( L_{\text{ave}} \) becomes independent of sample size, and \( \alpha \) and \( \beta \) are dimensionless constants.

In the current chapter, the molecular dynamics (MD) simulation is performed using the parallel code LAMMPS (Plimpton, 1995) to study the size and strain rate effects. The embedded-atom method (EAM) potential developed by Mishin et al. (1999) is used to model the Ni-Ni atomic interaction. Two pillars with circular cross section, heights of 90 nm and 300 nm, and aspect ratio of \( \text{Length : Diameter} = 2 : 1 \) are modeled, which consist of 13 million and 487 million atoms, respectively. The axis of pillars is along the \([1 1 1]\) direction. The boundary conditions are selected similar to the
A prescribed potential wall is incorporated to simulate the substrate. A large flat indenter is used to impose the compressive displacement with three strain rates of $\dot{\varepsilon}_1 = 6.66 \times 10^8 \text{s}^{-1}$, $\dot{\varepsilon}_2 = 3.33 \times 10^8 \text{s}^{-1}$, and $\dot{\varepsilon}_3 = 1.665 \times 10^8 \text{s}^{-1}$, which are in the common range of applied strain rates of the previous works. The following repulsive potential is incorporated to model the interaction between the indenter and Ni atoms:

$$E^{\text{ind}}(r) = \tilde{\varepsilon}_{\text{ind}}(r - r_c)^2 \quad r < r_c \quad (7.6)$$

where $\tilde{\varepsilon}_{\text{ind}} = 1 \text{ eV/Å}^2$, $r$, and $r_c = 0.3 \text{ nm}$ are the specified force constant, distance from particle to the indenter surface, and cutoff distance, respectively. The Si substrate is modeled using the Lennard–Jones (LJ) potential as follows:

$$E^{\text{LJ}}(r_{ij}) = 4\tilde{\varepsilon}
\left[\left(\frac{\tilde{\sigma}}{r_{ij}}\right)^{12} - \left(\frac{\tilde{\sigma}}{r_{ij}}\right)^6\right] \quad (7.7)$$

where $\tilde{\sigma}$ is the collision diameter at which $E^{\text{LJ}} = 0$, and $\tilde{\varepsilon}$ is the depth of the potential well. The LJ parameters are $\tilde{\varepsilon}_{\text{Ni-Si}} = 1.5231 \times 10^{-20} \text{ J}$ and $\tilde{\sigma}_{\text{Ni-Si}} = 3.0534 \text{ Å}$ with the cutoff distance equal to $2.5\tilde{\sigma}$.

The velocity Verlet algorithm with the time step of 5 fs is used to numerically integrate the equations of Motion. The simulation is conducted using the NVT ensemble. The triangulation method is incorporated to capture the precise cross section area during the simulation. The crystal Analysis tool (Stukowski and Albe, 2010; Stukowski et al., 2012; Stukowski, 2012; Stukowski, 2014) is used to extract the dislocation structure from the atomic trajectories. The dislocation network is visualized and analyzed using the software Paraview (Stukowski and Albe, 2010; Stukowski et al., 2012; Stukowski, 2012; Stukowski, 2014, 2007) and OVITO (Stukowski, 2010).

### 7.3 Results and Discussions

Since the samples are initially defect free at the start of MD simulation, which is not true in the real experiments, the stress after the initial nucleation is incorporated to study the size effects (see, e.g., Yaghoobi and Voyiadjis, 2016b). Fig. 7.1 presents the variations of true stress ($\sigma$) and
dislocation density ($\rho$) versus the strain ($\varepsilon$) in the case of the smaller pillar at three different strain rates of $\dot{\varepsilon}_1 = 6.66 \times 10^8 \text{ s}^{-1}$, $\dot{\varepsilon}_2 = 3.33 \times 10^8 \text{ s}^{-1}$, and $\dot{\varepsilon}_3 = 1.665 \times 10^8 \text{ s}^{-1}$. Fig. 7.1 shows that in the case of the smaller pillar, the sample strength is nearly independent of the strain rate. However, the dislocation density increases as the strain rate increases. The results indicate that the dislocation density is not an appropriate measure to study the size effects in the case of metallic samples of confined volumes.

Fig. 7.2 presents the $\sigma - \varepsilon$ and $\rho - \varepsilon$ in the case of the larger pillar at three different strain rates of $\dot{\varepsilon}_1 = 6.66 \times 10^8 \text{ s}^{-1}$, $\dot{\varepsilon}_2 = 3.33 \times 10^8 \text{ s}^{-1}$, and $\dot{\varepsilon}_3 = 1.665 \times 10^8 \text{ s}^{-1}$. The dislocation density shows a similar trend compared to that of the smaller sample as the strain rate varies. However, the results show that the strength of the sample decreases by decreasing the strain rate. Fig. 7.3 compares the responses of pillars with different sizes during the compression test for different strain rates. The results show that as the strain rate increases, less size effects are observed in the simulated samples. In other words, increasing the strain rate decreases the size effects.

![Fig. 7.1](image)

Fig. 7.1. The compressive responses of the pillar with the diameter of 45 nm at different strain rates of $\dot{\varepsilon}_1 = 6.66 \times 10^8 \text{ s}^{-1}$, $\dot{\varepsilon}_2 = 3.33 \times 10^8 \text{ s}^{-1}$, and $\dot{\varepsilon}_3 = 1.665 \times 10^8 \text{ s}^{-1}$. 

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Fig. 7.2. The compressive responses of the pillar with the diameter of 150 nm at different strain rates of $\dot{\varepsilon}_1 = 6.66 \times 10^8$ s$^{-1}$, $\dot{\varepsilon}_2 = 3.33 \times 10^8$ s$^{-1}$, and $\dot{\varepsilon}_3 = 1.665 \times 10^8$ s$^{-1}$.

In order to capture the observed results, the dislocation length distribution is studied for different sample sizes subjected to various strain rates. The distribution of each sample is obtained by averaging the distributions at five strain values of 0.1, 0.125, 0.15, 0.175, and 0.2. Fig. 7.4 presents the variation of probability density function (PDF) versus the dislocation link length in both pillar sizes at different strain rates. The vertical axis of Fig. 7.4, i.e. PDF, is presented in logarithmic form. In the case of dislocation length distribution function of bulk materials, the maximum PDF occurs for some dislocation length in the middle of the distribution, and the dislocation length distribution can be approximated by a Weibull distribution function, see e.g. El-Awady et al. (2009).

