Microscale Metal Forming: Mesoscopic Size Effect, Extrusion and Molding

Bin Zhang
Louisiana State University and Agricultural and Mechanical College, binattitude@gmail.com
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Bin Zhang
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M.S., Shanghai Jiao Tong University, 2013
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This dissertation is dedicated to my beloved parents and brother
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ABSTRACT

The continuing trend of metallic device and product miniaturization has motivated studies on microscale metal forming technologies. A better understanding of materials’ mechanical response and deformation behavior is of importance for the design and operation of micro metal forming processes. In this dissertation, uniaxial compression testing was conducted on Al ring and pillar specimens with characteristic dimensions at meso to micro scales. The experimental data reveals inadequacies of the existing surface layer model and provides a baseline for delineating deformation mechanisms in micro metal forming operations. Microscale reverse extrusion experiment was carried out on Cu and Al rod specimens with varying average grain sizes. Texture assessment on extruded Cu parts showed the texture components formed at tens of microns scale were consistent with those observed in macro scale extrusion. The grain size effect on both the mechanical response and deformation inhomogeneity was demonstrated and was further elucidated by a detailed comparison between the experimental results and the output of crystal plasticity finite element simulations. Another promising micro metal forming operation, namely microscale compression molding, was conducted on single crystal Al, using a series of rectangular double-punch sets with varying punch width and spacing in between. The characteristic molding pressure was observed to exhibit a significant dependence on both the spacing itself and the ratio of the spacing to the punch width. The molded features were characterized and the phenomenon of incomplete filling was observed and discussed. All these experimental results furnish new and basic knowledge for meso/micro scale metal forming technologies, as well as supplying data against which small scale plasticity theories/models can be tested.
CHAPTER 1. INTRODUCTION

1.1 Introduction

The ongoing trend of device and product miniaturization has motivated studies on micro-manufacturing technologies, which, generally, can be classified into two categories, one is lithography-based fabrication techniques widely adopted in micro-electromechanical system (MEMS) [1], and the other is mechanically-based micro-manufacturing processes. A broad range of mechanically-based fabrication technologies have already been developed, including micro-mechanical machining[2], micro-electrical discharge machining (EDM) [3], and laser beam machining[4] and micro-forming[5]. The first three fabrication technologies are serial in nature, and numerous sequential cuts are involved in the process of formation of a desired shape/structure. Micro-forming is a different approach and it only takes one or a few steps to achieve a desired shape or structure because multiple features can be formed in parallel.

In the context of metal forming, micro-forming techniques are defined to be the forming of parts or structures with at least two dimensions at micro scale[5]. This process presents a promising micro-manufacturing approach for its higher throughput, lower cost, and the good mechanical properties of the formed parts. Figure 1.1 shows some typical micro-parts fabricated by micro-forming processes. These micro-parts/features have been widely used in industrial products, like electronic consumer goods, automobiles, and medical applications like miniaturized robots for minimal invasive surgery or implants, or magnetic resonance imaging[6].

Knowledge, designs, and protocols of macroscale forming have been well-established and widely used. However, they cannot be simply and straightly transferred to micro-forming processes due to the existence of various size effects as the dimensions of tools and formed materials decrease to the sub-mm to micron scales [5]. Size effect has to be considered in the micro-forming process because it can strongly influence the materials’ mechanical properties and
deformation behavior, e.g., flow stress, ductility, anisotropy, and forming limit [7]. In what follows, a short review on microscale mechanical size effects and basic theories explaining the phenomena, as well as progress made in metal micro-forming processes is provided.

![Fig.1.1 (a) mechanical micro-parts (b) electronic part with micro-features][8]

1.2 Grain size effect

Metals and alloys are most common in their polycrystalline form, so effects that their grain size has on their mechanical response is of particular interest. It has long been observed that in conventional metallic materials, the flow stress varies with grain size according to the empirical Hall-Petch relation[9]:

\[
\sigma = \sigma_0 + K d^{-1/2}
\]  

where \( \sigma \) is the flow stress of interest, \( \sigma_0 \) is the lattice friction stress required to move individual dislocations, \( K \) is a material constant, and \( d \) is the average grain diameter.

This empirical relation has later been expanded to cover the strengthening effect of various types of boundaries introduced by plastic deformation[10], which in turn, has led to extensive research on its physical basis and practical application. For instance, many studies focus on
enhancing the mechanical properties of polycrystalline metallic materials by refining their grain size, the range of which is typically from a few microns to several hundred microns [11-14].

The Hall-Petch relation can be explained by dislocation formation and pile-up [15]. Briefly, due to the atom disorder arrangement at grain boundaries, a grain boundary can hinder the dislocation motion by forcing the dislocation to change its motion direction and by causing discontinuity of slip plane. The volume fraction of overall grain boundaries increases as the average grain size decreases, thus the mean travel distance of a dislocation decreases, causing dislocation pile-up at grain boundaries. This finally leads to the increase in strength.

New interaction phenomena between dislocations and grain boundaries have been observed when the grain size reaches to the nanoscale. When grain size is reduced to ~ 40 nm, the Hall-Petch relation still holds, though new alternative plastic deformation mechanisms have been observed, including deformation-induced grain rotation and growth [16-19], grain boundaries sliding [20-22], and dislocation emission and absorption at grain boundaries [23].

However, conventional Hall-Petch relation breaks down when the grain size is smaller than 20 nm whether produced by inert gas condensation [24] or electrodeposition [25], or plastic deformation. A softening effect has been revealed by extensive studies when grain size reaches a critical size, and several different deformation models have been proposed to explain this so-called “inverse” Hall-Petch relation [26-29]. Therefore, the grain size effect is also grain size dependent, and the deformation mechanism shifts from dislocation-mediated plasticity in the coarse-grained material to grain boundary-assisted deformation in the nano-crystalline region.

1.3 External characteristic specimen size effect

Mechanical properties of materials are of special interest in micro metal forming processes because they determine the materials’ mechanical response and deformation behavior, and thus
affect the geometrical accuracy and quality of the micro-formed parts. In the past two decades, flow stress of metallic materials has been measured on specimens with external characteristic sizes ranging from \(~1000 \, \mu m\) to \(~1 \, \mu m\) and below. In addition to grain size effect, it has been revealed that the external characteristic specimen size also has a significant effect on the materials’ flow stress. In what follows, we provide a short summary on both experiments and theories on two separate kinds of mechanical size effects, namely, a “smaller is weaker” trend and a “smaller is stronger” trend.

1.3.1 “Smaller is weaker” and the surface layer model

Uniaxial compression testing was conducted on specimens of copper, CuZn15 and CuSn6 by scaling down the standard upsetting test, and the measured flow stress was observed to decrease with decreasing specimens’ size while the grain size of the specimens was held constant \cite{30}. This effect has also been confirmed in the field of sheet metal forming \cite{31} and other research projects\cite{32}. This “smaller is weaker” effect can be explained, at least qualitatively, by the surface layer model \cite{5}. Briefly, the surface grains in the specimens have less constraints than the interior grains, therefore, the surface grains show a lower flow stress as compared to the inner grains based on the dislocation pile-up mechanism. This softening effect can be negligible at the macroscale, while at the meso/micro scale, it becomes significant as the volume fraction of surface layer grains increases rapidly as external specimen size decreases, which finally results in the observed overall decrease in the flow stress.

1.3.2 “Smaller is stronger” and the dislocation starvation mechanism

Uchic and Nix \cite{33} first conducted uniaxial compression on cylindrical single crystalline Ni micro-pillars fabricated by focused ion beam (FIB), and reported the so-called “smaller is
stronger” trend: in that the yield strengths of these cylindrical Ni pillars increased significantly as micro-pillar diameters decreased, as shown in Fig. 1.2.

Fig. 1.2 (a) Engineering Stress-strain curves from compression of pure Ni micropillars; (b) an SEM image of the deformed Ni micropillar with ~5µm diameter [33].

Since then, extensive studies have been reported on small scale plasticity by conducting compression, tension and torsion on FCC [34-36], BCC [37, 38], and HCP [39, 40] metals and alloys as well as a variety of metallic glasses [41-43], with external specimens sizes at nano- to micro- scales. These published results from both compression and tension of FCC metallic micropillars have been summarized and fitted into the form \( \frac{\tau_{\text{res}}}{\mu} = A \left( \frac{d}{b} \right)^m \), where \( \mu \) is the shear modulus, \( \tau_{\text{res}} \) is the resolved shear stress onto \(<1 1 0>/{1 1 1}\) slip system, \( d \) is the pillar diameter, and \( b \) is the Burgers vector [44, 45]. The fact that the strength of material highly depends on the characteristic specimen size can be explained by the dislocation starvation model. The model hypothesizes that the mobile dislocations inside a small nano/micro pillar have a greater probability of annihilating at a free surface than of interacting with one another, thereby shifting plasticity into a nucleation-controlled regime [46, 47].
1.4 Case studies on bulk micro metal forming

Micro metal forming technologies are of interest because the fabricated metal-based microscale devices possess many advantages that their Si-based counterparts do not have. For example, metal-based micro devices may function better under harsh conditions, like high stresses and high temperature. Various micro forming technologies have been developed to fabricate such micro devices, like micro extrusion [48-52], micro deep drawing [53-55], micro-punching and micro-blanking [56-59], micro imprinting and micro metal molding [60-63]. Here, we summarize progress in micro extrusion and micro metal molding.

1.4.1 Progress in micro extrusion process

Extrusion is a widely used traditional manufacturing technique. At the macro scale, the knowledge, tools, and methods for analysis of materials deformation behaviors have been well-established. At the micro scale, extrusion processes are still of interest because of their relative simplicity in design and operation. Engel and Eckstein [5] have reported the grain size effects by conducting a series of double-cup extrusion, forward rod-backward can extrusion experiments on CuZn15 brass alloy. They focused on grain-size-affected inhomogeneity by evaluating the final extruded parts shape. Results showed that specimens with fine grains produced more backward extrusion than specimens with coarse grains and that coarse-grained specimens tended to deform irregular extruded rim for the backward-extruded part. They also conducted forward extrusion of a 0.5 mm diameter billet with coarse grains and measured the hardness at various locations over a cross section of the extruded part. The maximum hardness was found in specific grains which were not in the expected regions, indicating the specific size, location, and orientation of these grains lead to more severe deformation. Cao et al. [51, 64] have also reported grain-size-affected deformation behavior by conducting micro forward extrusion on CuZn30 specimens with two
initial average grain sizes of 32 µm and 211 µm. As shown in Fig. 1.3 (a), the extruded pins using coarse-grained specimens showed a tendency to curve, while this behavior was not seen in extruded pins with fined-grained specimens. This indicated that the coarse-grained specimens underwent inhomogeneous deformation, and the individual grain size, location, and orientation would have a significant impact on the deformation behavior of the entire work piece. This deformation inhomogeneity was further verified by microstructural examinations (shown in Fig. 1.3 (b)) and micro hardness measurements.

Fig. 1.3 (a) extruded pins using fine-grained and coarse-grained specimens; (b) microstructure examination showed large grains in the center of the coarse-grained extruded pins at the curvature location [64].

Hot extrusion is also of interest because forming temperature affects the quality of the formed parts, for instance, the observed deformation inhomogeneity can be alleviated by increasing the forming temperature [65]. Hot extrusion also helps in forming more complicated features, like micro-gears [66], which is an important actuating component used widely in micro-electromechanical systems (MEMS). Studies [67-69] have reported extruded micro-gears using different kinds of commercial alloys and revealed elevated temperatures can reduce the extrusion pressure and improve the gear surface quality.
1.4.2 Progress in micro metal molding

High-aspect-ratio microscale structures (HARMS) are essential for the fabrication of most metal-based micro devices. One traditional approach to fabricate metal-based HARMS is based on combining deep X-ray/UV lithography (Lithographie) with electrodeposition (Galvaniformung) in the X-ray/UV LiG protocol [70]. Such a microfabrication protocol is unfortunately expensive and slow as compared to direct compression molding with microscale mold inserts, which has been successfully achieved in Al [63], Cu [71], Ni [72], and NiTi [73]. Figure 1.4 (a) shows such a surface coated microscale mold insert that consists of arrays of long, rectangular punches, and the width of each individual punch is ~150 µm. Figure 1.4 (b) shows the molded Cu microchannel arrays.

Fig. 1.4 Coated rectangular mold inserts and molded Cu: (a) a-Si:N coated inconel mold insert; (b) molded Cu microchannel array[72].

The mechanical response of elemental Al [74] and elemental Cu [75] during molding was also analyzed. The average molding pressure, \( p = P/A \), was measured as a function of the molding depth, \( \Delta \), where \( P \) is the total compression force applied on the insert, and \( A \) is the nominal contact area between the insert and the molded material. A simple, one-parameter scaling law were applied to the measured \( p-\Delta \) curves, where the average molding pressure was normalized by the materials’
flow stress $\sigma$ at the corresponding molding temperatures. The scaled $p/\sigma - \Delta$ curves collapsed onto a universal curve, as shown in Fig. 1.5, indicating the compression molding response of Al and Cu at punch width of $\sim$150 $\mu$m could be adequately described by continuum mechanics, a fact which was further verified by the results of finite element analysis (FEA) [76].

![Fig. 1.5 A simple, one parameter scaling of pressure-depth curves from Al molding experiments at different temperatures: (a) $P$-$d$ curves (b) an universal curve after scaling][74]

Chen et al [77] further extended the compression molding into the nano scale using a series of single, long, rectangular strip punches with punch width varying from $\sim$5 to $\sim$0.5 $\mu$m. The mechanical responses of molded single crystalline Al during molding was also analyzed. The molding depth was normalized by the each single punch width, which serves as the characteristic length in the problem. As shown in Figure 1.6, the so-obtained response curves exhibited a significant mechanical size effect, i.e., the characteristic molding pressure increased rapidly as the single punch width decreased to the nanoscale. Further extension of this nano/micro scale single rectangular punch molding experiment into metal molding using inserts with arrays of micro rectangular punches is of interest.
Fig. 1.6  (a) molding response curves in terms of nominal contact pressure vs. normalized molding depth (b) characteristic molding pressure vs. punch width [77].

1.5 Outline of the dissertation

In what follows, Chapter 2 shows the results of uniaxial compression of Al micro rings and Al micro pillar specimens. The data were analyzed in accordance with the surface layer model, and a master curve of Al flow stress as a function of the specimen characteristic external dimension was obtained. Chapter 3 analyzes the texture development and mechanical response of Cu specimens with a constant grain size during the microscale reverse extrusion experiment. The minimum sidewall thickness of the extruded parts reached ~25 μm. Chapter 4 documents the grain size effect during microscale reverse extrusion, and a detailed comparison of experimental results and crystal plasticity finite element (CPFE) simulation outputs was conducted. Chapter 5 demonstrates the compression molding results using both single rectangular punches and double-punches of varying dimensions. Incomplete filling was observed and molding responses were analyzed. Chapter 6 provides a brief summary of this dissertation.

1.6 References


CHAPTER 2. MECHANICAL RESPONSE OF MESOSCOPIC AL RINGS 
AND MICROSCALE AL PILLARS UNDER UNIAXIAL COMPRESSION

2.1 Introduction

Continued demand on device and system miniaturization motivates a continued interest in 
micro-forming technologies for manufacturing metal parts with sub-mm characteristic dimensions 
in the last two decades [1, 2]. Metal parts with characteristic dimensions of hundreds of microns 
and larger, such as springs, screws, and pins, have been fabricated through metal forming 
operations appropriately scaled down from macroscale designs [1, 2]. Metal parts with 
micron/sub-micron characteristic dimensions have been fabricated with lithography-based 
fabrication protocols rooted in Si very-large-scale-integrated circuit technologies [3]. Such 
micron/sub-micron metal parts with shape anisotropy and consequent magnetic anisotropy were 
shown to offer advantages, for instance, as contrast enhancement agents in magnetic resonance 
imaging (MRI) [4]. It is thus of interest to push metal forming technologies from >100 µm in 
characteristic dimension to the ~10 µm and ~1 µm scales.

The mechanical behavior of metals at the sub-mm to micron scales is an important consideration for forming process design and implementation. Over the past two decades, various 
measurements of the flow stress of metals have been performed on specimens with characteristic 
external sizes ranging from ~1000 µm to ~1 µm and below, and strong size effects on the strengths of materials have been observed. For moderately large sample sizes, i.e., in the ~100 µm to ~10 
µm range, size effects have been observed and associated with strain gradients imposed by deformation geometry, such as in torsion of thin wires [5] and bending of thin foils [6]. In wire
torsion and foil bending, it was shown that materials increasingly strengthen as the characteristic specimen dimension decreases [5, 6], due to increase in imposed strain gradients. In the absence of strain gradients, size effects have also been observed in samples with smaller sizes. At diameters ranging from ~40 µm down to ~0.2 µm, axial compression of single crystal cylindrical pillar specimens showed a pronounced increase in their flow stress as the diameter decreases [7, 8]. Such increase in strength has been attributed to a dislocation starvation mechanism, where the nucleation, subsequent propagation, and escape of dislocations carry a dominant influence on plastic deformation.