In the case of samples of confined volumes, however, the results show that most of the dislocation lengths are populated in the first length bean, which has the smallest length. In other words, the maximum PDF occurred at the smallest dislocation length bean. It is due to the activity of cross-slip as the major deformation mechanism in the samples with confined volumes. To verify the
proposed description, the dislocation visualization of both pillar sizes at $\varepsilon = 0.2$ for strain rate of $\dot{\varepsilon}_2$ is presented as an example in Fig. 7.5. The visualization results show that the main mechanism of deformation is cross-slip which produces many small dislocations. A similar trend can be observed in the results obtained by Hussein et al. (2015) in which many small dislocations are produced by incorporating the cross-slip in the DDD formulation.

In the case of the smaller pillar, the values of average dislocation length $L_{\text{ave}}$ are 25.29 Å, 27.03 Å, and 27.2 Å at strain rates of $\dot{\varepsilon}_1$, $\dot{\varepsilon}_2$, and $\dot{\varepsilon}_3$, respectively. In the case of the larger sample, the average dislocation length values are 21.09 Å, 23.2 Å, and 24.75 Å at strain rates of $\dot{\varepsilon}_1$, $\dot{\varepsilon}_2$, and $\dot{\varepsilon}_3$, respectively. As described by El-Awady (2014), $L_{\text{ave}}$ can be a function of dislocation density and sample size depending on the dislocation density of the sample. Considering the order of dislocation density which is $10^{16} \text{ m}^{-2}$, the $L_{\text{ave}}$ should follow Eq. (7.5b) in which the $L_{\text{ave}}$ is independent of the sample size and has an inverse relation with the dislocation density.

The results show that $L_{\text{ave}}$ independent of the sample size, i.e. $L_{\text{ave}}$ of the larger pillar are close to that of the smaller one and even the smaller pillar has slightly larger $L_{\text{ave}}$. Also, as the strain rate increases, $L_{\text{ave}}$ decreases which is due to the fact that increasing the strain rate increases the dislocation density. Eq. (7.5), which was proposed by El-Awady (2014), can be micromechanically justified based on the dislocation network characteristics. In the region of small dislocation densities, i.e. Eq. (7.5a), increasing the sample size and dislocation density increases the chance of larger dislocation formation. In the region of high dislocation densities, however, increasing the dislocation density increases the chance of dislocations colliding with each other and dislocation refinement which decrease the dislocation length and consequently $L_{\text{ave}}$.

The values of the $\frac{(L_{\text{ave}})_{\dot{\varepsilon}_2}}{(L_{\text{ave}})_{\dot{\varepsilon}_1}}$ for smaller and larger pillars are 1.08 and 1.17 which are very close to each other. However, the strength of the smaller sample does not change as the strain rate varies while the larger sample inhibits significant strain rate effects. The results show that the strain rate effects cannot be captured using $L_{\text{ave}}$. Another way to reach the same conclusion is since $L_{\text{ave}}$ is a
function of dislocation density, and dislocation density variation is not capable of capturing size effects as shown in Figs. 7.1 and 7.2. Accordingly, \( L_{\text{ave}} \) is also not an appropriate dislocation network property to study the size effects.

Another dislocation network property which can be incorporated to study the size effects is the largest dislocation length \( L_{\text{max}} \). Here, the \( L_{\text{max}} \) is averaged at five strain values of 0.1, 0.125, 0.15, 0.175, and 0.2 for each pillar size and strain rate. The values of \( L_{\text{max}} \) for the smaller sample and different rates of \( \dot{\varepsilon}_1, \dot{\varepsilon}_2, \) and \( \dot{\varepsilon}_3 \) are 377.9 Å, 415 Å, and 432.9 Å, respectively. In the case of the larger sample, \( L_{\text{max}} \) values are 605.8 Å, 783.4 Å, and 1095.6 Å for strain rates of \( \dot{\varepsilon}_1, \dot{\varepsilon}_2, \) and \( \dot{\varepsilon}_3 \), respectively. The values of the \( \frac{(L_{\text{max}})_{\dot{\varepsilon}_2}}{(L_{\text{max}})_{\dot{\varepsilon}_1}} \) for smaller and larger pillars are 1.14 and 1.81 which shows a great difference compared to the values of \( \frac{(L_{\text{ave}})_{\dot{\varepsilon}_2}}{(L_{\text{ave}})_{\dot{\varepsilon}_1}} \).

As an example, in the case of Fig. 7.5, the true stresses for smaller and larger pillars at \( \varepsilon = 0.2 \) and strain rate of \( \dot{\varepsilon}_2 \) are \( \sigma_{D=45 \, \text{nm}} = 4.72 \, \text{GPa} \) and \( \sigma_{D=150 \, \text{nm}} = 3.75 \, \text{GPa} \), respectively, where \( \sigma_1 / \sigma_2 = 1.26 \). The average dislocation length for the smaller and larger pillars are \( (L_{\text{ave}})_{D=45 \, \text{nm}} = 25.7 \, \text{Å} \) and \( (L_{\text{ave}})_{D=150 \, \text{nm}} = 26.1 \, \text{Å} \), respectively, which are nearly similar. The maximum dislocation length, on the other hand, for the smaller and larger samples are \( (L_{\text{max}})_{D=45 \, \text{nm}} = 442 \, \text{Å} \) and \( (L_{\text{max}})_{D=150 \, \text{nm}} = 641 \, \text{Å} \), respectively, which shows that the sample with smaller \( L_{\text{max}} \) has the larger strength.

7.4. Conclusions

The results show that the maximum dislocation length \( L_{\text{max}} \) is the appropriate dislocation network property to study the size effects and not the \( L_{\text{ave}} \). It is due to the fact that there are tremendous small dislocations induced by the cross-slip in the samples of confined volumes, which was also observed by Hussein et al. (2015). Accordingly, the average dislocation length is highly influenced by the small dislocations, and the effect of maximum source length on \( L_{\text{ave}} \) diminishes.
Fig 7.3. Variation of true stress versus the strain in the cases of the pillars with the diameters of 45 nm and 150 nm at different strain rates of: (a) $\dot{\varepsilon}_1 = 6.66 \times 10^8$ s$^{-1}$ (b) $\dot{\varepsilon}_2 = 3.33 \times 10^8$ s$^{-1}$ (c) $\dot{\varepsilon}_3 = 1.665 \times 10^8$ s$^{-1}$. 
Fig. 7.4. Probability distribution function of dislocation lengths at different strain rates and pillars with the diameters of: (a) $D = 45$ nm (b) $D = 150$ nm.
(a) \(D = 150\) nm  
(b) \(D = 45\) nm

Fig. 7.5. The dislocation network at \(\varepsilon = 0.2\) for the strain rate of \(\dot{\varepsilon}_2 = 3.33 \times 10^8\) s\(^{-1}\) and pillar diameters of: (a) \(D = 150\) nm (b) \(D = 45\) nm.

The maximum dislocation length \(L_{\text{max}}\), on the other hand, is fully capable of capturing size and strain rate effects. For example, the results observed in Fig. 7.3 can be fully explained by \(L_{\text{max}}\). The \(L_{\text{max}}\) in the larger pillar divided by that of the smaller pillar is 1.6, 1.89, and 2.53 at different
strain rates of $\dot{\varepsilon}_1$, $\dot{\varepsilon}_2$, and $\dot{\varepsilon}_3$ which shows that increasing the strain rate decreases the size effects by decreasing the difference between the $L_{\text{max}}$ for samples of different sizes.
CHAPTER 8
MICROSTRUCTURAL INVESTIGATION OF THE HARDENING MECHANISM IN FCC CRYSTALS DURING HIGH RATE DEFORMATIONS

8.1. Introduction

Up to now, many strengthening mechanisms have been identified in bulk metallic samples including forest hardening mechanism (i.e. interaction of dislocations with each other), Hall-Petch effects (i.e. interaction of dislocation with the grain boundary), solid-solution strengthening, precipitation strengthening, and variation of lattice resistance (Argon, 2008). When it comes to the high rate deformations, the experimental and theoretical studies have demonstrated intriguing deformation and strengthening mechanisms that are different from those of the slower rates. Several experimental techniques of gas guns, split Hopkinson bars, Z-pinch, and high-energy pulsed lasers have been developed to study the material behavior during high rate deformations (Park et al., 2015; Remington et al., 2017). The experiments cover a wide range of strain rates up to \(10^{10} \text{s}^{-1}\). Recently, Crowhurst et al. (2016) incorporated a laser pulse to accelerate the free surface of tantalum and studied its yielding at strain rates up to \(10^9 \text{s}^{-1}\). Remington et al. (2017) reviewed the application of high-power pulsed to induce the strain rates of \(10^6 - 10^{10} \text{s}^{-1}\). They addressed the fundamental issues at high strain rates including dislocation velocity, the slip-twinning transition, and the transition between thermally-activated and phonon drag regimes. The experimental results showed that the mechanisms governing the strengthening in the high rate deformations are different from those of the slower strain rates. As an example, Park et al. (2015) showed that the plastic flow in metallic materials is independent of the grain size at ultrahigh pressures and strain rates and the conventional Hall-Petch effect is not available anymore.

The governing deformation mechanisms in metallic samples depend on the applied strain rate. The Frank-Read source operation, which is a well-established deformation mechanism (see, e.g., Hirth and Lothe, 1982; Hull and Bacon, 2011), is the primary mechanism of deformation for strain
rates up to $10^3 - 10^6 \, s^{-1}$ (see, e.g., Gurrutxaga-Lerma et al., 2015). Increasing the strain rates, the cross-slip and homogenous dislocation nucleation become the dominant mechanisms of deformation. The relation between the material strength and strain rate have been captured using various mechanisms. The primary relation between the material strength and strain rate can be developed using the Arrhenius form equation that leads to a linear relation between the strength and the natural logarithm of strain rate. The linear relation is applicable for strain rates up to $10^4 \, s^{-1}$. In this range of strain rates, the thermal-activation mechanism controls the variation of strength as the strain rate changes (Barton et al., 2011; Barton and Rhee, 2013). Gurrutxaga-Lerma et al. (2015) captured the variation of strength versus the strain rate by relating the activation time and strength of Frank-Read sources to the applied strain rate that is applicable to strain rates up to $10^6 \, s^{-1}$. The relation between the strength and strain rate for strain rates higher than $10^4 \, s^{-1}$ can be described using the interaction between the moving dislocations with phonon drag (Barton et al., 2011; Barton and Rhee, 2013). Furthermore, in the cases of strain rates larger than $10^4 \, s^{-1}$, Fan et al. (2012) incorporated the non-Arrhenius behavior in their model to capture the relation between the strength and the applied strain rate.

Theoretical models which define the relation between the strain rate and strength of fcc metallic structures have been widely investigated (see, e.g., Hirth and Lothe, 1982; Hull and Bacon, 2011). The microstructural studies can be incorporated to evaluate and modify the developed models. In the case of fcc metallic structures, the hardening mechanism is controlled by the properties of dislocation network (see, e.g., Hirth and Lothe, 1982; Hull and Bacon, 2011). In order to study the evolution of dislocation network as the strain rate varies, the incorporated microstructural method should be able to capture dislocations as separate entities. Discrete dislocation dynamics (DDD) has been incorporated to study the hardening mechanisms in metallic materials using the dislocation network properties (see, e.g., El-Awady et al., 2016). In the case of high rate deformations, the cross-slip is one of the major governing deformation mechanisms. The
cross-slip has been incorporated in DDD using some ad-hoc assumptions (see, e.g., Wang et al., 2007; Hussein et al., 2015), and more investigations should be conducted to evaluate the accuracy of DDD to capture the cross-slip at high strain rates. Furthermore, Gurrutxaga-Lerma et al. (2013) showed that a dynamic DDD should be incorporated to capture the hardening mechanism at high strain rates. However, until now, an investigation of the hardening mechanism in bulk fcc materials at high rate deformations that relates the material strength to the dislocation network properties, such as dislocation length distribution, has not been conducted using a DDD which incorporates a well-established cross-slip model and appropriate dynamic framework.