In contrast to the above two examples of “smaller is stronger”, when polycrystalline cylinders with diameters ranging from ~3000 µm down to ~500 µm were tested under uniaxial compression, the flow stress was reported to decrease with decreasing cylinder diameter with the grain size held constant [1, 2, 9]. In the mesoscopic range of characteristic dimensions between ~400 µm and ~20 µm, data sets on the mechanical response of materials are scarce in the current literature. Further, mechanical response of materials obtained from simple testing geometries with free external specimen surfaces, such as uniaxial compression of free-standing cylinders, may be significantly different from their behavior during actual micro forming operations, in which almost all external surfaces of the material being formed are in contact with the forming tool and are subjected to significant external constraints [10]. Obtaining reliable uniaxial compression response of materials in this meso-range of characteristic specimen dimensions is thus of interest because: first, data obtained may elucidate mechanisms responsible for the transition from a “smaller is weaker” to a “smaller is stronger” behavior; second, knowledge gained from such simple tests with free external surfaces can offer a baseline for better delineation of the importance of external constraints for materials’ response during actual metal microforming operations.
Performing uniaxial compression on “free-standing” cylinders [11] becomes more challenging experimentally as the cylinder diameter decreases below ~500 µm. Unlike compression testing on FIB fabricated micron/sub-micron pillars [12], where the pillars are attached to substrates, the meso scale cylinders tested in micro-forming experiments are “free-standing” in the literal sense, i.e. the top and bottom cylinder surfaces only maintain frictional contacts with the loading platens. Due to difficulties with mechanical alignment and specimen handling, premature deformation instability is more likely to occur as the cylinder diameter decreases below ~500 µm. In a recent study [13], we examined the process of metal forming in a simple axisymmetric reverse extrusion geometry. Cup-shaped elemental Cu parts were formed through reverse extrusion of 3 mm diameter Cu rods placed in a die with a 3 mm diameter hole and compressed with center-aligned cylindrical punches of varying diameters. The characteristic dimension of forming, defined in this case by the sidewall thickness of the extruded axisymmetric cup structure, ranged from ~400 µm down to ~25 µm [13]. When the solid bottom of such an extruded cup is removed, for example by mechanical polishing, free-standing ring-shaped metal specimens are obtained with a fixed outer diameter, $D$, of 3 mm and varying wall thicknesses. Comparing uniaxial compression of “free-standing” metal cylinders with so obtained free-standing metal rings, as the characteristic specimen dimension, in one case defined by the cylinder diameter and the other by the ring wall thickness, decreases to below ~500 µm, it is more difficult to ensure the condition of true axial loading for the cylinder case. The ring-shaped specimens with a fixed outer diameter of 3 mm and decreasing ring wall thickness thus allow uniaxial compression testing to be conducted at decreasing characteristic specimen dimensions while not increasing the difficulty of mechanical alignment, as in the case of solid free-standing cylinders. In the present paper, we report on results of uniaxial compression testing of elemental Al ring-shaped specimens
with \( D = 3 \text{ mm} \), wall thicknesses of \(~400 \mu \text{m}, ~300 \mu \text{m}, \text{ and } ~200 \mu \text{m} \), and the Al grain size held approximately constant. Additional compression tests were performed on polycrystalline Al cylindrical pillar specimens, with diameters ranging from \(~20 \mu \text{m} \) to \(~60 \mu \text{m} \). These experiments were undertaken to obtain reliable size dependent uniaxial compression response data from Al specimens with characteristic specimen dimensions in the mesoscopic range.

2.2 Experimental procedures

2.2.1 Al rings and micro pillar fabrication

An axisymmetric reverse extrusion die-and-punch set was custom designed and built. Commercial Al 1100 alloy (Al 99.9 at.\%+) specimens were machined into solid cylinders, 3.0 mm in diameter and 4.0 mm in height. Prior to further experimentation, all machined Al cylinders were vacuum annealed at \(~10^{-7} \text{Torr} \) at 600 \(^\circ\text{C} \) for 1 hr. Annealed Al cylinders were placed into a nominally 3.0 mm cylindrical hole within a die made of tool steel. Axisymmetric, cup-shaped Al structures were formed through reverse extrusion, by compressing cylindrical rod punches with varying diameters and centered with respect to the die hole, into Al cylinder specimens confined within the die hole. Free-standing Al ring-shaped structures were made from reverse extruded Al cup structures by removing the solid bottom part through mechanical polishing. The nominal outer diameter of the extruded Al cups, and therefore of the ring-shaped Al specimens, was fixed at 3.0 mm. The nominal wall thickness of the ring-shaped specimens, \( t \), decreased from \(~400 \mu \text{m} \) to \(~200 \mu \text{m} \) as the punch diameter increased from 2.20 mm to 2.60 mm. Mechanical polishing of the ring-shaped Al specimens was controlled to achieve a constant ring height, \( H \), to wall thickness, \( t \), ratio, \( H/t \sim 2.5 \) for all specimens. After mechanical polishing, all ring-shaped Al specimens were annealed again in vacuum (\(~10^{-7} \text{Torr} \)) at 500 \(^\circ\text{C} \) for 10 hr to restore grain equilibrium through recrystallization, to eliminate dislocations stored within the specimens due to the severe plastic
deformation associated with the extrusion process, and to ensure similar grain size within all ring-shaped Al specimens prior to uniaxial compression testing. For comparison with the ring-shaped Al specimens, uniaxial compression of Al 1100 solid cylinder specimens, 3.0 mm in diameter and 4.0 mm in height, was also conducted. All machined 3.0 mm diameter Al cylinder specimens were annealed in vacuum (~10^{-7} Torr) at 400 °C for 1 hr prior to compression testing, in order to obtain a similar initial grain size as compared to the annealed ring-shaped Al specimens. Further details on the extrusion setup were provided elsewhere[13]. Figure 2.1(a) shows an SEM image of a typical ring-shaped Al specimen with a wall thickness of ~200 µm, after mechanical polishing and annealing at 500 °C for 10 hr. The top and bottom surfaces of the Al ring were parallel to each other, and perpendicular to the ring axial direction.

Fig. 2.1 (a) SEM image of a typical ring-shaped Al specimen with wall thickness of ~200 µm and height of ~500 µm. (b) SEM image of a typical PFIB fabricated Al pillar with top surface diameter of 24 µm. The images were taken at a 45° tilt.

Al micro-pillars were fabricated from as annealed Al rods by Xe^+ plasma focused ion-beam (PFIB) milling in a Tescan FERA-3 Model GMH Focused Ion Beam Microscope system operated at 30 kV ion beam voltage. Pillar milling was conducted through a series of concentric top-down annular pattern millings with the Xe^+ ion current varied from 100 nA for the initial coarse milling to 10 nA for the final fine milling. The diameters of fabricated micro-pillar were ~20, ~40, and
~60 μm, with height-to-diameter ratios ranging from 1:1 to 1.5:1. The top-down milling led to the existence of a pillar taper angle, which was measured in the SEM to be ~9°. Figure 2.1(b) shows one typical Al pillar fabricated with PFIB, with a top surface diameter of 24 μm and a height of 33 μm. Similar pillar morphologies were observed at other pillar diameters.

2.2.2 Microstructural characterization of Al rings and micro pillars

A FEI Quanta3D FEG dual-beam scanning electron microscope/focused ion beam (SEM/FIB) instrument was used to examine the morphology and microstructure of Al specimens. The SEM/FIB instrument included an X-ray energy dispersive spectroscopy (EDS) attachment (EDAX), and an electron backscatter diffraction (EBSD) attachment (EDAX). Prior to EBSD examination of Al ring and cylinder specimens, mechanical polishing was conducted with SiC polishing papers of various grit sizes, followed by a final vibratory polish with 50nm silica suspension on a GIGA 0900 Vibratory Polisher for 12 hr. EBSD was performed at 30 kV at scan steps of 0.2–0.3 μm. The default instrument definition of the EBSD coordinate system was used, with the specimen normal direction (ND) perpendicular to the polished surface being mapped, the rolling direction (RD) contained within the plane defined by ND and the electron beam direction, and the transverse direction (TD) perpendicular to both ND and RD, forming a right-handed Cartesian coordinate system. Analysis of raw EBSD data, including inverse-pole-figure orientation maps, pole figures (PFs), orientation distribution functions (ODFs), and grain size statistics, was carried out using the TSL OIM software [13].

2.2.3 Uniaxial compression on Al rings and micro pillars

Uniaxial compression testing on annealed Al ring and cylinder specimens began by specimen placement between a pair of 52100 steel rod platens. The bottom rod platen was fixed into a snugly-fitting blind hole on a bottom 4142 alloy steel plate. The top rod platen was put
through a snugly-fitting, through guiding hole on a top 4142 alloy steel plate. The bottom and top plates were aligned to ensure that specimen compression by the top and bottom rod platens occurred along the cylinder axial direction. The total axial compression force, \( P \), was measured through a MTS load cell with a full range of 25 kN. The axial displacement, \( \Delta \), was measured through an MTS extensometer, with a natural gauge length of 8 mm and an extension range of +/- 1.2 mm. Because of the small specimen length, the extensometer was attached to the sides of the bottom and top steel rod platens. A simple estimate showed that, in the present measurement load range, elastic displacements of the sections of the bottom and top rod platens included in between the extensometer gauge can be neglected, and displacements measured by the extensometer were equated to the specimen displacement. Measured \( P-\Delta \) curves were then converted into true axial compression stress vs. true axial strain (\( \sigma-\epsilon \)) curves. SEM examinations of the ring-shaped specimens after testing showed that no buckling occurred as a result of the axial compression at \( t \) values of \( \sim 400 \mu \text{m} \), \( \sim 300 \mu \text{m} \), and \( \sim 200 \mu \text{m} \). However, buckling-like ring structure collapse was observed at \( t \sim 100 \mu \text{m} \), preventing collection of valid flow stress data at characteristic specimen dimensions below 200 \( \mu \text{m} \) through the present ring compression experimental configuration.

Uniaxial compression on Al pillars was carried out on a Nanoindenter XP System (MTS Systems Corp., Knoxville, TN) with nominal load and displacement resolutions of 50 nN and 0.01 nm, respectively. A flat-ended cylindrical diamond punch with a diameter of \( \sim 76 \mu \text{m} \) was used. All Al micro-pillars were compressed under the displacement-controlled mode. For pillars of different height, the displacement rate was varied to achieve a constant engineering strain rate of \( 2.5 \times 10^{-4} \text{ s}^{-1} \). The diamond punch – pillar alignment was achieved with the help of an optical microscope attached to the XP system and a tilt-adjustable specimen stage.
2.3 Experimental results

2.3.1 Grain morphology and size examination

To examine the grain morphology and size, the Al ring specimen was further mechanically polished in a plane parallel to its axial direction until half of the ring was removed. Following a final vibratory polish, one section of the cross-sectional ring surface was examined with EBSD, as shown schematically in Figure 2.2(a). Figure 2.2(b) shows the inverse-pole-figure orientation map from the EBSD mapping area of Al ring cross section, indicated in the schematic of Figure 2.2(a).

![Fig. 2.2](image)

Fig. 2.2 (a) a cross sectional schematic drawing of the as-annealed Al ring specimen after mechanical polishing, with the approximate EBSD examination area indicated; (b) the Al grain morphology as indicated by the EBSD inverse-pole orientation map from the corresponding mapping area indicated in (a). (c) EBSD inverse-pole orientation map from the polished cross section of an as-annealed Al ring specimen with a wall thickness of 300µm and a height of 750µm; (d) EBSD inverse-pole orientation map from the polished cross section of an as-annealed Al ring specimen with a wall thickness of 400µm and a height of 1000µm. (e) EBSD inverse-pole orientation map on 3 mm diameter Al cylinder specimens after an anneal at 400 °C for 1 hr

The 500 °C annealing led to recrystallization within the as-extruded cup sidewall and grain growth from the micron sized fibrous grains developed due to severe plastic deformation due to extrusion[13]. Analysis of data shown in Figure 2.2(b) yielded an average Al grain size of 26.4 µm, with actual sizes ranging from ~10 µm to ~50 µm. Figures 2.2(c) and 2.2(d) show EBSD
inverse-pole-figure orientation maps from two ring-shaped Al specimens with \( t \) of ~300 \( \mu \text{m} \) and ~400 \( \mu \text{m} \), respectively. Both Al ring specimens were annealed at 500 °C for 10 hr. The average Al grain size obtained from Figs. 2(a) and 2(b) were 27.2 \( \mu \text{m} \) and 25.3 \( \mu \text{m} \), respectively. In both cases, actual Al grain sizes ranged from ~10 \( \mu \text{m} \) to ~50 \( \mu \text{m} \). Figure 2.2(e) shows EBSD observation made on 3 mm diameter Al cylinder specimens after an anneal at 400 °C for 1 hr, showing an average Al grain size of ~25 \( \mu \text{m} \) and actual grain size ranging from ~10 \( \mu \text{m} \) to ~50 \( \mu \text{m} \). Results shown in Figure 2.1 and 2.2 indicate that the present Al rod specimens, ring-shaped specimens and micro pillar specimens have similar initial grain size and morphology.

### 2.3.2 Shift correction on compression responses

Figure 2.3(a) shows the results of uniaxial compression testing on Al ring and cylinder specimens. The Al specimens were denoted by their characteristic dimensions, wall thicknesses for ring specimens and diameters for cylinder specimens. For each specimen designation, raw P-\( \Delta \) data were obtained from 6 nominally identical specimens and converted into \( \sigma-\epsilon \) curves. Each curve shown in Figure 2.3(a) represents the collection of all data points from the 6 specimens in the group, averaged within a strain range of \( 10^{-3} \), i.e., all data points with strains falling within 0 to \( 1\times10^{-3} \), \( 1\times10^{-3} \) to \( 2\times10^{-3} \), etc., were averaged together. Visually, the four curves show a clear decrease of the compression flow stress as the characteristic specimen dimension decreases from ~3000 \( \mu \text{m} \) to ~400 \( \mu \text{m} \), ~300 \( \mu \text{m} \), and ~200 \( \mu \text{m} \).

Variabilities existed in contacts between the ring specimens and the steel platens. Such geometrically non-perfect contacts resulted in horizontal shifts (\( \epsilon_0 \)) of the \( \sigma-\epsilon \) curves, as evident in Figure 2.3(a). For instance, \( \sigma_{200} \) and \( \sigma_{300} \) show very similar responses at the initial stage of loading (\( \epsilon < 0.05 \)), which then diverge in the later stage (\( \epsilon > 0.05 \)). On the other hand, \( \sigma_{400} \) and \( \sigma_{3000} \) grow closer in the later stage. Such imperfect contacts also resulted in the slope of the initial
portion of the measured $\sigma$-$\varepsilon$ curves being significantly lower than that corresponding to the expected elastic modulus for Al. To account for such mechanical misalignment effects, the early “elastic” portions of the $\sigma$-$\varepsilon$ curves were ignored, and later stages of the $\sigma$-$\varepsilon$ curves, in the strain range of $0.05 \leq \varepsilon \leq 0.33$, were fitted to the function

$$\sigma = K (\varepsilon - \varepsilon_0)^n. \quad (1)$$

Fig. 2.3 Uniaxial compression testing of Al ring and cylinder Al specimens: (a) an average of all data points obtained from 6 nominally identical specimens from one group; (b) power-law fits to the data shown in (a).

This fit yielded a varying $K$, 153-174MPa, and a relatively constant exponent $n$ between 0.262 and 0.270, with an average $\bar{n}$ of 0.266. Shown in Figure 2.3(b), a new fit with the constraint of $n = \bar{n}$ was then performed, which yielded $K$ values ranging from 153.6MPa to 174.3Pa. Both fitting schemes generated satisfactory goodness of fit, with $R^2 > 0.999$. The true, shift corrected, plastic behavior of the Al ring specimens was taken as

$$\sigma = Ke^{0.266}. \quad (2)$$

2.3.3 Correction for system compliance and pillar taper angle

The measured force-displacement ($P$-$\Delta$) curves obtained from Al micro pillar compression include system stiffness contribution. To determine this system stiffness contribution, in separate
experiments, the flat-ended diamond punch was indented directly onto the flat part of an Al specimen surface. Figure 2.4 shows a typical measured system compliance curve. This measured compliance was subtracted from the measured raw $P-\Delta$ curves obtained from all Al micro-pillars.

![System compliance measurement](image)

**Fig. 2.4** System compliance measurement

![Compression stress – engineering strain curves](image)

**Fig. 2.5** Compression stress – engineering strain curves for Al micro-pillars of varying diameters

Because of the existence of a taper angle on PFIB Al pillars, it is difficult to calculate the true strain from raw pillar compression force – displacement curves. The engineering strains were therefore calculated based on the initial pillars heights, as measured from the SEM images. Pillar compression stresses were calculated based on the top cross-sectional area of the pillar before
testing and on the already obtained engineering strain. Figure 2.5 shows the so-obtained compression stress – engineering strain curves for Al micro-pillars of various diameters.

Due to the existence of taper in PFIB Al pillars, the pillar top cross sectional area is the smallest, and thus the calculated stress based on it is higher than the actual stress experienced by the pillar. To correct for the effect of the taper angle, a continuum plasticity finite element analysis (FEA) was carried out using the commercial ABAQUS FEA package. The Young’s modulus and Poisson’s ratio of Al were taken respectively as its bulk values, $E = 70$ GPa and $\nu = 0.33$. The pillar geometry was built to be the same as the Al pillar shown in Figure 2.1(b), namely, a top diameter of $24 \, \mu m$, a height of $33 \, \mu m$, and a taper angle of $9^\circ$. As the true mechanical response of the Al micro-pillar is unknown, the input material behavior to the FEA was taken as the measured plastic response of the 3.00 mm diameter Al rod specimen, shown as $\lambda = 1$ in Figure 2.6(a). Then this measured input flow stress was scaled down by a factor of 2 and scaled up by a factor of 5, shown respectively as $\lambda = 0.5$ and $\lambda = 5$ in Figure 2.6(a), to capture the effect of variation in the pillar response, unknown. Figure 2.6(b) displays the FEA outputs. The stresses calculated by dividing the compression force by either the pillar top cross section area or the average cross section area are denoted respectively as $\sigma_{top}$ and $\sigma_{avg}$. With scaling factors of $\lambda = 0.5$, 1.0, and 5.0, the FEA generated $\sigma_{avg}$ values, based on the average area, show good agreement with the corresponding input stress – strain curves, while the $\sigma_{top}$ values are significantly higher at the same engineering strain.
Fig. 2.6 FEA of compression of tapered pillars: (a) flow stress vs. plastic strain; (b) FEA outputs

Fig. 2.7 Plot of ratio of $\sigma_{\text{top}}/\sigma_{\text{avg}}$ vs. engineering strain

The ratio of $\sigma_{\text{top}}$ and $\sigma_{\text{avg}}$ is plotted as a function of the engineering strain, as shown in Fig. 2.7. As engineering strain varies from 0 to 15%, the value of $\sigma_{\text{top}} / \sigma_{\text{avg}}$ varies from 1.46 to 1.32, about 10%. The variation of $\sigma_{\text{top}} / \sigma_{\text{avg}}$ with engineering strain exhibits a similar trend even when $\lambda$ varies significantly, from 0.5 to 5.0. The values of $\sigma_{\text{top}} / \sigma_{\text{avg}}$ at the engineering strain of 10% are respectively 1.349, 1.352, and 1.359 at $\lambda$ values of 0.5, 1.0, and 5.0, with an average of 1.353. Based on the FEA results above, we divide the experimentally obtained Al pillar compression stress, e.g., those shown in Figure 2.5, by 1.353 to arrive at the true pillar compression stress.
2.4 Discussion

The 10% proof stress in compression for the 3 mm diameter Al cylinder specimen and the Al ring specimens is plotted vs. the characteristic specimen dimension. As shown in Figure 2.8, the “smaller is weaker” softening behavior is evident.

![Graph of 10% proof stress in compression vs. characteristic specimen dimension.](image)

Fig. 2.8 10% proof stress in compression vs. the characteristic specimen dimension. The line through the data points is a guide to the eye.

A rationalization of the observed decrease in uniaxial compression flow stress as the characteristic specimen dimension decreases is offered by the surface layer model, which states that a layer next to the specimen free surface possesses a decreased flow stress as compared to that of the specimen interior, taken as the bulk flow stress $\sigma_b$ [1, 14-19]. Figure 2.9 shows schematically a ring-shaped specimen, with the two surface layers of thickness $s$ beneath the ring outer and inner surfaces. Ignoring end effects, the volume fraction of the two surface layers within the ring specimen, $f_s$, is given by

$$f_s = \frac{2s}{t},$$  

where $s$ and $t$ are respectively the surface layer thickness and the wall thickness of the ring.
Fig. 2.9 A schematic illustration of the Al ring specimen and the surface layer model.

To first order, the surface layer model assumes that the stress responses are uniform in the surface layers ($\sigma_s$) and in bulk ($\sigma_b$), respectively. The overall flow stress of the ring can be approximated by the rule of mixture[14]:

$$\sigma_{ring} = (1 - f_s)\sigma_b + f_s\sigma_s. \quad (4)$$

Due to the different magnitudes of plastic deformation in the surface layer and the bulk, a slight load transfer exists at the surface/bulk interfaces, leading to a deviation of $\sigma_{ring}$ from the rule of mixture estimation. However, this deviation is extremely small according to our finite element analysis (FEA), and does not affect the validity of Eq. (4).

Taking the surface layer model literally, we assume that the respective stress responses of the surface layer and the bulk remain unique when different samples are considered, i.e., the values of $\sigma_s$ and $\sigma_b$ are constants within the realm where the surface layer model remains applicable. We identify the bulk behavior with the result of present compression measurement on the 3 mm diameter Al cylinder specimen, i.e., $\sigma_b = \sigma_{3000}$. In particular, the 10% proof stress in compression for $\sigma_{3000}$, and therefore $\sigma_b$, is 94.5 MPa. We further identify the surface layer behavior with the
presently available literature data on compression response of Al pillars with the largest diameter, which is ~ 6 µm [20, 21]. According to this data of Ng and Ngan, the average of 10% proof stress in compression for single crystal and bi-crystal 6 µm diameter Al pillars is 66.7 MPa, which is taken to be the 10% proof stress of $\sigma_s$. Knowing $\sigma_s$ and $\sigma_b$, we then obtained the values of $s$ from measured results for $\sigma_{ring}$, in particular the value of its 10% proof stress in compression, following Eq. (3) to (4). The result of this determination is $s_{200} = 39.4 \mu$m, $s_{300} = 20.6 \mu$m, and $s_{400} = 2.9 \mu$m. The variation of $s$ with respect to the ring wall thickness, $t$, is shown in Figure 2.10(a) (blue solid circles), together with a simple linear fit to the three data points (blue dashed line). This linear fit indicates that $s$ reaches ~55 µm when $t$ decreases to ~110 µm. That is, $2s$ approaches $t$ as $t$ decreases to ~110 µm, and the surface layer volume fraction approaches 1. As the characteristic dimension of the specimen increases beyond ~400 µm, the exact magnitude of the surface layer thickness becomes increasingly less material, as $\sigma_b$ dominates its overall mechanical response. For convenience, we assume that the surface layer thickness approaches a constant in this limit, i.e., $s = 2.9 \mu$m for $t > 400 \mu$m. As such, a complete description of the function $s(t)$ for $t > \sim 110 \mu$m is shown Figure 2.10(a).

It is well acknowledged that the surface grains of a material are subjected to less constraints as compared to internal grains, therefore leading to significantly lower dislocation densities near the grain boundaries as well as within the grain interior [15, 22]. Correspondingly, a common practice of defining the surface layer is to identify the thickness of the surface layer with the average grain size, i.e. $s \sim d$. Accordingly, the surface layer thickness should be identical for samples with identical, uniform grain sizes. However, the present data suggests that thickness of the surface layer $s$ is strongly dependent on the sample size $t$. Although $s$ is comparable to $d$ for $t$ of ~300 µm, $s$ is only around 10% of $d$ for large samples ($t > 400 \mu$m) and exceeds $d$ for smaller
specimens ($t = 200 \mu m$). This contradiction suggests that the classical surface layer model may be inadequate in describing the actual materials behavior in the mesoscale in two regards. First, under the assumption of a unique surface layer mechanical response, the thickness of the surface layer may not be necessarily coupled to the grain size of the sample. This calls for a more definitive way to distinguish the surface layer from the bulk. Second, under the assumption of $s$-$d$ unity, i.e., $s \sim d$, the constitutive behavior of the surface layers may not be unique across samples with different sizes, and thus needs to be better understood. Reconciliation of both aspects rests in better understanding towards the near-surface evolution of dislocation structures and remains to be clarified in future studies combining in situ microscale testing with grain level computational plasticity modeling.

Fig. 2.10 (a) The surface layer thickness as a function of the wall thickness of Al ring-shaped specimens; (b) a “master curve” showing the variation in the 10% proof stress in compression of Al as characteristic specimen dimension varies.

The present Al ring compression experiments left a gap in the compression response of Al in the characteristic specimen dimension range from ~10 $\mu m$ to ~200 $\mu m$. Additional compression experiments were therefore conducted on PFIB fabricated Al micro-pillars with diameters ranging from ~20 $\mu m$ to ~60 $\mu m$. Collecting the present ring compression and pillar compression data together with existing Al pillar compression data in the literature [12, 20, 21, 23-31], Figure 2.10(b)
plots the dependence of the uniaxial compression response of Al, in terms of the 10% proof stress in compression, vs. the characteristic specimen dimension in a “master curve” fashion. For the present compression experiments conducted on Al ring specimens, six data points, corresponding to six compression tests, are shown for each ring wall thickness. The “smaller is stronger” and “smaller is weaker” trends are observed at the two extremes of length scales. At 200 µm and above, due to the softening effect of the surface layers, the flow stress reduces with reducing length scale – smaller is weaker. On the other hand, at 5 µm and below, dimensional reduction leads to significant increase in strength due to the dislocation starvation mechanism – smaller is stronger.

Several comments are in order. First, it should be noted that the exact value of the surface layer thickness, according to the literal interpretation of the surface layer model, is dependent upon the choice of the value of $\sigma_s$. For example, if $\sigma_s$ is taken instead as the average 10% proof stress in compression obtained from the present experiments on ~20 µm diameter Al pillars, ~77 MPa, the surface layer thickness $s$ would then have been 4.6, 32.7, and 62.6 µm at $t = 400$, 300, and 200 µm, respectively. While the $s$ values vary, the same trend that $s$ increases significantly with decreasing $t$ remains. Second, the present results indicate that $f_s$ approaches 100% at characteristic specimen dimensions of ~110 µm (or ~150 µm if $\sigma_s$ is taken to be 77 MPa). This gives an estimated limit of applicability for the surface layer model: the surface layer softening effect no longer dominates the mechanical response as $f_s$ approaches 100%. This is also consistent with the master curve plot of Figure 2.10(b), where the softening trend at 200 µm and above does not seem to continue to the data points at 100 µm and below. In particular, the pillar compression data at pillar diameters of 20 – 60 µm indicate a relatively flat response vs. the external specimen size. Third, as has been pointed out before [20, 31], large scatter in flow stress exists in the transition range of 5 to 100 µm, where the detailed structures of each pillar, whether it is a single crystal, bi-
crystal with different boundary orientation, or poly-crystal, give rise to significantly different measured responses.

2.5 Summary

In summary, the present work extends data on uniaxial compression response of elemental Al to characteristic dimensions below 500µm, and confirms the “smaller is weaker” trend in the mesoscale range of 200 – 500 µm. The present work partially fills in the data gap for the compression response of Al in the mesoscale by bridging the nm to the mm scale, reveals aspects of the surface layer model that require further understanding and clarification, and provides a baseline for delineating deformation mechanisms truly important to metal micro forming operations.

Clarification: The simulation work in 2.3.3 was conducted by Dr. Shao and his student, Shahrior Ahmed.

2.6 References


CHAPTER 3. ASSESSING TEXTURE DEVELOPMENT AND MECHANICAL RESPONSE IN MICROSCALE REVERSE EXTRUSION OF COPPER

3.1 Introduction

The continuing trend of device and product miniaturization has motivated studies on micro engineering [1]. In addition to the design, assembly, and modeling/analysis components, micro engineering encompasses a broad range of fabrication technologies, including micro mechanical machining [2], micro electrical discharge machining [3], laser beam machining [4], and micro forming [5]. While the first three fabrication processes are serial in nature, in which the formation of a desired shape/structure takes numerous sequential cuts to achieve, forming is typically a parallel process in which a desired shape/structure is achieved in one or a few steps. For metal-based microscale shapes/structures with sufficient geometrical simplicity, fabrication by micro forming tends to be of higher throughput and lower cost, and is thus of current research and industrial interest.

In micro forming processes, the presence of various mechanical size effects complicates straightforward extension of macroscale forming designs and protocols down to the sub-mm/micron scales. Such mechanical size effects also make the prediction of forming behavior more difficult. Out of the four elements of a micro forming system, namely materials, processes, tools, and machines [5], materials behavior during forming at the sub-mm to micron scales is a key factor in determining the process design and forming success, and continues to be the subject of current investigation. Micro forming experiments, similar to their macroscale counterparts,
typically involve plastic deformation to large strains. While numerous studies have documented changes in materials’ grain structure and texture due to severe plastic deformation at characteristic specimen sizes ranging from mm to tens of mm [6-9], the modification to materials’ microstructure due to micro forming, e.g., changes in grain size, grain morphology, and texture, at characteristic dimensions typical of micro forming operations, from sub-mm to tens of microns, remains to be studied in more detail. Similarly, the mechanical response of materials during micro forming needs further study. On one hand, the mechanical response of materials at sub-mm scales under simple deformation geometries, e.g., uniaxial compression of cylindrical specimens, has been measured in compression experiments and reported to exhibit softening with decreasing specimen diameter, when the specimen diameter is in the range of mm to several hundred microns and the materials’ grain size is held constant [10]. On the other hand, output from finite element analysis (FEA) modeling of micro forming experiments in more complex deformation geometries, e.g., double cup extrusion, using stress-strain curves obtained from micro compression experiments, does not show good agreement with experimental measurements [11].

In this chapter, a reverse extrusion experiment was conducted on Cu 110 alloy specimens in a simple axisymmetric geometry. A companion continuum plasticity FEA model of the reverse extrusion process was set up, the output of which was compared to results of experimental measurements. We demonstrate reverse extrusion of Cu over a wide range of characteristic dimensions, assess changes to Cu grain size, grain morphology, and texture due to the deformation process, measure the mechanical response associated with the extrusion, and compare the measured mechanical response to output from a continuum plasticity FEA.

3.2 Experimental procedures
A simple axisymmetric reverse extrusion die-and-punch set was custom designed and built. Commercial Cu 110 alloy (Cu 99.9 at.%+) specimens were machined into cylinders, 3.0 mm in diameter and 4.0 mm in height. All machined Cu cylinders were annealed in vacuum (~10^{-7} Torr) at 600 °C for 1 hour prior to further experimentation. Cu cylinders were placed into a nominally 3.0 mm cylindrical hole within a die made of D2 tool steel. Cylindrical rod punches made of 52100 tool steel, with diameters ranging from 2.20 mm to 2.95 mm, were fitted into a segmented punch assembly (Fig. 3.1(a)), which was then placed into a snugly fitting guide hole of an upper 4142 alloy steel plate. This upper plate was aligned with the lower die to ensure that the punch is nominally centered with respect to the 3.0 mm hole within the die (Fig. 3.1(b)). During the reverse extrusion operation, a hydraulically driven actuator compressed the punch assembly and drove the punch into the Cu cylinder placed within the die hole. The speed of punch displacement was set at 0.01mm/s. Figure 3.1(c) shows a cross-sectional schematic of the axisymmetric reverse extrusion process, which produced axisymmetric, cup-shaped Cu parts of varying wall thicknesses. The nominal wall thickness of the extruded part varied with the punch diameter $D$, decreasing from 400 µm to 25 µm as $D$ increases from 2.20 mm to 2.95 mm. The total compression force, $P$, on the punch was measured through a MTS load cell with a full range of 25kN. The total displacement of the actuator, $\Delta$, was measured from readings of the linear variable displacement transducer (LVDT) attached to the actuator. A set of $P – \Delta$ curves were measured as a function of $D$, with multiple measurements performed at each $D$ value.

In order to obtain stress-strain curves as input to the continuum plasticity FEA, uniaxial compression experiments were conducted on annealed Cu 110 cylinders used for the reverse extrusion experiments. Cu cylinder specimens, 3.0 mm in diameter and 4.0 mm in height, were placed between a pair of 52100 tool steel rod platens. The bottom rod platen was fixed into a
snugly fitting blind hole on a bottom 4142 alloy steel plate. The top rod platen was put through a snugly fitting, through guiding hole on a top 4142 alloy steel plate. The bottom and top plates were aligned to ensure that compression of the Cu cylinder specimens by the top and bottom rod platens occurred along the cylinder axial direction. The total compression force was measured through a MTS load cell with a full range of 25kN.

Fig. 3.1 The axisymmetric reverse extrusion setup: (a) the segmented punch assembly with several cylindrical punches shown on the side, (b) the entire die-and-punch set, (c) a schematic cross sectional drawing of the reverse extrusion process. The sidewall thickness of the extruded part decreases with increasing punch diameter D.