Another method to investigate the microstructural evolution of metallic structures as the strain rate varies and its relation with the hardening mechanism is to model the sample with full atomistic details using molecular dynamics (MD). After defining appropriate atomic interactions, MD is fully capable of capturing all the deformation mechanisms, such as cross-slip, without using any ad-hoc approximation. However, MD, due to its nature, has limitations on the simulation length scale and time scale. Nowadays, by developing efficient parallel algorithms and powerful supercomputers, samples consisting of one billion atoms can be modeled using atomistic simulation. In the case of time scale, although the MD cannot capture the quasi-static experiments, it is an appropriate method to model the high rate deformations. Unlike the DDD which directly reports the dislocation properties, MD only calculates the atomic trajectories and velocities. Accordingly, the dislocation properties should be obtained from the atomic trajectories using some post-processing methods. Stukowski (2012) implemented and compared various methods of defect detection in crystalline materials including energy filtering, centrosymmetry parameter analysis, bond order analysis, Voronoi analysis, adaptive common neighbor analysis, and neighbor distance analysis. Stukowski and his co-workers (Stukowski and Albe, 2010; Stukowski et al., 2012; Stukowski, 2012; Stukowski, 2014) have developed the Crystal Analysis Tool which is able to extract the required dislocation information including the dislocation length, position, and Burgers vector from atomic trajectories. Until now, the
dislocation density or the total length of dislocations are the only variables which have been considered to study the hardening mechanisms in fcc bulk metallic samples (Yaghoobi and Voyiadjis, 2016a; Abraham et al., 2002; Bringa et al., 2006; Buehler et al., 2004; Remington et al., 2006; Chen et al., 2012; Gao et al., 2015; Voyiadjis and Yaghoobi, 2015; Voyiadjis and Yaghoobi, 2016). Accordingly, deeper understanding of dislocation network properties, such as dislocation length distribution, average length of dislocations, and maximum dislocation length should be acquired to fully capture the hardening mechanisms.

One of the most important properties of dislocation network is the dislocation length distribution. In the case of metallic bulk samples and quasi-static strain rates, El-Awady et al. (2009) incorporated the experimental results reported by Mughrabi (1976) and described the probability density function (PDF) of the dislocation length using a Weibull distribution. In the cases of metallic samples of confined volumes with free surfaces, such as pillars, the PDF of the dislocation length changes due to the interaction of dislocations with free surfaces (El-Awady et al., 2009; Voyiadjis and Yaghoobi, 2017). Accordingly, the size effects in pillars can be captured using the modified dislocation length distribution (El-Awady et al., 2009; Voyiadjis and Yaghoobi, 2017). In the cases of bulk metallic samples and high rate deformations, however, the evolution of dislocation length distribution has not been investigated as the strain rate varies.

In this work, the large scale atomistic simulation is incorporated to investigate the hardening mechanism in fcc bulk materials during high rate deformations using the properties of dislocation length distribution. The variations of dislocation network and its characteristic lengths including the average and maximum dislocation length versus the strain rate are quantitatively studied using large scale atomistic simulation. As the strain rate increases, the dislocation density increases to sustain the imposed plastic flow. Accordingly, increasing the dislocation density leads to the reduction of the characteristic lengths of dislocation network. Some characteristic lengths of the dislocation network including the average and maximum dislocation length are investigated to capture the variation of
strength as the strain rate changes. The smaller dislocation characteristic lengths lead to the larger strength. It is due to the fact that larger stresses are required to activate smaller dislocations. Accordingly, increasing the strain rate leads to the material hardening.

### 8.2. Simulation details and methodology

Large scale atomistic simulation of a Ni single crystal cube, as an example of fcc material, during uniaxial compression test is incorporated to study the hardening mechanism in the region of high strain rates ($\dot{\varepsilon} \approx 10^8 \text{ s}^{-1}$). The cube with the dimensions of 1500 Å, 1500 Å, and 1500 Å along [1 1 0], [1 1 2], and [1 1 1] directions, respectively, is generated (Fig.1). The parallel code LAMMPS (Plimpton, 1995) is incorporated to perform the atomistic simulation. The sample consists of around 310,000,000 atoms. The embedded-atom method (EAM) potential developed by Mishin et al. (1999) is used to model the Ni-Ni atomic interaction. The periodic boundary conditions are incorporated for all three directions. Newton’s equations of motion are integrated using the velocity Verlet algorithm with a time step of 1.5 fs. The simulations are performed using NPT ensemble. The sample is relaxed for 100 ps with the temperature increasing from 1 K to 300 K and zero pressure in all directions. Next, it is relaxed for another 100 ps at 300 K and zero pressure in all directions. Finally, the uniaxial compression is conducted along [1 1 1] direction from the sample top and bottom with three constant strain rates of $\dot{\varepsilon}_1 = 2.0 \times 10^8 \text{ s}^{-1}$, $\dot{\varepsilon}_2 = 1.0 \times 10^9 \text{ s}^{-1}$, and $\dot{\varepsilon}_3 = 5.0 \times 10^9 \text{ s}^{-1}$ at 300 K and zero pressure on the lateral boundaries (Fig. 8.8.1).

The Crystal Analysis Tool developed by Stukowski and his co-workers (Stukowski and Albe, 2010; Stukowski et al., 2012; Stukowski, 2012; Stukowski, 2014) is incorporated to extract the dislocation structures from the atomic trajectories, and provide additional information such as dislocation length and Burgers vector. The Crystal Analysis Tool is developed based on the common-neighbor analysis method. The atomic arrangements and structures are initially identified. Next, a pattern matching algorithm groups atoms into so-called clusters. A Delaunay mesh is generated that connects all atoms. The elastic deformation gradient tensor is computed for the constructed
Delaunay mesh. The elastic deformation gradient is multi-valued if a dislocation intersects a tessellation element. Next, trial circuits are built on the interface mesh to define the dislocation lines. The code provides a one-dimensional line representation of the dislocation segments. The dislocation network is visualized and analyzed using the software Paraview (Henderson, 2007). Finally, the dislocation density is obtained by dividing the total dislocation length by the sample volume.

![Graph showing uniaxial compression of a Ni sample](image)

Fig. 8.1. Geometry and orientation of the simulated sample.

### 8.3. Results and discussions

The strain rate $\dot{\varepsilon}$ induced by dislocation movements can be described as follows (Meyers and Chawla, 2009):

$$
\dot{\varepsilon} = \eta b \rho_m \bar{v} + \eta b \dot{\rho}_m \bar{l},
$$

(8.1)

where $\bar{v}$ is the average dislocation velocity, $\rho_m$ is the mobile dislocation density, $\bar{l}$ is the distance associated with the displacement of nucleated dislocations, $\dot{\rho}_m$ is the mobile dislocation nucleation rate, $b$ is the Burgers vector magnitude, and $\eta$ is a microstructure factor on the order of unity. Based on Eq. (8.1), high strain rates can be achieved through the movement of dislocation network with
large $\rho_m$ at subsonic velocities, the movement of a dislocation network with small $\rho_m$ at very high (supersonic) velocities, or the large nucleation rate of mobile dislocations (i.e. large $\dot{\rho}_m$) associated with $\dot{l}$. Gurrutxaga-Lerma et al. (2015) incorporated the three dimensional dislocation dynamics and showed that in the region of high strain rates $\dot{\varepsilon} \approx 10^8 \text{s}^{-1}$, the dislocation velocity changes very slightly by increasing the strain rate. In other words, according to Eq. (8.1), the increase in the strain rate is sustained either by increasing the mobile dislocation density or nucleation rate of mobile dislocations. Remington et al. (2017) proposed an analytical dislocation velocity expression based on the atomistic simulation results reported by Tang et al. (2011) and Deo et al. (2005) as follows:

$$\bar{v} = v_s \left\{ 1 - A \exp \left[ -B \left( \frac{\sigma}{\tau_p} \right)^m \right] \right\}$$

where $v_s$ is the shear wave velocity, $A$, $B$, and $m$ are fitting parameters, and $\tau_p$ is the Peierls-Nabarro stress. Based on Eq. (8.2), the dislocation velocity cannot exceed the shear wave velocity. One should note that the results reported by Deo et al. (2005) are obtained for movement of a single dislocation. In the case of a dislocation interacting with other dislocations, especially in samples with large dislocation density, the average dislocation velocity becomes much smaller.

Fig. 8.2 (a) shows the variation of dislocation density $\rho$ versus the strain $\varepsilon$ for different strain rates of $\dot{\varepsilon}_1$, $\dot{\varepsilon}_2$, and $\dot{\varepsilon}_3$. The dislocation density in all three samples starts from nearly a zero value because all samples are initially defect free. Fig. 8.3 shows the initial stages of dislocation nucleation in the case of $\dot{\varepsilon}_1 = 2.0e8 \text{s}^{-1}$. The majority of dislocations are Shockley partial dislocations with the Burgers vector of $1/6 \langle 1 1 2 \rangle$, which are illustrated with green color in Fig. 8.3. The results show that the cross-slip is the dominant deformation mechanism that increases the pinning point numbers. The dislocations pinned at their ends are then elongated and provide the required mobile dislocation length to sustain the applied strain rate. The output of atomistic simulation is total dislocation density and not the mobile one, and it is nearly impossible to obtain the mobile dislocation density. Accordingly, in order to study the trend of hardening mechanisms, it is assumed that the mobile
dislocation density is a constant share of the total one. In the initial stages of plasticity [Figs. 8.3. (a) and (b)] since the value of the mobile dislocation density is small, large dislocation nucleation rate is required to sustain the applied strain rate that results in a large initial increase of the total dislocation density. As the applied strain rate increases, the required dislocation nucleation rate also increases that leads to the larger dislocation density. Fig. 8.2 (a) shows that after the initial sharp increase in dislocation density, there is a drop in the dislocation content. It is due to the annihilation of dislocations nucleated at the large stresses prior to the softening. Since the samples do not experience those large stresses after softening, a portion of annihilated dislocations cannot be nucleated again. Furthermore, a higher strain rate leads to a higher drop in the dislocation density. It is due to the fact that the stress values experienced before the softening are larger for higher strain rates. Accordingly, a larger portion of annihilated dislocations cannot be further nucleated after softening. The dislocation nucleation rate decreases as the mobile dislocation density increases, which activates the term \( \eta b \rho_m \vec{v} \) to sustain the imposed strain rate [Fig. 8.3. (c)]. The molecular dynamics results show that in the range of high strain rates (\( \dot{\varepsilon} \approx 10^8 \text{s}^{-1} \)), as the strain rate increases, the dislocation density increases. The range of dislocation densities are in the order of \( 10^{17} \text{m}^{-2} \) that is large compared to the quasi-static experiments due to the high strain rates. Fig. 8.2 (b) investigates the variation of stress \( \sigma \) versus the strain \( \varepsilon \) for different strain rates of \( \dot{\varepsilon}_1, \dot{\varepsilon}_2, \) and \( \dot{\varepsilon}_3 \). The initial response is elastic and the sample is defect free. After the initial dislocation nucleation, the stress drops since the movement of nucleated dislocations sustain the applied plastic flow. The results show that the material strength increases as the strain rate increases. In order to study the hardening due to strain rate increase, the dislocation length distribution should be investigated.

El-Awady et al. (2009) incorporated a Weibull distribution to capture the dislocation length distribution in a pillar with the diameter of \( D \) as follows:

\[
 f(\lambda) = \frac{\beta}{\theta} (\frac{\lambda}{\theta})^{\beta - 1} e^{-\left(\frac{\lambda}{\theta}\right)} \text{ for } \lambda \geq 0, \tag{8.3}
\]
where \( f(\lambda) \) is the probability density function of dislocation source length of \( \lambda \), \( \beta > 0 \) is the shape parameter, and \( 0 < \theta < D \). El-Awady et al. (2009) used the experimental data of Mughrabi (1976), which is illustrated in Fig. 8.4, to obtain the parameters of the Weibull distribution. In the case of high rate deformations, Fig. 8.5 shows the PDFs of the dislocation length for the simulated strain rates of \( \dot{\varepsilon}_1, \dot{\varepsilon}_2, \) and \( \dot{\varepsilon}_3 \) and selected strains of 0.21, 0.33, and 0.45 obtained from atomistic simulation. The results show that the PDF of the dislocation length at high strain rates is different from that of the slower rates, and the maximum probability of dislocation length belongs to the very small dislocations. It is due to the activation of cross-slip that is the dominant deformation mechanism at high rate deformations (Fig. 8.3). During the high rate deformations, many small dislocations are activated via the cross-slip that their movement are immediately ceased because the stresses required to activate these sources are very large. Fig. 8.5 shows that as the strain rate increases, the PDF of the dislocation length moves toward the smaller dislocation lengths. To unravel the mechanism that controls the dislocation length distribution, one should investigate the variation of dislocation density versus the strain rate. Fig. 8.2 (a) shows that the dislocation density increases as the strain rate increases which leads to the smaller dislocation lengths (Fig. 8.5). In other words, increasing the dislocation density increases the probability of dislocations colliding with each other that decreases the dislocation lengths. The material hardening as the strain rate increases [Fig 2. (b)] can be qualitatively captured using the results presented in Fig. 8.5 that show that the observed hardening is due to the dislocation length reduction.