In one set of measurements, the displacement was measured through an MTS 632.26F-20 extensometer, with a natural gauge length of 8mm and an extension range of +/- 1.2mm. Because of the small length of the Cu cylinders, the extensometer was attached to the sides of the bottom and top rod platens as shown in Fig. 3.2(a). A simple estimate showed that, in the measurement load range, elastic displacements of the sections of the bottom and top rod platens included in between the extensometer gauge can be neglected, and displacements measured by the extensometer were equated to the specimen displacement. In another set of measurements, the
extensometer was not used and the specimen displacement was taken as that measured from the LVDT attached to the actuator. Results of the two sets of measurements, expressed in terms of true stress – true strain ($\sigma - \varepsilon$) curves, are displayed in Fig. 3.2(b). As shown in Fig. 3.2(b), good agreement exists between multiple measurements in one data set, and between measurements across the two data sets, with and without extensometer use. Measurements to a larger strain range was achieved without use of the extensometer.

Fig. 3.2 Measuring stress-strain behaviour of annealed Cu cylinders in uniaxial compression: (a) the measurement setup; (b) typical uniaxial true stress – true strain curves obtained with and without the use of the extensometer.

A FEI Quanta3D FEG dual-beam scanning electron microscope/focused ion beam (SEM/FIB) instrument was used to examine the morphology and microstructure of extruded Cu parts. The SEM/FIB instrument included a Schottky electron column with a 1.3 nm resolution, a high current Ga$^+$ ion column with a 7 nm resolution and a maximum ion current of 65 nA, an X-ray energy dispersive spectroscopy (EDS) attachment (EDAX), and an electron backscatter diffraction (EBSD) attachment (EDAX). As-annealed Cu cylinders and cup-shaped extruded Cu specimens were mechanically polished, followed by a final ultrasonic vibratory polishing with 50
nm silica suspension on a GIGA 0900 Vibratory Polisher for 12 hours prior to EBSD examinations. EBSD was performed at 30 kV, at a scan step of 0.5 μm for as-annealed specimens and 0.2 μm for as-extruded ones. Polished as-annealed and as-extruded specimens were mounted on a commercial 70° tilt stage. Figure 3.3(a) shows a schematic of the EBSD measurement geometry, together with the instrument definition of the coordinate system, including the specimen normal direction (ND), rolling direction (RD), and transverse direction (TD). By default, ND is perpendicular to the polished surface being mapped; RD is contained within the plane defined by ND and the electron beam direction, and perpendicular to ND. For an extruded cup structures, as illustrated in Fig. 3.3(a), polished specimens were mechanically aligned such that extruded cup sidewalls were parallel to RD. For all EBSD runs, over 85% of the data had confidence index (CI) larger than 0.1. Sample texture information, i.e., pole figures (PFs) and orientation distribution functions (ODFs), was obtained by analyzing raw EBSD data with the TSL OIM software. The harmonic series expansion method contained within the TSL OIM software was used, with a series rank of 16 and a Gaussian smoothing half-width of 0.3°. As shown in Fig. 3.3(b), processing of all EBSD data obtained from extruded cup structures began with a coordinate rotation along RD of 90°, such that ND of the rotated coordinate system is perpendicular to the cup sidewall and pointing to the outside of the cup.

Site-selective, instrumented nanoindentation was carried out using a NanoMechanics NanoFlip device. The NanoFlip was installed into the Quanta3D SEM/FIB instrument, such that indentations were conducted on specimen surfaces observable through SEM imaging before, during, and after indentation. By moving the sample stage on which the specimen was mounted, indents could be placed at the desire locations. Indentations were conducted using a Berkovich
diamond indenter. The Oliver-Pharr indenter tip calibration procedures were followed, using a factory supplied fused silica as the calibration specimen.

Fig. 3.3 Schematics of EBSD measurement geometry and coordinate systems: (a) EBSD measurement and the default coordinate system. The specimen illustrated is an extruded cup structure after polishing; (b) the coordinate system used for EBSD data processing, obtained from the default coordinate system by a 90° rotation with respect to the RD direction.

Fig. 3.4 Site-selective indentation on as-annealed Cu: (a) an SEM image of the 2×5 array of Berkovich indents made on well-polished surface of an as-annealed Cu specimen. A portion of the Berkovich indenter is visible on the lower left corner of the image; (b) measured elastic modulus and hardness values as a function of indent position (indent number).

Figure 3.4(a) shows an example of a 2×5 array of Berkovich indents made on top of a well-polished surface of an as-annealed Cu specimen, made at the same maximum load of 50 mN. The elastic modulus $E$ and hardness $H$ measured from the 10 indents are shown in Fig. 3.4(b), with
averages of \( E = 111 \pm 1 \) GPa and \( H = 0.69 \pm 0.02 \) GPa. The measured value of \( E \) is consistent with the tabulated value for bulk Cu [12].

3.3 FEA procedures

Figure 3.5(a) shows a schematic illustration of the continuum plasticity FEA model for simulating the present axisymmetric reverse extrusion process. The die and the punch were assumed to be rigid, and the Cu was assumed to be deformable. To reduce the computational cost, one half of the entire problem was modeled using the four-node axisymmetric element with reduced integration (CAX4R) in the commercial ABAQUS FEA package. The elastic properties of Cu were taken at their bulk values, \( E \) of 110 GPa and Poisson’s ratio \( \nu \) of 0.34. A series of simulations were carried out, varying the diameter of the punch in exact accordance with the D values used in the experiments. In all simulation runs, the plastic behavior of Cu was kept the same and assumed to be that obtained from the uniaxial compression tests on annealed Cu cylinders, with the \( \sigma - \varepsilon \) curves given in Fig. 3.2(b). Stresses within the elastic-plastic Cu were determined following a large deformation formalism, as implemented within the ABACUS package. Figure 3.5(b) illustrates the finite element mesh for the deformable Cu, together with the rigid punch and die.

A direct comparison between the implicit and the explicit integration schemes were made. Output following the implicit integration scheme showed good stability, while that following the explicit integration scheme exhibited oscillations. The implicit time integration scheme was therefore chosen for all the simulations in the present work. Mesh convergence tests were carried out. Figure 3.5(c) shows one example of mesh convergence test results for the case of the 2.20 mm diameter punch. The numerical results obtained using 750, 1500, 2500, and 3500 elements were compared, with the results using 2500 and 3500 elements being almost equal to each other.
in all cases. A mesh density with 3500 elements were therefore used in all the simulations. Loading rate sensitivity tests were carried out. Simulation runs were carried out at punch velocities of 0.01 mm/sec (actual velocity used in the experiments), 1 mm/sec, and 1000 mm/sec, and the results showed good agreement with each other. To reduce the computational cost, the punch velocity was set to 1000 mm/sec in all simulations.

Fig. 3.5 (a) A schematic of the FEA model of the axisymmetric reverse extrusion process; (b) the initial mesh for Cu (3500 elements); (c) a comparison between FEA simulation outputs with 750, 1500, 2500, and 3500 elements at a punch diameter of 2.2 mm.

The interaction between contacting surfaces is an important part of the FEA model. Because details on factors influencing this interaction, including the magnitude of normal pressure and tangential relative velocities, were not measured from the present reverse extrusion experiments, the Coulomb’s law of friction was assumed in present FEA simulation, which involve a single material’s parameter, namely the friction coefficient $f$. The value of $f$ for all simulations was kept the same and selected to be 0.02, after preliminary simulations with four different $f$ values of 0.0, 0.02, 0.05, and 0.10 were carried out. The reason for selecting $f$ to be 0.02 is detailed further in the results section.
3.4 Results and discussion

3.4.1 Grain morphology and texture evolution examination

Typical morphologies of cup-shaped Cu specimens obtained from reversed extrusion with punches of different diameters are illustrated in Fig. 3.6.

Fig. 3.6 SEM images of Cup-shaped Cu specimens obtained from reverse extrusion: (a) transverse cross-section of a cup-shaped specimen with a sidewall thickness of ~400µm, extruded with a 2.2mm diameter punch; (b) longitudinal cross-section of the specimen shown in (a); (c) longitudinal cross-section of a cup-shaped specimen with a sidewall thickness of ~100µm, extruded with a 2.8mm diameter punch; (d) longitudinal cross-section of a cup-shaped specimen with a sidewall thickness of ~25µm, extruded with a 2.95mm diameter punch. The substance exhibiting varying contrasts in the images is the epoxy used to encapsulate the specimens prior to mechanical polishing.

Figure 3.6(a) shows a transverse cross section SEM image of one such specimen, extruded using a punch of 2.2 mm in diameter. The transverse cross section shows that the thickness of the extruded cup sidewall is ~400 µm, in accordance with the die and punch diameters of 3.0 and 2.2 mm, respectively. The sidewall thickness appears to be relatively uniform along the perimeter. The corresponding longitudinal cross section SEM image, shown in Fig. 3.6(b) and obtained by mechanical polishing of the cup-shaped specimen to approximately the specimen center position, indicates that the extruded cup sidewall is relatively uniform in thickness along the length of the
cup. The cup height of the extruded specimen shown in Figs. 3.6(a) and 3.6(b) exceeds 1600 µm, corresponding to a height to sidewall thickness ratio over 4. Figures 3.6(c) and 3.6(d) show longitudinal cross section SEM images of two additional cup-shaped specimens, extruded using punches of diameters 2.80 mm and 2.95 mm, respectively. In Figs. 3.6(c) and 3.6(d), the cup sidewall heights exceed respectively 1000 µm and 650 µm, corresponding respectively to height to sidewall thickness ratios over 10 and over 25. Non-uniformities in sidewall thicknesses along the cup perimeter were observed at larger punch diameters and smaller extruded cup sidewall thicknesses. For example, sidewall thickness measurements on the specimen extruded using the 2.80 mm diameter punch ranged from 100 to 120 µm. Such sidewall non-uniformities reflect, among other factors, random errors in die hole and punch alignment in the present reverse extrusion setup.

![EBSD orientation maps](image)

Fig. 3.7 EBSD orientation maps of as-annealed and extruded Cu specimens: (a) a typical as-annealed Cu specimen; (b) a polished longitudinal cross section surface on which orientation maps were obtained at different locations; (c), (d), (e), and (f) show respectively orientation maps at locations A, B, C, and D marked in (b).

The EBSD orientation maps shown in Fig. 3.7 illustrate typical Cu grain morphologies. Figure 3.7(a) shows a typical orientation map obtained from an as-annealed Cu specimen. Cu
grains within as-annealed specimens appear to be mostly equiaxed, with an average grain size of ~9 µm and twins present within grains. To illustrate the modification to as-annealed Cu grain morphology due to plastic deformation associated with the reverse extrusion process, EBSD orientation maps were obtained from the polished longitudinal cross section surface of an extruded Cu specimen with a sidewall thickness of ~400 µm (punch diameter 2.2 mm), at locations in the middle of the sidewall along the extrusion direction as illustrated in Fig. 3.7(b). Figures 3.7(c), 3.7(d), 3.7(e), and 3.7(f) show EBSD orientation maps obtained at locations A, B, C, and D marked on Fig. 3.7(b), respectively. At location A (Fig. 3.7(c)) close to the original specimen surface, the material did not undergo severe plastic deformation and the Cu grain structure exhibits similarity to that of the as-annealed specimen. At location B (Fig. 3.7(d)) close to half way up the extruded cup sidewall, all Cu grains are elongated along the extrusion direction. Some fibrous grains go through the entire image, from the bottom to the top. This indicates that this area has undergone severe plastic deformation. Also, the width of the fibrous grain appears to decrease from the centre of the cup sidewall toward the edge, indicating the existence of differential straining in the extruded cup sidewall. Materials closer to the external surfaces in contact with the punch appear to go through a higher amount of plastic straining, while the plastic strain at the centre of the cup sidewall appears to be less. At location C (Fig. 3.7(e)) close to the bottom corner of the extruded cup, the Cu grain structure appears to be elongated along the material flow direction, as shown by the arrow in Fig. 3.7(e). At location D (Fig. 3.7(f)), the amount of Cu grain deformation appears smaller again, consistent with the expectation that the amount of plastic strain at location D should be smaller.
Fig. 3.8 Measured hardness values along the extruded cup sidewall, from location A to D shown in Fig. 3.7(b)

Nanoindentation hardness measurements were conducted from location A on the top of extruded cup sidewall to location D along the straight line in middle of the sidewall, as shown in Fig. 3.7(b). Measured hardness values, shown in Fig. 3.8, ranged from ~1.0 GPa to ~1.6 GPa. First, all measured hardness values are higher than that of the as-annealed Cu specimen, measured at 0.69 GPa. Second, measured hardness increases from location A to location B then decreases from location B to location D. Data shown in Fig. 3.8 corroborate the EBSD data shown in Fig. 3.7, which indicate that the highest amount of plastic straining occurs approximately half way along the extruded cup sidewall. A higher degree of plastic straining at location B results in a higher amount of strain hardening, leading to higher hardness values being measured at location B.

Grain morphology change and texture development due to severe plastic deformation associated with the reverse extrusion process are illustrated first in Fig. 3.9. Figure 3.9(a) shows a longitudinal cross section SEM image of a cup-shaped Cu specimen, extruded with a punch of 2.8 mm diameter with a sidewall thickness of ~100 µm. Figures 3.9(b) and 3.9(c) show EBSD orientation maps obtained from locations marked area 1 and area 2 in Fig. 3.9(a). The approximately 50 µm × 150 µm areas probed by EBSD were in the middle of the extruded cup
sidewall. As shown in Figs. 3.3(b) and 3.9(a) as well as stated in the experimental procedures section, prior to EBSD data processing, we set RD to be parallel to the extrusion direction and ND to be perpendicular to the normal of the extruded cup sidewall, pointing to the cup outside. This is done in analogy to the conventional sheet metal rolling process, where ND is set along the direction of sheet metal compression [13]. Cu grains at the top of Fig. 3.9(b), at locations closest to the original specimen surface, still exhibit similarities to the equiaxed morphology found within as-annealed specimens. Cu grains at the bottom of Fig. 3.9(b), ~80 µm into the original specimen surface, are already elongated. Figure 3.9(c) shows that, in area 2 close to the middle of the cup sidewall, all Cu grains are elongated, forming fibrous structures.

Fig. 3.9 EBSD examination of the texture development due to reverse extrusion: (a) a longitudinal cross section SEM image of an extruded cup sidewall, with a thickness of ~100 µm; (b) an orientation map obtained from area 1 marked in (a); (c) an orientation map obtained from area 2 marked in (a).

Figure 3.10(a) shows (001), (110), and (111) PFs from EBSD data shown in Fig. 3.9(c). Figure 3.10(b) shows the corresponding ODF. Examination of the data shown in Fig. 3.10(b) indicate the presence of strong Copper (\{112\}<11\overline{1}>), Brass (\{110\}<\overline{1}12>), and S
components and a weaker Goss (\(\{110\}<001>\)) component. In the present axisymmetric reverse extrusion experiments, the characteristic dimension for the plastic deformation is defined by the thickness of the extruded cup sidewall. Although this characteristic dimension of deformation is ~100 µm in the present case, the deformation texture components observed in Fig. 3.10(b) are, interestingly, coincident with those observed in macroscale sheet metal rolling, i.e., Copper, Brass, S, and Goss [14].

![Texture development due to reverse extrusion (~100 µm thick sidewall): (a) (001), (110), and (111) pole figures obtained from EBSD data collected from area 2 of the extruded cup sidewall shown in Figs. 9(a) and 9(c); (b) the corresponding ODF. The presence of strong Copper, Brass, and S components and a weaker Goss component are identified by symbols.](image)

Further illustrations of grain morphology change and texture development associated with the reverse extrusion process are shown in Fig. 3.11. Figure 3.11(a) shows a longitudinal cross section SEM image of a section of the sidewall of a cup extruded using a 2.95 mm diameter punch. The nominal sidewall thickness is ~25 µm, and the actual sidewall thickness is measured to be ~30 µm. After mechanical polishing, a portion of the sidewall, outlined with the white rectangular box, was polished with Ga⁺ focused ion beam. In this case, the FIB polishing extended through the entire sidewall thickness, from the external sidewall surface to the internal. The EBSD orientation
map of the FIB polished rectangular area is shown in Fig. 3.11(b). Visual inspection suggests again that the grain width is smaller when the grains are near the external and internal sidewall surfaces and larger when the grains are in the center of the cup sidewall.

![Image](image.png)

**Fig. 3.11** EBSD examination of the texture development due to reverse extrusion: (a) a longitudinal cross section SEM image of an extruded cup sidewall, with a thickness of ~30 µm. The entire EBSD mapping area, outlined with the white rectangular box, was polished with Ga+ focused ion beam; (b) The EBSD orientation map of the rectangular mapping area shown in (a). The black arrow shows the location and direction for the misorientation angle measurement shown in (c); (c) a plot of the point-to-point misorientation angle along the path outlined by the black arrow in (b). Structures are highly textured.

To quantify this observation, Fig. 3.11(c) plots the point-to-point misorientation angle obtained from the data shown in Fig. 3.11(b), along the path outlined by the black arrow from point A on the external sidewall surface to point B on the internal sidewall surface. Taking the existence of a grain boundary between two consecutive points probed as being indicated by a misorientation angle in excess of 15°, data shown in Fig. 3.11(c) show that the grain widths along
the path from A to B range from ~0.5 to 5 µm, and that grains with widths closer to 0.5 µm are predominantly located near the external sidewall surface in contact with the die and the internal sidewall surface in contact with the punch.

Recognizing that as-annealed Cu 110 starting specimens contain largely equiaxed grains with a relatively uniform size of ~9 µm, the width of each grain within the extruded cup sidewall can be taken as an indication of the amount of plastic straining undergone by that grain. Thus data shown in Figs. 3.11(b) and 3.11(c) again indicate the existence of differential straining in the extruded cup sidewall. Grains closer to the contacting sidewall surfaces appear to go through a higher amount of plastic straining, while the plastic strain of grains at the center of the cup sidewall appears to be less.

Fig. 3.12 Texture development due to reverse extrusion (~25 µm thick sidewall): (a) (001), (110), and (111) pole figures obtained from EBSD data collected from the area of the extruded cup sidewall shown in Figs. 11(a) and 11(b); (b) the corresponding ODF. The presence of strong Copper and S components and weaker Brass and Goss components are identified by symbols.

Figure 3.12(a) shows (001), (110), and (111) PFs from EBSD data shown in Fig. 11(b). Figure 3.12(b) shows the corresponding ODF. The presence of Copper (\{112\}<11\overline{1}\>) and S (\{123\}<63\overline{4}\>) texture components in the ODF are identified in Fig. 3.12(b), together with intensities close to, but not at the exact, Brass (\{110\}<\overline{1}12\>) and Goss (\{110\}<001\>)
component locations in the Euler angle space. At the characteristic dimension for plastic deformation of ~25 µm, the deformation texture components observed in Fig. 3.12(b) continue to correspond to those observed in macroscale sheet metal rolling, i.e., Copper, Brass, S, and Goss. Development of the same Cu texture components in the present case of axisymmetric reverse extrusion at characteristic dimensions of ~100 µm and ~25 µm suggests that the deformation mechanisms operating in macroscopic sheet metal rolling continues to operate in plastic deformation associated with reverse extrusion at the much smaller characteristic dimensions.

Change in grain morphology in cup shaped Cu 110 specimens offers additional clues to the process of plastic deformation associated with the reverse extrusion geometry. Figures 3.13(a) shows a longitudinal cross section EBSD orientation map of one extruded cup shaped Cu specimen, in a region immediately underneath a punch of 2.95 mm diameter and close to one punch corner. The area mapped by EBSD is fitted into the associated SEM image shown in Fig. 3.13(b) at the right scale and location.

Fig. 3.13 EBSD examination of grain deformation underneath the punch: (a) a longitudinal cross section EBSD orientation map of the region immediately underneath the punch. The black dashed line is a guide to the eye for delineating the boundaries between Cu regions with different degrees of deformation; (b) The longitudinal cross section SEM image of the specimen from which the EBSD orientation map of (a) is taken, together with the orientation map (to scale and at location).
Three distinct regions can be identified from the grain morphologies. Region I immediately underneath the punch show grain morphologies similar to those observed in as-annealed specimens, largely retaining the initial equiaxed grain structure. This indicates that minimal plastic strains are associated with grains in region I. Grains within region II, next to region I, show pronounced elongation along directions consistent with expected materials flow, indicating severe plastic straining. The amount of elongation of grains within region III, next to region II and further away from the sinking punch, appears to be smaller again, indicating reduced amounts of plastic strain of grains in this region. Such observations on grain morphology change suggest that plastic deformation associated with the present reverse extrusion geometry has qualitative similarities to indentation of an elasto-plastic half space by a flat-ended rectangular strip punch. In that instance, continuum plasticity FEA modeling showed the presence of an “elastic enclave” underneath the punch center, limiting plastic flow to the surface to regions around the punch corners [15]. The present grain morphologies observations, especially in region I, is qualitatively consistent with the existence of a similar elastic enclave immediately underneath the punch in the present reverse extrusion geometry. The black dashed lines shown in Figs. 3.13(a) and 3.13(b) serve as an approximate delineation for the boundaries between regions I, II, and III with different degrees of plastic deformation. As shown in Fig. 3.13(b), region II delineated between the two black dashed lines undergoes the highest degree of plastic deformation, and supplies the majority of Cu material making up the extruded cup sidewall.

Figure 3.14 shows overall morphologies of Cu 110 cups obtained from reverse extrusion. The change in overall cup morphology illustrates the transition from homogeneous to inhomogeneous plastic deformation as the characteristic dimension for deformation, for the present experiments the thickness of the extruded cup sidewall, decreases.
Fig. 3.14 SEM images of extruded Cu 110 specimens obtained at a 45 tilt angle: a typical image of extruded cup with sidewall thickness of (a) ~100 µm; (b) ~25 µm.

Figure 3.14(a) shows that, at a sidewall thickness of ~100 µm, the sidewall height is relatively uniform around the cup perimeter and the extruded structure appears to be axisymmetric. In contrast, at a sidewall thickness of ~25 µm, Fig. 3.14(b) shows significant differences in sidewall height around the cup perimeter. In conventional deep drawing of sheet metals, deformation inhomogeneity, manifested in the so called earring formation, is related to macroscale mechanical anisotropy of the starting sheet metal due to its prior processing [16, 17]. In the present case, the starting Cu 110 rod specimens should retain little mechanical anisotropy due to the 600 °C annealing. The observed difference in sidewall height around the cup perimeter, shown in Fig. 3.14(b), is believed to be a result of the inhomogeneous deformation behaviour due to differences in grain size and orientation as a function of position around the extruded cup sidewall. Data shown in Fig. 3.14 suggest that such inhomogeneous deformation behavior becomes apparent when the total number of grains across the sidewall thickness becomes small, ~20 according to data shown in Fig. 3.11(c), as the sidewall thickness decreases to ~25 µm. The transition from homogeneous to inhomogeneous plastic deformation associated with grain size change in small scale metal forming has been observed previously [18], and needs to be better understood and controlled in order to achieve satisfactory final formed shapes at such small scales.
### 3.4.2 Mechanical responses analysis

The average mechanical response of axisymmetric reverse extrusion was measured through the force – displacement curves. A typical raw data set is shown in Fig. 3.15(a), plotting the total compression force $P$ versus the total actuator displacement $\Delta_{\text{total}}$ obtained from a reverse extrusion run with a 2.2 mm diameter punch. Because measured $\Delta_{\text{total}}$ includes a system compliance contribution, $\Delta_{\text{sys}}$, a separate measurement was made by removing the 3.0 mm diameter Cu rod specimen from the die hole and having the punch bottom surface contacting the bottom steel base plate directly, keeping all other components in the system intact. The result of such a system compliance measurement is shown in Fig. 3.15(b), where the measured total displacement is taken to be equal to $\Delta_{\text{sys}}$. Figure 3.15(c) shows the result of subtracting the system compliance contribution from the measured total displacement, i.e., $P$ vs. $\Delta_{\text{total}} - \Delta_{\text{sys}}$. It is noted that the curve shown in Fig. 3.15(c) exhibits an initially more compliant section (outlined by the circle) where $\Delta_{\text{total}} - \Delta_{\text{sys}}$ reaches ~80 µm when $P$ is ~400 N (noted by the arrow). This initially more compliant section is followed by a stiffer section, where $P$ increases more rapidly with increasing $\Delta_{\text{total}} - \Delta_{\text{sys}}$. This stiffer section is followed by a “knee”, after which $P$ again increases more slowly with further increase in $\Delta_{\text{total}} - \Delta_{\text{sys}}$, in an almost linear fashion. Stylus profilometer measurements were conducted on Cu rod specimens after the value of $\Delta_{\text{total}} - \Delta_{\text{sys}}$ reached ~80 µm, which showed that the depths of actual indentation on the top surface of the rod specimens were ~10 µm or less. This discrepancy is believed to result from misfits existing between the Cu rod specimen and the die hole, due to imperfections in die and Cu rod specimen geometries. Measured initial displacements, instead of being from actual punch penetration into the top specimen surface, are rather due to expansion of the rod specimen under compression to fill any misfit gaps between the rod specimen and the die hole. Only after such misfit gaps were filled does $P$ increase more rapidly with increasing actual punch penetration into the top specimen surface. To correct for such misfits, the
initially more compliant section of the curve shown in Fig. 3.15(c) was removed by shifting the origin of the x-axis forward by ~70 µm, as shown in Fig. 3.15(d). The x-axis of the final reverse extrusion mechanical response curve shown in Fig. 3.15(d) is believed to represent actual punch displacement into the top specimen surface, $\Delta$. All other raw $P - \Delta_{\text{total}}$ curves were processed in the same manner as illustrated in Fig. 3.15 to arrive at the actual mechanical response associated with the axisymmetric reverse extrusion, i.e., the $P - \Delta$ curves.

Fig. 3.15 Measuring mechanical response of axisymmetric reverse extrusion: (a) a typical raw $P - \Delta_{\text{total}}$ curve obtained with a 2.2 mm diameter punch; (b) a typical system compliance measurement using the same punch and reverse extrusion components without the Cu rod specimen; (c) compliance subtracted reverse extrusion response; (d) the true reverse extrusion response correcting for initial misfits between the rod specimen and the die hole.

Figure 3.16(a) shows a collection of such $P - \Delta$ curves. A number of repeat measurements were conducted at each punch diameter, and it is apparent that reasonable agreements exist between repeat measurements. As the punch diameter increases from 2.20 mm to 2.95 mm and
the nominal extruded cup sidewall thickness decreases from ~400 µm to ~25 µm, the $P - \Delta$ curves exhibit systematic variations. All the response curves are qualitatively similar, exhibiting an initially stiffer section where $P$ increases more rapidly with increasing $d$, followed by a “knee” after which $P$ increases more slowly with $d$ in an approximately linear fashion. As the punch diameter increases from 2.20 mm to 2.95 mm, the $P - \Delta$ curves show obvious and systematic “stiffening”, i.e., $P$ increases significantly at the same $d$ value as $D$ increases. Figure 3.16(b) shows a collection of simulated $P - \Delta$ curves from the continuum plasticity FEA model.

Fig. 3.16 Measured and simulated mechanical response of axisymmetric reverse extrusion: (a) a set of experimental $P - \Delta$ curves obtained with punches of varying diameters; (b) a set of FEA output $P - \Delta$ curves obtained with punches of varying diameters and a constant friction coefficient of 0.02.

To illustrate the comparison between experimentally measured extrusion response and the corresponding continuum plasticity FEA simulations, Fig. 3.17(a) replots the experimental $P - \Delta$ curves for the case of the 2.20 mm diameter punch together with various FEA output curves assuming different values of the friction coefficient $f$. The FEA outputs included cases with $f$ ranging from 0 to 0.3. $f$ values higher than 0.3, e.g., $f = 0.5$, caused the simulation to stop without progressing to larger $\Delta$ values. It is apparent from Fig. 3.17 (a) that the FEA output curves exhibit the same trend as that observed in the experiments, in all cases exhibiting 1) an initially stiffer
section, 2) a “knee” followed by a less stiff section, with \( P \) still increasing with increasing \( \Delta \). The slope of the portion of the simulated curve after the knee increases with increasing value of \( f \). The FEA output with \( f = 0.02 \) fits best with the experimental \( P - \Delta \) curves. Figures 3.17 (b) and 3.17 (c) show respectively the same experiment – FEA comparison plots for the cases of the 2.60 mm and 2.95 mm diameter punches. For the case of the 2.60 mm diameter punch, the FEA output curves exhibit similar trends as shown in Fig. 3.17 (a). However, the best fit with the experimental \( P - \Delta \) curves is the FEA output with \( f = 0.05 \) instead of \( f = 0.02 \). In Fig. 3.17 (c), only the FEA output curve corresponding to \( f = 0.3 \) is shown, and is seen to lie significantly below the experimental \( P - \Delta \) curves. For punch diameters less than 2.6 mm, the FEA outputs based on \( f \) ranging from 0.02 to 0.05 are in reasonable agreement with the experimental \( P - \Delta \) curves.

It is of interest to note that the FEA output curves corresponding to \( f = 0 \) exhibit a positive slope after the knee, see for example the \( f = 0 \) curves displayed in Figs. 3.17(a) and 3.17 (b) for the cases of the 2.20 mm and 2.60 mm diameter punches. Stated differently, the FEA simulations show that the extrusion forces continue to increase way past the “knee” points on the force – displacement curves even in cases of zero friction. Again referencing the situation of indentation of an elasto-plastic half space by a flat-ended rectangular strip punch, the existence of the “knee” point on the force – displacement curve in that instance signals the formation of a self-similar plastic zone underneath the strip punch. As the punch displacement \( d \) increases past the knee point, this self-similar plastic zone would travel with the strip punch into the indented material in a steady-state fashion. Data shown in Fig. 3.13, indicating the existence of an elastic enclave underneath the reverse extrusion punch, also suggest the existence of a self-similar plastic zone underneath the reverse extrusion punch in the present experiments. Assuming the existence of such a steady-state plastic zone underneath the reverse extrusion punch, the contribution to the
total extrusion force from reaction forces at the plastic zone boundaries underneath the punch is expected to be a constant, in keeping with the steady-state assumption.

Fig. 3.17 Comparing FEA output with experimental reverse extrusion response curves: experimental $P$–$\Delta$ curves plotted together with FEA output with different values of friction coefficient for the case of (a) 2.20 mm diameter punch; (b) 2.60 mm diameter punch; (c) 2.95 mm diameter punch.

However, the present experimental results suggest that the Cu grains along the extruded cup sidewalls may not have reached steady state. For example, the Cu grain morphologies shown in Fig. 3.7 suggest that the plastic strain increases from point D to point B along the cup sidewall shown in Fig. 3.7(b), with corroboration from the site-selective indentation data shown in Fig. 3.8. Recognizing that the Cu material exhibits significant strain hardening, it can be argued that the
contribution to the total extrusion force from plastic flow of the materials along the extruded cup sidewall would keep increasing as long as a portion of the sidewall materials are being increasingly strained plastically, even in the case of zero friction. With non-zero friction, the FEA outputs shown in Fig. 3.17 exhibit increasing slope of the $P - \Delta$ curve past the knee point with increasing $f$ value, consistent with the expectation that sidewall friction would further contribute to the total extrusion force. Whether and when the materials making up the extruded cup sidewall will reach steady state are of interest, and remain to be answered through future studies.

The present axisymmetric reverse extrusion experiments do not provide all necessary input parameters to the continuum plasticity FEA. In one extrusion geometry, although an average extrusion pressure is given by $P/(\pi D^2/4)$ and known from experimentation, actual contact pressures at the surfaces of the punch and the die hole could not be obtained from the present experimental setup. Furthermore, the experiment did not determine the actual friction coefficient $f$, and whether it varied with location along the extruded cup sidewall or as the sidewall thickness of the extruded cup decreased. Given these considerations, Fig. 3.15(b) shows a collection of simulated $P - \Delta$ curves from the continuum plasticity FEA model, assuming a constant $f$ value of 0.02 independent of punch diameter. As shown in Fig. 3.17(a), the choice of $f = 0.02$ was based on the best fit between the experimental $P - \Delta$ curves and the FEA output in the case of $D = 2.2$ mm. In the absence of further information, the assumption of a constant friction coefficient was made. Qualitatively, it is apparent that the FEA output exhibits the same trend as that observed in the reverse extrusion experiments, in all cases exhibiting 1) an initially stiffer section, 2) a “knee” followed by a less stiff and approximately linearly increasing section. Quantitatively, Fig. 3.17(c) indicates that the experimental $P - \Delta$ curves lies significantly above the FEA outputs, and agreement between experimental $P - \Delta$ curves and FEA outputs cannot be obtained as the extruded
cup sidewall thickness decreases to ~25 µm, even with a 15-fold increase in \( f \) value from 0.02 to 0.3.

Besides friction considerations, the existence of inhomogeneous plastic strain within the extruded cup sidewall, as suggested by the grain morphology observations shown in Figs. 3.7 and 3.11, implies the existence of plastic strain gradients across the sidewall thickness and potential strain gradient hardening of the Cu being extruded\[19, 20\]. This implies a potential change in the actual \( \sigma - \varepsilon \) relationship for the reverse extrusion from that obtained from the uniaxial compression experiment shown in Fig. 3.2(b). Such a change would also run contrary to the assumed constant \( \sigma - \varepsilon \) relation in the entire collection of FEA simulations, and contribute to further discrepancies between FEA outputs and experimental measurements. The extent of strain gradient contribution to the total extrusion force remains to be determined through future studies. Lastly, the continuum plasticity FEA does not take the influence of crystal texture on material flow behavior into account. While the present comparison between experimental \( P - \Delta \) measurements and continuum plasticity FEA outputs serves to establish a baseline for further experiment – model comparisons, better agreement may be expected by incorporating crystal texture and strain gradient effects into future modeling efforts.