In order to quantitatively analyze the dislocation length distribution, some characteristic lengths should be introduced. The most appropriate choice is the average length of mobile dislocations \( \bar{\lambda}_m \). However, from the output of atomistic simulation, it is nearly impossible to obtain \( \bar{\lambda}_m \). On the other hand, two other characteristic lengths that can be introduced are the average dislocation length \( \bar{\lambda} \) and the maximum dislocation length \( \lambda_{\max} \). The stress required to activate a dislocation source has an inverse relation with the dislocation length. Accordingly, considering the
same applied stress, the length of a mobile dislocation is larger than that of an immobile one. Since \( \bar{\lambda} \) includes both mobile and immobile dislocations, therefore \( \bar{\lambda} < \bar{\lambda}_m \). The maximum dislocation length \( \lambda_{\text{max}} \), on the other hand, is the largest dislocation in the sample and \( \lambda_{\text{max}} > \bar{\lambda}_m > \bar{\lambda} \). Instead of studying the average length of mobile dislocations \( \bar{\lambda}_m \), one can study the variation of maximum dislocation length \( \lambda_{\text{max}} \) and the average dislocation length \( \bar{\lambda} \) as the upper and lower bounds of \( \bar{\lambda}_m \). A new term \( \sigma_{\bar{\lambda}} \) should be added to the general definition of material strength to incorporate this hardening mechanism. In the case of pillars that the dislocation length distribution changes because of the dislocation interaction with free surfaces, Parthasarathy et al. (2007) proposed that the resolved shear strength has an inverse linear relation with the mean value of the source length. Parthasarathy et al. (2007) developed the model for the operation of Frank-Read sources in the case of pillars. Here, the dislocation length distribution changes due to the strain rate variation. However, the Frank-Read source operation is not the governing deformation mechanism anymore, and cross-slip controls the plastic deformation (Fig. 8.3). In the case of cross-slip, the smaller dislocations are still harder to be activated. Accordingly, an inverse linear relation between \( \sigma_{\bar{\lambda}} \) and \( \bar{\lambda}_m \) is proposed here which can be described as follows:

\[
\sigma_{\bar{\lambda}} = \frac{\bar{\eta} G b}{\bar{\lambda}_m},
\]

(8.4)

where \( \bar{\eta} \) is a geometrical constant, and \( G \) is the shear modulus. In the case of fcc metals, the majority of the dislocations are Shockley partial dislocations. Accordingly, the Burgers vector of the Shockley partial dislocations can be incorporated in Eq. (8.4). Since the samples are all from Ni, it can be assumed that the Burgers vector for different applied strain rates and strains are similar. Consequently, in the cases of simulated samples, Eq. (8.4) states that the stress has a linear relation with \( 1/\bar{\lambda}_m \). In order to study Eq. (8.4), the applied stress \( \sigma \), maximum dislocation length \( \lambda_{\text{max}} \), and average dislocation length \( \bar{\lambda} \) of the sample subjected to the applied strain rates of \( \dot{\varepsilon}_1 = 2.0 \text{e8 s}^{-1} \) and
\( \dot{\varepsilon}_2 = 1.0 \times 10^9 \text{ s}^{-1} \) are normalized using the results obtained from the sample subjected to the strain rate of \( \dot{\varepsilon}_3 = 5.0 \times 10^9 \text{ s}^{-1} \) for different strain values.

Fig. 8.2. The compressive response of Ni single crystal cube at different strain rates of \( \dot{\varepsilon}_1 = 2.0 \times 10^8 \text{ s}^{-1} \), \( \dot{\varepsilon}_2 = 1.0 \times 10^9 \text{ s}^{-1} \), and \( \dot{\varepsilon}_3 = 5.0 \times 10^9 \text{ s}^{-1} \).

Fig. 8.3. Initial dislocation nucleation and evolution in Ni single crystal cube subjected to the strain rate of \( \dot{\varepsilon}_1 = 2.0 \times 10^8 \text{ s}^{-1} \) at different strains of: (a) \( \varepsilon = 0.012 \) (b) \( \varepsilon = 0.018 \) (c) \( \varepsilon = 0.036 \).

In the cases of \( \dot{\varepsilon}_1 \), Fig. 8.6 shows that \( (\sigma) \dot{\varepsilon}_3 / (\sigma) \dot{\varepsilon}_1 \) is between 1.9-2.6, \( (\bar{\lambda}) \dot{\varepsilon}_3 / (\bar{\lambda}) \dot{\varepsilon}_1 \) is between 1.3-2.1, and \( (\lambda_{\text{max}}) \dot{\varepsilon}_3 / (\lambda_{\text{max}}) \dot{\varepsilon}_1 \) is between 2.3-3.5. The results show that \( (\sigma) \dot{\varepsilon}_3 / (\sigma) \dot{\varepsilon}_1 \) is larger than \( (\bar{\lambda}) \dot{\varepsilon}_3 / (\bar{\lambda}) \dot{\varepsilon}_1 \) and smaller than \( (\lambda_{\text{max}}) \dot{\varepsilon}_3 / (\lambda_{\text{max}}) \dot{\varepsilon}_1 \) for all strain values. In the case of \( \dot{\varepsilon}_2 \), Fig. 8.6 shows that \( (\sigma) \dot{\varepsilon}_3 / (\sigma) \dot{\varepsilon}_2 \) is between 1.3-1.9, \( (\bar{\lambda}) \dot{\varepsilon}_3 / (\bar{\lambda}) \dot{\varepsilon}_2 \) is between 1.2-1.6, and \( (\lambda_{\text{max}}) \dot{\varepsilon}_3 / (\lambda_{\text{max}}) \dot{\varepsilon}_2 \) is between 1.5-2.1. Again, the results show that \( (\sigma) \dot{\varepsilon}_3 / (\sigma) \dot{\varepsilon}_2 \) is larger than \( (\bar{\lambda}) \dot{\varepsilon}_3 / (\bar{\lambda}) \dot{\varepsilon}_2 \) and smaller
than $(\lambda_{\text{max}})\dot{\varepsilon}_2/(\lambda_{\text{max}})\dot{\varepsilon}_3$. The results show that the normalized values of $\sigma$ can be correlated with the normalized values of $1/\bar{\lambda}$ and $1/\lambda_{\text{max}}$, and Eq. (8.4) can capture the results obtained from the atomistic simulation. One should notice that the oscillations in normalized $\lambda_{\text{max}}$ is due to the stochastic nature of $\lambda_{\text{max}}$, while the normalized $\sigma$ and $\bar{\lambda}$ do not exhibit large oscillations.

Fig. 8.4. Probability distribution function of dislocation link-length. The data is experimentally measured for Cu (Mughrabi, 1976) and used by El-Awady et al. (2009) in their DDD calculations.