3.5 Summary

Cup-shaped Cu structures were formed by axisymmetric reverse extrusion, with cup wall thicknesses ranging from ~400 µm down to ~25 µm. The severe plastic deformation associated with the reverse extrusion process led to significant changes in Cu grain morphology, grain size, and texture along sidewalls of extruded cups. While the characteristic dimension for the present reverse extrusion experiments began at ~400 µm and decreased down to ~25 µm, pole figure and orientation distribution function analysis of EBSD data observed the presence of Copper, S, Brass,
and Goss texture components, the same as those observed during macroscale sheet metal rolling. This suggests that the deformation mechanisms operating in macroscale rolling deformation continues to operate in reverse extrusion down to a characteristic dimension of ~25 µm. Observation of Cu grain morphology at different locations along the extruded cup sidewall indicates that the amount of plastic strain is the highest in the middle of the sidewall and decreases toward the cup rim and the base. This observation is corroborated by site-specific nanoindentation hardness measurements. Grain morphology observations further suggest the presence of plastic strain gradients across the thickness of extruded cup sidewalls. Extrusion force – punch displacement curves were measured as a function of extruded cup sidewall thickness, and compared to outputs of a continuum plasticity FEA in corresponding geometries. While the FEA model generates $P - \Delta$ curves qualitatively similar to those observed in experiments, detailed comparison showed that experimentally measured extrusion force significantly exceeded the FEA prediction at the small sidewall thickness of ~25 µm. A transition from homogeneous to inhomogeneous deformation was observed, manifested as significant differences in the sidewall height around the sidewall perimeter, as the characteristic dimension of deformation decreases to ~25 µm, leading to the presence of ~20 grains across the extruded cup sidewall thickness. The present work on small scale axisymmetric reverse extrusion suggests that the effects of friction, crystal texture, and strain gradients should be further explored in the future in order to bring improved correspondence between modeling and experimentation.

Clarification: The FEA simulation in this chapter was conducted by Dr. Voyiadjis and his student, Dr. Song.

3.6 References


CHAPTER 4. GRAIN-SIZE AFFECTED MECHANICAL RESPONSE AND DEFORMATION BEHAVIOR IN MICROSCALE REVERSE EXTRUSION

4.1 Introduction

With the ongoing trend of product and device miniaturization, there exists a growing demand for metallic micro parts, examples include mechanical micro connector pins, miniature contact springs, and electronic parts with micro features [1]. These micro parts have already been widely used in industries such as automobiles, electronics, etc. Different functional metallic micro parts can be produced using a variety of manufacturing processes, including micro machining [2-4] and micro forming [5, 6]. Micro forming is defined to include forming processes to produce parts with at least two characteristic dimensions at the sub-mm scale, and has the advantages of low-cost, high-throughput, clean surface finish, and good mechanical properties of the formed parts [7]. Often, micro forming has been executed by scaling down the macroscale forming processes that have been well established. However, the existence of various mechanical size effects impedes the straight forward transfer of the knowledge gained in the macroscale forming domain to forming at characteristic dimensions from hundreds of microns and beyond to tens of microns and below. For example, the deformation behavior of metals at sub-mm to micron scales shows a significant and duplex dependence on the external specimen size. Uniaxial compression of ring and pillar specimens of Al revealed that its flow stress exhibits a “smaller is weaker” behavior and a “smaller is stronger” behavior as the characteristic specimen dimension decreases from ~1000 µm to ~1 µm and below, at a relatively constant grain size [8].

In recent years, extensive studies have been conducted to investigate the materials’ deformation behavior during various micro forming processes, including micro extrusion [9-13], micro deep drawing [14-16], micro-punching and micro-blanking [17-20], micro imprinting and micro molding [21-25]. This paper focuses on micro extrusion because of its relative simplicity.
in design and operation and because it exemplifies basic materials issues present in meso to micro scale metal forming. In our previous work, we conducted axisymmetric reverse extrusion of Cu, obtained cup-shaped extruded Cu parts with sidewall thicknesses ranging from ~400 µm down to ~25 µm, and analyzed the texture evolution and mechanical responses with the initial grain size of Cu held approximately constant [26]. A transition from homogeneous to inhomogeneous deformation was observed as the sidewall thickness of the extruded cup structure decreased, to the point that a relatively small number of Cu grains were present across the cup sidewall. This motivated us to further investigate the influence of microstructure of the material being formed on the mechanical response and deformation behavior during micro forming. Cao et al [12, 27] reported grain-size-affected deformation behavior by conducting micro forward extrusion on CuZn30 specimens. The specimens before extrusion had two different average grain sizes, ~30 µm and ~200 µm. The pins extruded using coarse-grained specimens showed a tendency to curve, towards different directions and in different degrees of severity. In contrast, this behavior was not observed in pins extruded with fine-grained specimens. This indicated that the coarse-grained specimens underwent inhomogeneous deformation, consistent with results from Engel and Eckstein [5]. Most studies available in the literature on micro extrusion [9-13] focused on the grain-size affected deformation inhomogeneity, while few examined the grain-size dependent mechanical response. The paucity of data on the latter may stem from the increased difficulty to obtain accurate mechanical response curves at smaller specimen dimensions.

It is well-known that the yield strength of metals obeys the Hall-Petch relation [28]: the yield strength is proportional to the inverse square root of the average grain diameter, \(d\). This relation is valid for grain diameter from the mm scale to the sub-micron scale. However, in most micro forming geometries, the relation between mechanical response and initial grain size of the
material being formed is more complex. Unlike mechanical testing in simple geometries, e.g., uniaxial tension/compression, where the majority of external specimen surfaces is free and unconstrained, in metal forming operations, e.g., the present axisymmetric reverse extrusion geometry, almost all the surfaces of the material being formed are constrained between the die and the punch. To examine this matter in more detail, in this paper, a microscale axisymmetric reverse extrusion experiment was conducted on both Cu 110 alloy and Al 1100 alloy, each with three distinctly different average grain sizes. Deformation behaviors and mechanical response curves, i.e., total compression force vs. punch penetration depth, were obtained and analyzed. Accompanying crystal plasticity finite element (CPFE) simulations were also performed to provide additional insight on the respective effects of grain size and initial texture on the mechanical response and shape of the extruded part. The present work demonstrates grain size effects in micro forming and provides baseline data for future work.

In what follows, Section 2 describes procedures for experimentation and CPFE simulations; Section 3 presents major results and discussion; and Section 4 gives a brief summary.

4.2 Procedures and methodology

4.2.1 Experimental procedures

The equal-channel angular pressing (ECAP) protocol presents an efficient method for grain refinement by imposing severe plastic deformation on the workpiece.

Figures 4.1(a) and (b) show respectively an optical image of a custom designed and constructed ECAP device and a schematic illustration of its operation. The die, made of 34CrNiMo6 alloy steel, has two cylindrical channels with the same diameter of 3.0 mm, a corner angle $\Psi$ of 36.9°, and an intersection angle between the two channels $\Phi$ of 90° [29]. As-received commercial Cu 110 alloy (Cu 99.9 at.%+) and Al 1100 alloy (Al 99.9 at.%+) specimens were
machined into cylindrical rods, 3.0 mm in diameter and ~30 mm in length. All rods were ECAP processed by being pressed by a cylindrical punch made of high speed steel with a close sliding fit to the die channel through the ECAP device, either 1 time or 8 times (1 pass or 8 passes). For multiple ECAP passes, the rod was rotated by 90° (processing route Bc as described in [29]) in the same direction between each pass. Prior to each pass, the die was lubricated with “Rhenus SU 200A” deep drawing oil. Then, the ECAP processed rods were cut into specimens with heights of 3.0 mm, while keeping the diameter at 3.0 mm.

Fig. 4.1 (a) An optical image of a custom designed and constructed ECAP device; (b) a schematic illustration of the ECAP process.

The ECAP processed Cu and Al rod specimens were vacuum annealed in a tube furnace evacuated by a turbomolecular pump backed by a mechanical vane pump. The base pressure of the tube furnace was ~10⁻⁷ Torr. Annealing occurred at different temperatures and durations to obtain various final grain sizes.

Uniaxial compression testing of ECAP processed and annealed 3 mm diameter rod specimens was conducted on a hydraulically driven MTS 858 uniaxial testing frame. True stress
true strain (\(\sigma - \varepsilon\)) curves were obtained by compressing rod specimens between one pair of steel platens. Further details on the protocol for uniaxial compression testing has been reported elsewhere [8, 26].  

Axisymmetric reverse extrusion was conducted on ECAP processed and annealed Cu and Al rod specimens, 3 mm in diameter, using a previously designed and built die-and-punch set. Further details on the design and construction of the extrusion setup were also described previously [26]. Cu and Al cylinders were placed into a nominally 3.0 mm diameter, blind, cylindrical hole within a die. During reverse extrusion, cylindrical rod punches with diameters of 2.60 mm and 2.80 mm were centered with respect to the die hole and compressed into Cu and Al cylinder specimens. Additional extrusion runs were conducted on Cu rod specimens with an initial average grain size of \(~8 \mu\text{m}\), using 2.2 mm and 2.95 mm diameter punches. Cup-shaped parts resulted from the reverse extrusion process, the sidewall thickness, \(t\), varied with the punch diameter, \(D\).  
The reverse extrusion was conducted on the same hydraulically driven MTS 858 system. Load control was used for all extrusion experiments, at a loading rate of 10N/s and an unloading time of 100 s. Typical total run time, including loading and unloading, was \(~1000\) s. In all extrusion runs, no lubricant was used and dry contact occurred between the punch, die, and the extruded Cu(Al) material. The total compression force, \(P\), on the punch was measured through a MTS load cell with a full range of 25 kN. The total displacement of the actuator, \(\Delta_{\text{total}}\), was measured from readings of the linear variable displacement transducer (LVDT) attached to the actuator. A set of \(P - \Delta_{\text{total}}\) curves were measured as a function of \(D\), with multiple measurements performed at each \(D\) value on nominally identical rod specimens.  
An FEI Quanta3D FEG Dual-Beam scanning electron microscope/focused ion beam (SEM/FIB) instrument was used to examine the grain size and grain morphology of the starting,
ECAP processed, and annealed rod specimens as well as reverse extruded cup-shaped parts. The SEM/FIB instrument included an electron backscatter diffraction (EBSD) attachment (EDAX). Prior to EBSD examinations, specimens were mechanically polished using SiC papers of different grit sizes, followed by a final vibratory polishing with 50 nm silica suspension on a Pace Technologies GIGA 0900 Vibratory Polisher for 12 hours. EBSD mapping was performed at 30 kV and 23 nA at a scan step of 0.5 µm for initial rod specimens and 0.2 µm for as-extruded cup-shaped parts. Raw EBSD data was then analyzed with the TSL OIM software.

4.2.2 Methodology of crystal plasticity finite element simulations

Three-dimensional CPFE simulations [30-32] were performed to simulate the deformation process of polycrystalline Al rod specimens during axisymmetric reverse extrusion. Only the slip modes of plastic deformation were enabled, no twinning was allowed. Under this condition, qualitatively similar results are expected from simulations of deformation of Cu samples, as will be discussed later. The simulation setup, illustrated by Fig. 4.2(a), featured a quarter model of rod specimens, 1.5 mm in height and 3 mm in diameter. Symmetric boundary conditions were applied on surfaces perpendicular to x- and y- directions (referred to as x and y faces), respectively. Specifically, displacements \( u_x = 0 \) on the x face and displacements \( u_y = 0 \) on the y face. The die-sample and punch-sample contacts were modeled as frictionless contacts. Three punch diameters, \( D = 2.73 \text{ mm}, 2.60 \text{ mm} \) and \( 2.40 \text{ mm} \), were considered, giving rise respectively to three extruded cup sidewall thicknesses of \( t = 135 \text{ µm}, 200 \text{ µm} \) and \( 300 \text{ µm} \). To help the convergence of the implicit finite element simulations, the edge of the punch was given a fillet radius of \( R = 200 \text{ µm} \). The total punch displacements simulated were \( 204 \text{ µm}, 300 \text{ µm} \) and \( 434 \text{ µm} \), respectively, for the three different \( t \) values. The choice of the punch displacements ensured the formation of extruded cups with the same nominal height of \( H = 1205 \text{ µm} \). A total of five samples with varying initial
grain sizes, \(d = 375 \, \mu m, 255 \, \mu m, 188 \, \mu m, 112 \, \mu m, \) and \(20 \, \mu m,\) were considered. The models contained 50, 160, 400, 771 and 65400 grains, respectively, as shown in Figs. 4.2(b)-1(f). These grains were generated by the Neper software using Voronoi-based tessellation [33, 34]. The initial orientations of the grains are also generated by Neper to mimic a powder texture, i.e., a nearly-random distribution of orientations [35]. Even so, it may be difficult to reproduce a truly randomized texture for samples with few grains (such as the 50-grain case). For models contained a low number of grains, namely 50, 160, 400 and 771 (see Figs. 4.2(b)-1(e)), each grain was meshed into a number of elements. For the model containing 65400 grains (see Fig. 4.2(f)), each element represented one grain. To improve computational efficiency, the mesh size of the reverse extrusion models is non-uniform—with the finest elements, with a mesh size of 40 \(\mu m,\) placed near the top perimeter and coarsest element placed near the bottom. This strategy, best illustrated in Fig. 4.2(f), ensured that the regions undergoing the extrusion plastic flow had the highest mesh density. Material at the bottom portions of the models, even though having the coarsest mesh density, only provides essential elastic support. Therefore, the artificial grain coarsening effect in the 65400-grain model due to mesh variations is not expected to impact the predicted overall load response during extrusion.

For clarity, the meshing is not shown for the models containing 50, 160, 400 and 771 grains, but all models were meshed following the strategy for the 65400-grain model (see Fig. 4.2(f)). Owing to similar efficiency considerations, the grain size for the model containing 771 grains (see Fig. 4.2(e)) has been substantially coarsened near the base of the model. All models for reverse extrusion contained 70,000 ~ 90,000 elements.
Fig. 4.2 Crystal plasticity finite element (CPFE) simulations of deformation associated with axisymmetric reverse extrusion: (a) a schematic illustration of the CPFE simulation setup; (b), (c), (d), (e), and (f) illustrate grains and meshes for simulations with varying grain sizes.

The most densely meshed regions in the reverse extrusion models, as stated, have a mesh size of 40 μm. A baseline mesh convergence is quickly established based on the distribution of the surface elastic stress under shallow penetration conditions. The setup of the model is shown in Fig. 4.3. Isotropic elastic properties of Al have been assigned to the sample. Uniform mesh sizes of 11.8 μm, 26.7 μm, 40 μm, 90 μm and 135 μm have been considered. The die penetration depth is a shallow 0.00025 μm. As shown in Fig. 4.3, a mesh size of 40 μm can capture the distribution of surface Von Mises stress imposed by this die-punch loading geometry reasonably well.

The CPFE simulations of reverse extrusion were performed using ABAQUS® with an user defined subroutine (UMAT) developed by Marin et al., the detailed derivation and numerical implementation of which were well documented [30, 32, 36]. Therefore, only a brief description of the model formulation is given below. The present authors have utilized this model to analyze the tensile deformation characteristics of nanoscale metal adhesion layers sandwiched between ceramic layers [37].
Fig. 4.3 Mesh convergence on the surface elastic stress. Left panel shows the setup of a quarter-sample model, which similar to what is shown in Fig. 4.2(f). Right panel shows the profile of the surface Von Mises stress as a function of Radial position.

The slip kinetics, assumed to be identical for all slip systems, has the following form:

\[ \dot{\gamma}^\alpha = \dot{\gamma}_0 \left[ \frac{\tau^\alpha}{\kappa^\alpha} \right]^{1/m} \text{sign}(\tau^\alpha), \] (1)

where \( \dot{\gamma}^\alpha \) is the shear strain rate of slip system \( \alpha \), \( \dot{\gamma}_0 \) is the characteristic (or reference) shear strain rate, \( \kappa^\alpha \) is the current strength (or critical resolved shear stress, CRSS) of slip system \( \alpha \), while \( \tau^\alpha \) is the current resolved shear stress (RSS) of slip system \( \alpha \). \( \kappa^\alpha \) is given by its rate, i.e.,

\[ \kappa^\alpha = h_0 \left( \frac{\kappa^\alpha_{S,S} - \kappa^\alpha_{S,0}}{\kappa^\alpha_{S,S} - \kappa^\alpha_{S,0}} \right) \sum_{\alpha=1}^{12} |\dot{\gamma}^\alpha|, \] (2)

where \( \kappa^\alpha_{S,0} \) and \( \kappa^\alpha_{S,S} \) are the starting and saturation CRSS of slip system \( \alpha \), \( h_0 \) is the initial strain hardening rate, due to dislocation accumulations. To calibrate the associated kinematic constants for single crystal Al, material point simulations comprising 500 grains using mean field assumptions were performed[32, 37]. The calibrated constants, along with the elastic constants of Al, are shown in Table 1.

Table 4.1 Elastic constants and constants governing the flow kinetics of single crystal Al utilized in the present work.

<table>
<thead>
<tr>
<th>( C_{11} )</th>
<th>( C_{12} )</th>
<th>( C_{44} )</th>
<th>( h_0 )</th>
<th>( \kappa^\alpha_{S,0} )</th>
<th>( \kappa^\alpha_{S,S} )</th>
<th>( m )</th>
<th>( \dot{\gamma}_0 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>108.2 GPa</td>
<td>61.3 GPa</td>
<td>28.5 GPa</td>
<td>150 MPa</td>
<td>5 MPa</td>
<td>40 MPa</td>
<td>0.02</td>
<td>1.0</td>
</tr>
</tbody>
</table>
As shown in Fig. 4.4, this set of constants can reproduce the experimentally observed uniaxial compression behavior of Al rod samples with an initial grain size of 15 μm reasonably well.

![Graph showing comparison between experimental and CPFE simulations](image)

**Fig. 4.4** Comparison between the experimental compressive response of Al sample (grain size 15 μm) and the material point CPFE simulations comprising 500 grains using mean field assumptions.

Under the conventional CPFE formalism, the yield point of a material is dependent only on the constants selected to describe the slip kinetics (Eqs. (1) and (2)). The true Hall-Petch scaling effect on the yield point cannot be captured unless advanced plasticity theories are implemented. However, a Hall-Petch-like behavior, namely, a grain-refinement-induced strengthening effect after the yield point, has been demonstrated through CPFE models [38] by maintaining the mesh size and reducing the modeled grain size. Such strengthening behavior likely originates from the artificial stiffening of grains when less elements are involved in a grain, which amplifies the effect of the intergranular deformation incompatibility.

To establish a baseline for the effect of grain size in the CPFE formulation, a set of six simulations of uniaxial compression of cylindrical specimens were performed, varying the grain size and the mesh size. A combination of three grain sizes (1700 μm, 600 μm and 300 μm) and
two mesh densities (100,000 and 200,000 total elements per structure) were considered, see Fig. 4.5. The compressed cylindrical specimens had an overall height of 7 mm and diameter of 3 mm. For each structure, the simulation was run to a total compressive strain of 10%. The true stress – engineering strain curves were fitted to the Ludwik flow rule, i.e., \( \sigma = K \varepsilon^n \), and extrapolated up to a total strain of 40%. Based on the extracted flow curves, Hall-Petch constants were then obtained at various strain levels, as shown in Table 4.2.

It should be noted that the grain-size dependence of strain-hardening rate, observed experimentally for Cu and Al, i.e., larger grain sizes correspond to higher strain-hardening rates, was not captured by the present CPFE simulations. Instead, the opposite is predicted by the simulations, i.e., a larger grain size correspond to a smaller strain hardening rate. This is also reflected in Table 4.2 in that the CPFE generated Hall-Petch slope \( k \) increases with increasing strain. This discrepancy may be expected, as the present CPFE model does not incorporate the additional hardening effects due to the presence of strain gradients or GNDs, as discussed later.

Fig. 4.5 Finite element meshes containing ~100,000 elements for three different average grain sizes, i.e. \( d = 1700 \, \mu \text{m} \) (left), 600 \( \mu \text{m} \) (middle) and 300 \( \mu \text{m} \) (right).
Table 4.2 Hall-Petch constants extracted at different total strain values from CPFE simulations of Al rod specimens under uniaxial compression.

<table>
<thead>
<tr>
<th>Strain</th>
<th>No. of Elements</th>
<th>( \sigma_0 ) (MPa)</th>
<th>( k ) (MPa ( \cdot ) ( \mu )m(^{-1/2} ))</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1</td>
<td>100,000</td>
<td>68.9</td>
<td>227.9</td>
</tr>
<tr>
<td></td>
<td>200,000</td>
<td>65.1</td>
<td>272.5</td>
</tr>
<tr>
<td>0.2</td>
<td>100,000</td>
<td>95.4</td>
<td>439.8</td>
</tr>
<tr>
<td></td>
<td>200,000</td>
<td>90.2</td>
<td>483.4</td>
</tr>
<tr>
<td>0.3</td>
<td>100,000</td>
<td>115.2</td>
<td>631.3</td>
</tr>
<tr>
<td></td>
<td>200,000</td>
<td>108.9</td>
<td>668.3</td>
</tr>
<tr>
<td>0.4</td>
<td>100,000</td>
<td>131.64</td>
<td>808.7</td>
</tr>
<tr>
<td></td>
<td>200,000</td>
<td>124.53</td>
<td>836.7</td>
</tr>
</tbody>
</table>

4.3 Results and discussion

4.3.1 Grain size and macroscale flow stress

Fig. 4.6 Grain morphology observed from EBSD IPF maps: (a) Cu, \( d \sim 2 \) \( \mu \)m; (b) Cu, \( d \sim 8 \) \( \mu \)m; (c) Cu, \( d \sim 50 \) \( \mu \)m; (d) Al, \( d \sim 5 \) \( \mu \)m; (e) Al, \( d \sim 15 \) \( \mu \)m. \( d \) denotes the average grain size obtained from EBSD data via the linear interception method.

Figure 4.6 shows typical microstructures of 3 mm diameter Cu and Al rod specimens after ECAP and annealing. The average grain size, \( d \), was measured by the linear interception method,
obtained from the average of grain intercepts to 64 horizontal lines automatically generated by the TSL OIM software. No evidence for significant differences in grain morphology across the entire cross section was observed from the rod specimens after ECAP and annealing. Figure 4.6 (a) shows an EBSD inverse pole figure (IPF) orientation map of one Cu rod specimen annealed at 150 °C for 1 h after 8 passes through the ECAP device. An ample amount of twinning within Cu grains is observed in the map shown in Fig. 4.6(a). The average Cu grain size was determined to be ~2 μm, including intersections with twins in the grain size calculation. Two analogous EBSD IPF maps for annealed Cu rod specimens are shown in Figs. 4.6(b) and 4.6(c). These two specimens were subjected to 1 pass through the ECAP device, and annealed respectively at 600 °C for 8 h and at 800 °C for 22 h. The resulting d values were ~8 μm and ~50 μm, respectively, again including twin intersections. Figures 4.6(d) and 4.6(e) show two EBSD IPF maps obtained from two Al rod specimens subjected to 8 passes and 1 pass through the ECAP device, respectively. The two specimens were then annealed at 200 °C for 1 h and at 400 °C for 1 h, respectively. No twins are evident from the maps shown in Figs. 4.6(d) and 4.6(e), in contrast to the numerous twins present within maps shown in Figs. 4.6(a), 4.6(b), and 4.6(c), and consistent with the known high twin boundary energy for Al and low twin boundary energy for Cu [39]. The d values for the two annealed Al rod specimens were determined from data shown in Figs. 4.6(d) and 4.6(e) to be ~5 μm and ~15 μm, respectively. EBSD mapping was also conducted on Al rod specimens subjected to 1 pass through the ECAP device and annealed at 600 °C for 24 h. Only a few complete grains were mapped in the 500 μm × 500 μm mapping area, indicating that the average Al grain size was larger than ~200 μm in this case. The processing conditions for obtaining the final grain sizes in Cu and Al rod specimens for use in axisymmetric reverse extrusion are summarized in Table 4.3.
Table 4.3 Summary of conditions for ECAP and post-ECAP annealing of 3 mm diameter Cu and Al rod specimens.

<table>
<thead>
<tr>
<th>Material</th>
<th>ECAP process</th>
<th>Heat treatment</th>
<th>Average grain size (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cu 110 alloy</td>
<td>8 pass</td>
<td>150 °C x 1 h</td>
<td>2</td>
</tr>
<tr>
<td>Cu 110 alloy</td>
<td>1 pass</td>
<td>600 °C x 8 h</td>
<td>8</td>
</tr>
<tr>
<td>Cu 110 alloy</td>
<td>1 pass</td>
<td>800 °C x 22 h</td>
<td>50</td>
</tr>
<tr>
<td>Al 1100 alloy</td>
<td>8 pass</td>
<td>200 °C x 1 h</td>
<td>5</td>
</tr>
<tr>
<td>Al 1100 alloy</td>
<td>1 pass</td>
<td>400 °C x 1 h</td>
<td>15</td>
</tr>
<tr>
<td>Al 1100 alloy</td>
<td>1 pass</td>
<td>600 °C x 24 h</td>
<td>&gt;200</td>
</tr>
</tbody>
</table>

Figure 4.7 shows macroscale mechanical response of ECAP processed and annealed Cu and Al rod specimens. Uniaxial compression true stress – true strain ($\sigma$-$\varepsilon$) curves were obtained from 3 mm diameter Cu and Al rod specimens with varying initial average grain sizes, shown respectively in Figs. 4.7(a) and 4.7(b). In Figs. 4.7(a) and 4.7(b), average $\sigma$-$\varepsilon$ curves obtained from averaging up to 6 separate measurements on nominally identical specimens are plotted with corresponding error bars. Power-law fits to the data curves, in the form $\sigma = K(\varepsilon - \varepsilon_0)^n$, are also shown, and are seen to yield good representations of the measured data. For both Cu and Al specimens, flow stresses were observed to increase as $d$ decreases. As $d$ increases, the measured $\sigma$-$\varepsilon$ curves show increased scatter. Because of the small rod diameter, contacts between the top and bottom surfaces of Cu and Al rod specimens and the steel platens were not perfectly aligned. Such imperfect contacts resulted in early onset of plastic deformation near the contact regions of the Cu and Al rod specimens. Consequently, slopes of the initial portion of the measured $\sigma$-$\varepsilon$ curves are significantly lower than that corresponding to the expected elastic modulus for bulk Cu or Al. The determination of the initial yield stress from the measured $\sigma$-$\varepsilon$ curves is thus more uncertain. However, the influence of such mechanical misalignment on the accuracy of the
measured $\sigma$-$\varepsilon$ curves diminishes at larger strains, and flow stress values at larger strains are more reliable. Values of flow stress at different total strains were obtained through the fitted $\sigma$-$\varepsilon$ curves. Figures 4.7(c) and 4.7(d) plot the flow stress values at different total strains, $\varepsilon$, vs. $d^{1/2}$ for Cu and Al rod specimens, respectively. For both Cu and Al, flow stress values scale linearly with $d^{1/2}$ at a fixed value of $\varepsilon$. Data shown in Fig. 4.7 demonstrate that the macroscale flow stress $\sigma$ of the present ECAP and annealed Cu and Al rod specimens obey the Hall-Petch scaling: $\sigma = \sigma_0 + kd^{-1/2}$.

It is noted that the strain-hardening rate of Al rod specimens demonstrates a clear dependence on the initial grain size, i.e., the strain-hardening rate of the coarse-grained samples is noticeably higher than the finer-grained ones (Fig. 4.7(b)). This is in agreement with what was reported in prior studies[40]. Hence, the slope $k$ in the Hall-Petch relation appears to reduce with increasing strain. Similar behavior, albeit less apparent, was also observed for Cu (Fig. 4.7(c)). Indeed, grain-size dependence of initial strain hardening rate has been reported for Cu [41]. The higher strain-hardening rate in coarse-grained metals has been attributed to the presence of a larger population of geometrically necessary dislocations (GNDs) [42]. At the continuum level, the effect of GNDs can be accounted for by the formalism of strain gradient plasticity, which is absent from the formulation of the present CPFE model as discussed below.
Fig. 4.7 Macroscale mechanical response of ECAP processed and annealed Cu and Al 3 mm diameter rod specimens: average true stress - true strain curves obtained from uniaxial compression of (a) Cu and (b) Al; values of flow stress at various total strains vs. the inverse square root of the average grain size for (c) Cu and (d) Al.

4.3.2 Mechanical responses analysis

Axisymmetric reverse extrusion was conducted on ECAP and annealed Cu and Al rod specimens. The raw total compression force vs. the total actuator displacement \( P - \Delta_{\text{total}} \) curves were measured as a function of the punch diameter \( D \). Figure 4.8(a) shows a typical \( P - \Delta_{\text{total}} \) curve obtained from an axisymmetric reverse extrusion run using a punch with \( D = 2.60 \) mm on an ECAP processed and annealed 3 mm diameter Cu rod specimen, with an average grain size \( d \) of ~8 \( \mu \)m. It is noted that the measured \( \Delta_{\text{total}} \) included a system compliance contribution, \( \Delta_{\text{sys}} \).
Fig. 4.8 Measuring mechanical response of microscale axisymmetric reverse extrusion: (a) a total compression force – total displacement ($P - \Delta_{\text{total}}$) curve; (b) the corresponding system compliance ($P - \Delta_{\text{sys}}$) curve; (c) the mechanical response curve with the system compliance contribution removed, $P - (\Delta_{\text{total}} - \Delta_{\text{sys}})$; (d) the final response ($P - \Delta$) curve after correcting for misfit gaps between the rod specimen and the die hole.

To measure the $\Delta_{\text{sys}}$, a separate measurement was made by removing the 3.0 mm diameter Cu rod specimen from the die hole and having the punch bottom surface contacting the bottom steel base plate directly, keeping all other components in the system intact. The result of such a system compliance measurement is shown in Fig. 4.8(b). Figure 4.8(c) shows the result of subtracting the system compliance contribution from the measured total displacement, i.e., $P$ vs. $\Delta_{\text{total}} - \Delta_{\text{sys}}$. It is noted that the curve shown in Fig. 4.8(c) exhibits an initially much more compliant section, which is believed to be caused by the misfit gaps existing between the Cu rod specimen and the die hole prior to the beginning of the extrusion experiment, arising from imperfections in die hole and rod specimen geometries. Consequently, measured initial displacements are not the actual punch penetration into the specimen top surface, but mostly contributed from expansion of
the rod specimen under compression to fill any misfit gaps between the rod specimen and the die hole[26].

To correct for such misfit gaps, a height measurement was conducted on the extruded Cu cup structure, whose measured mechanical response curves were shown in Fig. 4.8. The height measurement was conducted at six different locations with 60° intervals along the rim of the extruded cup sidewall, then the average height value, \( H \), of these six measurements was calculated. As shown in Fig. 4.9(a), \( H = 0.980 \) mm for this case. During the reverse extrusion operation, the extruded material volume should be conserved. Thus, as illustrated in Fig. 4.9(b), the volume conservation equation is written as:

\[
\frac{\pi D^2 \Delta}{4} = \frac{\pi (D_0^2 - D^2)(H - \Delta)}{4}
\]

(1)

where \( D \) is the punch diameter, \( D_0 \) is the diameter of the die hole (3.0 mm), \( \Delta \) is the actual punch penetration depth into the specimen, and \( H \) is the average height of extruded cup. For \( D = 2.6 \) mm and 2.80 mm, \( H/\Delta \) can be calculated from Eq. (1) to be \(~4.0\) and \(~7.8\), respectively. Thus, the actual punch penetration depth \( \Delta \) for the specimen shown in Fig. 4.9(a) should be 0.245 mm, \(~0.015\) mm smaller than the measured value, 0.260 mm, as shown in Fig. 4.8(c). Therefore, the \( P \) vs. \( (\Delta_{\text{total}} - \Delta_{\text{sys}}) \) curve shown in Fig. 4.8(c) was shifted 0.015 mm along the x-axis to arrive at the actual mechanical response curve associated with the axisymmetric reverse extrusion, i.e., the \( P - \Delta \) curve as shown in Fig. 4.8(d), in which \( \Delta \) represents the true punch penetration into the rod specimen.

Figure 4.10 displays so-processed \( P - \Delta \) curves. Up to 6 repeat measurements were conducted at each combination of initial Cu(Al) grain size and punch diameter \( D \). Average \( P - \Delta \) curves were obtained by averaging results of repeat measurements on nominally identical samples, and displayed in Fig. 4.10 with corresponding error bars.
Fig. 4.9 (a) An SEM image of the extruded cup structure whose mechanical response curve is shown in Fig. 4.8; (b) a schematic illustrating volume conservation in the present axisymmetric reverse extrusion geometry.

Fig. 4.10 Mechanical response in microscale reverse extrusion: average P–Δ curves for (a) Cu and (b) Al as a function of the starting average grain size and the punch diameter. For clarity, error bars from averaging repeat measurements on up to 6 nominally identical samples were only displayed at selected points, instead of over the entire P–Δ curve.

For both Cu and Al specimens, all measured response curves appear to be qualitatively similar, exhibiting an initially stiffer section where P increases more rapidly with increasing Δ, followed by a “knee” after which P increases more slowly with Δ in an approximately linear fashion. As D increases from 2.60 mm to 2.80 mm and the nominal extruded cup sidewall thickness t decreases correspondingly from ~200 µm to ~100 µm, the measured P – Δ curves show
obvious and systematic stiffening, i.e., $P$ increases significantly at the same $\Delta$ value as $D$ increases. This is consistent with our previous findings: the extrusion mechanical response showed significant stiffening as $t$ decreased [25].