8.4. Conclusions

The present work investigates the hardening mechanism in fcc metallic structures during high rate deformations using the large scale atomistic simulation both qualitatively and quantitatively. The dislocation network properties including the dislocation length distribution, average length of dislocations, and maximum dislocation length are incorporated to study the
hardening mechanism. First, the dislocation length distribution variation as the strain rate changes is studied during high rate deformations. The results show that the distribution of dislocation length at high strain rates is different from that of the slower rates due to the activation of cross-slip mechanism which induces many small dislocations. As the strain rate increases, the dislocation lengths decreases which qualitatively explains the increase in material strength. In order to quantitatively describe the strength enhancement as the strain rate increases, the variations of two dislocation network properties including the average and maximum dislocation length are studied as the strain rate changes. An inverse linear equation is proposed to capture the relation between the material strength and average length of mobile dislocations. The inverse linear relation shows a good agreement with the atomistic simulation results.

![Graph showing dislocation length distribution](image)

**Fig. 8.5.** Variation of probability distribution function of dislocation length as the strain rate changes for different strain rates of $\dot{\varepsilon}_1 = 2.0 \text{e}8 \text{ s}^{-1}$, $\dot{\varepsilon}_2 = 1.0 \text{e}9 \text{ s}^{-1}$, and $\dot{\varepsilon}_3 = 5.0 \text{e}9 \text{ s}^{-1}$.  

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Fig. 8.6. Variation of the normalized maximum dislocation length \( (\lambda_{\text{max}})_{\hat{e}}/(\lambda_{\text{max}})_{\hat{e}_3} \), normalized stress \( (\sigma)_{\hat{e}_2}/(\sigma)_{\hat{e}_1} \), and normalized average dislocation length \( (\bar{\lambda})_{\hat{e}}/(\bar{\lambda})_{\hat{e}_3} \) versus the strain for the applied strain rates of: (a) \( \hat{e}_1 = 2.0 \text{e}8 \text{ s}^{-1} \) (b) \( \hat{e}_2 = 1.0 \text{e}9 \text{ s}^{-1} \).
CHAPTER 9
CONCLUSIONS

1.1. Basic conclusions

In this dissertation, the size and strain rate effects of fcc metallic samples of confined volumes are investigated using large scale atomistic simulations. Accordingly, a holistic view is presented using large scale Molecular Dynamics that attempts to identify the dependence of the material behavior on the microstructure in fcc metals. The governing mechanisms of size and strain effects are investigated using atomistic simulation for a wide range of sample sizes at the atomic scale subjected to various strain rates. Additional work is needed to couple the identified mechanisms with the continuum models. To do so, the observed hardening mechanisms should be incorporated to develop the microstructural physically based continuum models, which is left for future work.

The outcome of this work will lead to a more accurate understanding of the material behavior in micro and nanosized metallic samples such as thin films and nanolayered systems that are of paramount importance to micro manufacturing. Examples of possible benefits include better understanding, controlling, and accelerating the development in new micro and nanotechnology such as microelectromechanical systems (MEMES), nanocoatings, thin films, nanocomposites, ultrafine grain bulk materials, and multilayer systems. This kind of research will have profound implications in both scientific knowledge and a wide range of technologies in most industries such as in the petrochemical industry, healthcare, conservation of materials and energy, and biology.

In Chapter 1, a review of the experimental and modeling background of size effects in metals are presented. In Chapter 2, the effects of different boundary conditions on the simulation of nanoindentation are investigated using Molecular Dynamics (MD). The MD simulations of nanoindentation on Ni single crystal thin films are conducted using various boundary conditions and thicknesses. The coupling effects of indenter size and boundary conditions are studied using the spherical indenter with different radii. Silicon substrate is used as one of the boundary conditions.
Three different atomic potentials including embedded-atom method (EAM), Tersoff, and Lennard-Jones (LJ) are incorporated to model the atomic interactions of Ni-Ni, Si-Si, and Ni-Si, respectively. The effect of boundary conditions on the elastic behavior of thin films with various thicknesses is investigated. The results show that the effect of boundary conditions on the elastic response is a function of the film thickness and indenter radius, such that as the thickness increases and the indenter radius decreases, the boundary conditions effects become less significant. Next, the nucleation and early evolution of dislocations in the simulated samples is addressed. Three different patterns of dislocation structures are observed which are governed by two mechanisms of indentation and bending. Finally, the incipient plasticity in thin films with different boundary conditions and thicknesses is investigated. The results show that the dislocation structure controls the mean contact pressure at the onset of plasticity.

In Chapter 3, the size effects during nanoindentation in Ni thin films are studied using large scale atomistic simulation. The main focus of this chapter is to evaluate the available theoretical models of size effects during nanoindentation using atomistic simulation. First, the dislocation nucleation and evolution in the simulated samples are studied. In the next step, the plastic zone size is obtained for each sample at several indentation depths incorporating the dislocation visualization. The results show that the plastic zone size divided by the contact radius is not a constant factor and varies as the indentation depth changes. The total length of dislocations located in the plastic zone is measured in the simulated samples and compared to that of the corresponding theoretical models. The results obtained from the atomistic simulation verify the theoretical predictions of the dislocation length. Next, the variation of hardness obtained directly from the molecular dynamics outputs, which is the indentation force over the contact area, is studied. In the case of conical indenter, the theoretical predictions of hardness have been verified using both experiments and simulations, and the current simulation shows the same trend, i.e. the hardness decreases as the indentation depth increases. However, in the cases of flat indenters, the theoretical models have not
been validated using any experiments or simulations. Here, in the cases of flat indenters, the simulation results verify the theoretical predictions of hardness. They show that the hardness increases as the indentation depth increases. The variation of dislocation density as a function of indentation depth is then studied. In the case of nanoindentation experiment, the validity of Taylor hardening model, i.e. the relation between the hardening and dislocation density, which has not been previously studied with full atomistic details, is investigated. Accordingly, the hardness obtained directly from the simulation is compared with the one calculated from the dislocation density and theoretical size effects models.

In Chapter 4, the large scale atomistic simulation is incorporated to investigate the size effects in a nanoscale single crystal Ni thin film during indentation. The results show that the hardness decreases as the dislocation density increases, and the forest hardening model cannot capture the strength size effects during nanoindentation at small length scales. It is observed that the size effects is initially controlled by dislocation nucleation and source exhaustion. As the indentation depth increases, the dislocation length and density increase. Consequently, the number of dislocation sources and their characteristic length increase which decreases the material strength. Finally, increasing the dislocation length and density, the dislocation interaction mechanism also becomes important.

In Chapter 5, the effects of grain boundary (GB) on the sources of size effects are investigated. Up to now, several studies have been conducted to address the role of GBs in size effects from the atomistic point of view. However, a rigorous and comprehensive study which addresses the effects of GB for fcc metals on different governing mechanisms of size effects as the sample length scale varies has not been presented yet. Here, samples with different length scales are studied to capture the role of GB in size effects as the grain size changes. The response of single and bi-crystal Ni thin films with two different sizes are studied during nanoindentation experiment using large scale atomistic simulation. Various symmetric and asymmetric tilt GBs are incorporated to study the
effects of GB geometry on the response of samples during nanoindentation. The sources of size effects are analyzed in each sample using the atomistic information obtained from the simulations. The results show that the size effects mechanism influenced by GBs changes from dislocation nucleation and source exhaustion to the forest hardening mechanism as the grain size increases. In the case of small bi-crystal samples, dislocation nucleation and source exhaustion govern the size effects. The GB contributes to the dislocation nucleation beneath the indenter which reduces the strength of sample by providing required dislocations to sustain the imposed plastic deformation. Also, the GB itself is the source of defects which can affect the sample strength depending on the indentation depth at which the dislocation is nucleated from the grain boundary. Increasing the indentation depth, some of the dislocations are blocked by the GB. However, there is no noticeable additional hardness due to the dislocations blockage by GB. Furthermore, the results show that the coherent twin boundaries shows the best performance during the nanoindentation. In the case of large bi-crystal samples, the GB does not influence the size effects at lower indentation depths where the source exhaustion is the controlling mechanism of size effects. However, at higher indentation depths, the dislocation interaction with GB contributes to the forest hardening mechanism and induces some hardening. It is observed that the dislocations are firstly absorbed by the GB. Increasing the indentation depth, some dislocations start dissociating into the next grain. However, it is observed that more dislocations are nucleated in the upper grain. The results show that the main role of GB at larger length scale is to change the pattern of dislocation structure in a way that the dislocations are piled up near the GB which increases the hardness.

In Chapter 6, the different mechanisms of size effects in fcc metallic samples of confined volumes are studied during high rate compression tests using large scale atomistic simulation. Different mechanisms of size effects, including the dislocation starvation, source exhaustion, and dislocation source length effect are investigated for pillars with different sizes. The results show that the controlling mechanisms of size effects depend only on the pillar size and not on the value of
applied strain. Dislocation starvation is the governing mechanism for very small pillars, i.e. pillars with diameters less than 30 nm. Increasing the pillar size, the dislocation exhaustion mechanism becomes active and there is no more source-limited activations. Next, the average dislocation source length is obtained and compared for pillars with different sizes. The results show that in the case of high rate deformations, the source length does not depend on the sample size, and the related size effects mechanisms are not active anymore. Also, in the case of high rate deformations, there are no size effects for pristine pillars with the diameters larger than 135 nm. In other words, increasing the strain rate decreases the pillar size at which there is no more size effects in the absence of strain gradient. The governing mechanisms of plastic deformation at high strain rate experiments are also different from those of the quasi-static tests. First, the diameter in which the dislocation nucleation at the free surface becomes the dominant mechanism changes from around 200 nm to 30 nm. Next, in the case of the pillars with larger diameters, the plastic deformation is governed by the cross-slip instead of the operation of truncated dislocation sources, which is dominant at slower rates of deformation. In order to study the effects of pillar initial structure on the controlling mechanism of size effects, an initial loading and unloading procedure is conducted on some samples prior to the compression test. In the case of the nanopillars with the height smaller than 45 nm, the results show that the pre-straining does not change the controlling size effects mechanism except for the initial phase of dislocation nucleation. In the case of the pillar with the height of 0.3 \( \mu m \) and diameter of 0.15 \( \mu m \), however, increasing the initial dislocation density leads to the activation of the forest hardening mechanism. In other words, as the strain increases, the dislocation density increases, which activates the mechanism of dislocation interaction with each other and increases the pillar strength.

In metallic samples of confined volumes, the size effects are governed by the characteristics of the dislocation network. However, there are only few studies that quantitatively tried to relate the dislocation network properties to the sample size effects. In Chapter 7, the
dislocation length distribution in pillars with different sizes is investigated during compression test with different strain rates using large scale atomistic simulation. The size and strain rate effects are then investigated using the observed dislocation length distribution. It is shown that in the cases of the studied samples, one should incorporate the largest dislocation length and not the mean dislocation length to capture the size and strain rate effects.

In Chapter 8, the hardening mechanisms in fcc metallic structures during high rate deformations are studied by incorporating the dislocation network properties. The large scale atomistic simulation is used to study the variations of dislocation length distribution and its characteristic lengths as the strain rate changes. First, the dislocation length distributions at different strain rates are studied to qualitatively capture the relation between the material strength and applied strain rate. It is observed that increasing the strain rate decreases the dislocation network lengths. Accordingly, the required stress to activate the dislocation sources increases. Since dislocation movements sustain the imposed plastic flow, higher activation stress leads to material hardening. Furthermore, the results show that the properties of dislocation length distribution at high deformation rates are different from those of lower strain rates due to the activation of cross-slip mechanism at high strain rates. In order to quantitatively describe the relation between the material strength and dislocation network properties as the strain rate varies, the variations of average and maximum dislocation length are investigated in the cases of different applied strain rates. The relation between the material strength and average length of mobile dislocations is captured using an inverse linear equation which shows a good agreement with the atomistic simulation results.

1.2. Future perspectives

The next step will be using the microstructural information obtained from large scale atomistic simulation to evaluate the available continuum theories of size effects. Accordingly, in the case of any inconsistency, the continuum model will be modified according to the observed
mechanisms. In the case of strain gradient plasticity models, one should introduce a length scale. Most of the proposed length scales are phenomenological and have not been developed according to a microstructural-based framework. Based on the results of this work and the observed size and strain rate effects mechanisms, a size and strain rate dependent length scale will be presented. Another future research could be developing a physically based hardening model using the atomistic simulation results which includes different hardening mechanisms. The model can be incorporated in the gradient plasticity framework.

Another aspect of future research will be addressing the coupling effects of temperature with strain rate and size of the sample using large scale atomistic simulation. The study can be conducted in both pillars, metallic samples with free surfaces, and bulk material samples. The obtained results can shed light to the governing mechanisms of hardening in metallic samples. Finally, other crystal structures of bcc and hcp can be addressed using large scale atomistic simulation. Accordingly, the coupling effects of temperature, sample size, and strain rate can be investigated in metallic samples with bcc and hcp structures.
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VITA

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