The influence of the starting grain size on the extrusion process is first reflected in its influence on the extrusion mechanical response. In the case of Al, Fig. 4.10(b) shows that the average $P - \Delta$ curve associated with $d = 5 \mu m$ lies above that associated with $d = 15 \mu m$, while the average $P - \Delta$ curve associated with $d = 15 \mu m$ appears to lie closer to that associated with $d > 200 \mu m$. In the case of Cu, Fig. 4.10(a) shows that the average $P - \Delta$ curve associated with $d = 2 \mu m$ lies above that associated with $d = 8 \mu m$, consistent with the Al case. However, the average $P - \Delta$ curve associated with $d = 50 \mu m$ appears to largely overlap with that associated with $d = 8 \mu m$ when $D = 2.80 \text{ mm}$, and lie above that associated with $d = 8 \mu m$ when $D = 2.60 \text{ mm}$. Thus visual inspection alone suggests that the mechanical response associated with reverse extrusion of Cu and Al rod specimens with the largest $d$ value, $\sim 50 \mu m$ in the case of Cu and $> 200 \mu m$ in the case of Al, may exhibit anomalous behavior, i.e., the average $P - \Delta$ curves appear not to conform to the same Hall-Petch scaling for the macroscale flow stress.

To further quantify this statement, an estimate of a characteristic strain associated with the deformation resulting from the axisymmetric reverse extrusion needs to be made. This is because the macroscale flow stress depends on this characteristic strain, as illustrated by the data shown in Fig. 4.7. It should also be noted that the deformation process associated with axisymmetric reverse extrusion leads to inhomogeneous plastic straining within the extruded material. As shown previously, the location where the most severe plastic straining takes place is approximately half way along the sidewall of the extruded cup [25].
Fig. 4.11 (a) Cross-sectional image of one extruded cup-shaped Cu structure with a sidewall thickness of ~400 µm, the initial average Cu grain size is ~8 µm; (b) and (c) two distinct EBSD IPF maps taken from non-overlapping areas within the white square shown in (a); (d), (e), and (f) show the point-to-point misorientation angle plots along three paths denoted as A to B, C to D, and E to F in (b) and (c).

Figure 4.11(a) shows a cross section of one extruded cup-shaped Cu structure with a sidewall thickness of ~400 µm. The starting average grain size is 8 µm. Figures 4.11(b) and 4.11(c) show two distinct EBSD IPF maps taken from non-overlapping areas within the middle of the extruded Cu cup sidewall, as indicated by the white square area shown in Fig. 4.11(a). The observed grain morphologies indicate significant plastic deformation, changing the originally largely equiaxed Cu grains shown in Fig. 4.6(b) into elongated fibrous grain structures along the extrusion direction. Figures 4.11(d), 4.11(e), and 4.11(f) show the point-to-point misorientation angle plots obtained along three paths outlined by the black arrows denoted as point A to B, point C to D, and point E to F in Figs. 4.11(b) and 4.11(c). The grain width was taken as the spacing between two consecutive peaks, the misorientation angles of which differ by more than 15°. The average grain width, \( w \), was then calculated by averaging the so-obtained grain width values. As shown in Figs. 4.11(d), 4.11 (e), and 4.11 (f), the number of grains obtained along paths A-B, C-
D, and E-F are 30, 28 and 30, respectively. The average $w$ value obtained from paths A-B, C-D, and E-F is ~5.0 µm. Similar EBSD measurements were carried out on extruded Cu specimens with sidewall thickness of ~200 µm, ~100 µm and 5 µm, and on extruded Al cup structures with initial grain size of $d$~15 µm and extruded sidewall thickness of ~200 and ~100 µm.

Fig. 4.12 Estimate of characteristic strain associated with axisymmetric reverse extrusion: (a) average width $w$ of elongated Cu and Al grains in the middle of sidewalls of extruded cup structures as a function of the sidewall thickness $t$; b) an estimated characteristic plastic strain $\epsilon_b$ as a function of $t$. The results from CPFE simulations on Al deformation are included in (b).

Figure 4.12(a) plots values of so-obtained average grain width, $w$, in the middle of sidewalls of extruded Cu and Al cup structures as a function of the nominal cup sidewall thickness, $t$. It shows that $w$ does not decrease linearly with decreasing $t$. Recognizing that the $d$ values are respectively ~8 µm and ~15 µm for the Cu and Al rod specimens, and that the initial Cu and Al grains are largely equiaxed, the $w$ value can be taken to reflect the amount of plastic straining undergone by the corresponding grains. An estimate of this characteristic strain, $\epsilon_b$, was taken as $\ln(d/w)$. Then $\epsilon_b$ was plotted versus $t$, as shown in Fig. 4.12(b). Figure 4.12(b) shows that $\epsilon_b$ increases significantly as $t$ decreases. The reasonable agreement between the Cu case and the Al case, even when the initial grain sizes in the two cases were different, suggests that this characteristic plastic strain is mainly a function of the extrusion geometry. According to data
shown in Fig. 4.12(b), the characteristic plastic strain values at $t = 200 \, \mu\text{m}$ and $100 \, \mu\text{m}$ are respectively 0.75 and 1.12 for Cu and 0.84 and 1.10 for Al.

![Normalized mechanical response of microscale axisymmetric reverse extrusion](image)

Fig. 4.13 Normalized mechanical response of microscale axisymmetric reverse extrusion: compression pressure scaled by macroscale flow stress vs. punch penetration for (a) Cu and (b) Al rod specimens at different initial average grain sizes.

The measured extrusion mechanical response, as expressed by the average $P - \Delta$ curves shown in Fig. 4.10, is then scaled by the appropriate macroscale flow stresses, $\sigma$. The compression pressure associated with the extrusion process is calculated from the compression load and the punch diameter, $p = P/(\pi D^2/4)$. Figure 4.13 shows $p$ normalized by $\sigma$, plotted versus the punch penetration $\Delta$. For each punch diameter $D$, the characteristic plastic strain value was obtained from data shown in Fig. 4.12(b). The corresponding $\sigma$ value was then obtained at this strain value for the appropriate starting average grain size, from data shown in Figs. 4.7(c) and 4.7(d). Examining the $p/\sigma - \Delta$ curves shown in Fig. 4.13(b), it is seen that the scaled extrusion mechanical response curves for the largest average Al grain size, $d > 200 \, \mu\text{m}$, lie consistently above the curves corresponding to $d \sim 15 \, \mu\text{m}$, in line with the impression obtained from a visual examination of the average $P - \Delta$ curves shown in Fig. 4.10. However, the statement is tempered by the fact that the scaled curves lie within one standard deviation of another, thus the existence of scaling anomaly,
if any, is not strong according to the present experimental results on Al. In the Cu case, the $p/\sigma - \Delta$ curves shown in Fig. 4.13(a) for the largest average Cu grain size, $d \sim 50 \, \mu m$, lie above those corresponding to the ones for $d \sim 8 \, \mu m$, significantly outside the error band. Thus the present experiments on Cu show a clear indication of a scaling anomaly: the extrusion pressure scaled by the corresponding macroscale flow stress appears to be stiffer, by $15 \text{--} 20\%$, for the largest Cu grain size, $d \sim 50 \, \mu m$, as compared to that for the medium Cu grain size, $d \sim 8 \, \mu m$. The characteristic extrusion dimension (CED) for the present axisymmetric reverse extrusion geometry is defined by the extruded cup sidewall thickness, $t$. Results shown in Fig. 4.13 suggest that, at the 100-200 $\mu m$ length scale, the influence of the starting average grain size on the extrusion mechanical response may be such that, as $d/t$ becomes sufficiently large, the extrusion response no longer follows the one-parameter scaling with the scaling parameter being the macroscale flow stress, as expected from continuum plasticity.

4.3.3 Inhomogeneous deformation in microscale reverse extrusion

Figure 4.14 shows SEM images of extruded cup-shaped parts using Cu rod specimens with various average grain sizes. At a fixed $D = 2.60 \, mm$ or $t = 200 \, \mu m$, a change from homogeneous to inhomogeneous deformation is evident as the Cu grain size increases. More axisymmetric cup-shaped parts were formed from Cu rod specimens with $d \sim 2 \, \mu m$ (Fig. 4.14(a)), with the height of the extruded cup sidewall being more uniform around the perimeter. Cups extruded with Cu rod specimens with $d \sim 50 \, \mu m$ (Fig. 4.14(c)) exhibit irregular sidewall heights around the perimeter, deviating from axisymmetry.
Fig. 4.14 Morphology of extruded, cup-shaped, Cu structures. Top row, punch diameter $D=2.60$ mm, cup sidewall thickness $t=200$ µm, the average Cu grain sizes $d$ are (a) ~2 µm; (b) ~8 µm; (c) ~50 µm; Bottom row, $D=2.80$ mm, $t=100$ µm, $d$ values are (d) ~2 µm; (e) ~8 µm; (f) ~50 µm.

The same phenomena can be observed for extruded Cu specimens at a fixed $D = 2.80$ mm or $t = 100$ µm (Fig. 4.14 (d) to Fig. 4.14 (f)). On the other hand, given the same $d$, decreasing $t$ leads to more irregularities in cup height around the perimeter. The same change from
homogeneous to inhomogeneous deformation can be seen in extruded cup-shaped Al parts as well, as shown in Fig. 4.15.

Fig. 4.16 Transverse cross-sectional images of extruded cup-shaped Cu parts, with starting average grain sizes of (a) d~2 µm and (b) d~50 µm; (c), (d), (e) EBSD IPF maps taken at locations denoted respectively as 1, 2, and 3 in (a); (f) shows the EBSD IPF map corresponding to (b).

Figure 4.16 shows EBSD IPF maps of cup-shaped Cu parts, extruded by a punch with $D = 2.60$ mm. Figure 4.16(a) shows a longitudinal cross section SEM image of one extruded Cu cup. In this case, $t = 200$ µm and $d \sim 2$ µm. Figures 4.16(c), 4.16(d), and 4.16(e) show EBSD IPF maps taken at three different locations, denoted respectively as 1 (close to the original top surface of the starting Cu rod specimen), 2 (in the middle across the extruded cup sidewall and significantly below the original Cu surface), and 3 (close to the bottom corner of the extruded cup structure) in Fig. 4.16(a). Figure 4.16(b) shows a longitudinal cross section SEM image of another cup-shaped Cu part, with $d \sim 50$ µm, extruded with the same punch. Figure 4.16(f) shows the corresponding EBSD IPF map. From Figs. 4.16(c)-4.16(f), it is clear that formation of the cup sidewall from a rod specimen with $d \sim 2$ µm involved a large number of Cu grains. In contrast, for the Cu rod
specimen with d~50 µm, only a few Cu grains participated in the deformation. In the latter case, the orientation and location of individual Cu grains strongly affect the homogeneity of deformation, thus strongly impacting the final shape of the extruded cup-shaped part, here manifested mainly in whether the height of the cup sidewall deviates from axisymmetry. Similar observations of inhomogeneous deformation behavior when the total number of grains participating in the forming process becomes small have been made in previous micro forming studies [5, 6, 27].

4.3.4 CPFE simulations of microscale reverse extrusion

Accompanying the experimental observations shown above, CPFE simulations were performed on axisymmetric reverse extrusion of Al. Because the present CPFE model formulation includes slip deformation but not twinning, the choice of simulating Al is thus consistent with the experimentally observed lack of twinning in Al. Furthermore, CPFE simulations on Cu using the present model were not performed since we expect similar results, as Cu and Al would be simulated with identical deformation modes (slip, albeit with slightly different slip kinetics), without consideration of twinning. The experimental estimate of the characteristic strain associated with the axisymmetric reverse extrusion process finds good quantitative agreement with the CPFE simulation results, superimposed as blue solid circles with error bars with the experimental data points shown in Fig. 4.12(b). To extract the characteristic strain from the CPFE simulations, the principal logarithmic total strains were analyzed. The logarithmic total strain has the following form:

$$\varepsilon^{LE} = \ln V$$  \hspace{1cm} (3)

where $V$ is the left stretch tensor, following the polar decomposition of the deformation gradient, i.e. $F = V \cdot R$, where $F$ is the deformation gradient and $R$ is the right rotation tensor. The mean values of the maximum principal strains of extruded cup wall sections were obtained from
ABAQUS® and then averaged among the simulations performed at the same punch diameter. As shown in Fig. 4.12(b), this characteristic strain obtained from CPFE simulations clearly increases with increasing punch diameter $D$ and decreasing cup sidewall thickness $t$, and exhibits a similar magnitude as well as a similar dependence on $t$ as compared to the experimental data points.

Fig. 4.17. Simulated mechanical response of Al during axisymmetric reverse extrusion: (a) compression pressure vs. punch displacement at different $D$ values; (b) compression pressure normalized by flow stress vs. punch displacement at different $D$ values.

The simulated compression pressure for extrusion, $p = P/(\pi D^2/4)$, was plotted as a function of punch penetration $\Delta$ in Fig. 4.17(a). The CPFE analyses show $p - \Delta$ responses that vary inversely with grain size, i.e. smaller $d$ values lead to stiffer mechanical responses, in qualitative agreement with experimental observations for Al specimens shown in Fig. 4.10(b). However, the experimentally observed scaling behavior, shown in Fig. 4.13, was not reproduced in the CPFE simulations. Separate CPFE simulations were performed on uniaxial compression of rod specimens and detailed in Section 4.2.2. Values of flow stress at a total strain of 40% were extrapolated from these simulations, specifically $\sigma = \sigma_0 + k d^{-1/2}$ with $\sigma_0 = 125$ MPa and $k = 837$ MPa $\cdot \mu m^{-1/2}$ as shown in Table 4.2 in Section 4.2.2. If the compression pressure $p$ in the CPFE generated $p - \Delta$ curves are scaled by such CPFE generated $\sigma$ values, this normalization would make
the $p/\sigma - \Delta$ curves more spread out as compared to the original $p - \Delta$ curves, rather than collapsing them. This normalization would also invert the trend of finer grain sizes bringing stiffer extrusion responses, and give the least stiff $p/\sigma - \Delta$ response to the smallest grain size.

This discrepancy may be due to the inability of the CPFE model to capture the experimentally observed grain-size dependent strain-hardening rates. As observed experimentally for both Cu and Al, larger grain sizes correspond to higher strain-hardening rates. Instead, the opposite is predicted by the CPFE model, i.e., a larger grain size correspond to a smaller strain hardening rate. This is also reflected in Table 4.2, in that the CPFE generated Hall-Petch slope $k$ increases with increasing strain, contrary to experimental observations shown in Fig. 4.7. As noted before, the present CPFE model does not incorporate any additional hardening effects due to the presence of strain gradients or GNDs.

However, a quasi one-parameter scalability can be achieved with the CPFE generated $p - \Delta$ curves. Figure 4.17(b) displays the $p - \Delta$ curves obtained from CPFE simulations, normalized by flow stress values prescribed by a Hall-Petch like relation, $\sigma$ (MPa) = 125 + 58$d^{-1/2}$. The $\sigma_0$ parameter (125 MPa) here is identical to that obtained from the CPFE simulations of uniaxial compression at 40% strain, while the $k$ parameter (58 MPa $\cdot$ $\mu$m$^{-1/2}$) is greatly reduced from the CPFE generated value (Table 4.2). This normalization appears to “collapse” the $p/\sigma - \Delta$ curves corresponding to each $D$ value. The collapsed $p/\sigma - \Delta$ curves shown in Fig. 4.17(b) indicate that the CPFE generated $p - \Delta$ curves cannot reproduce the experimentally observed Hall-Petch scaling reversals shown in Fig. 4.13.
Fig. 4.18. Simulated height variation of extruded cup sidewalls, measured by standard deviations of the wall height: (a) sidewall height variation vs. grain size at various punch diameters; (b) sidewall height variation vs. the normalized sidewall thickness; (c) evolution of sidewall height variation during extrusion: normalized height variation vs. normalized punch penetration depth. The scatter arises from averaging simulations corresponding to different grain sizes; (d) sidewall height variation vs. $1/J_{ODF}$ based on the starting texture of the extruded material.

Inhomogeneous deformation has also been observed in the CPFE simulations of axisymmetric reverse extrusion. Figure 4.18(a) shows the height variation of the extruded cup sidewalls. This variation is measured by the standard deviation of the sidewall height (SD), plotted versus $d$ for different punch diameters. The SD value shows a clear increasing trend with increasing $d$ for $D$ of 2.4 mm and 2.6 mm. At the largest punch diameter of $D=2.73$ mm (data points shown as red squares in Fig. 4.18(a)), a similar overall trend is preserved although the results show substantial scatter. In Fig. 4.18(b), following Meng and Fu[43], SD is plotted vs. the sidewall
thickness normalized by the starting average grain size, $t/d$. As shown, an approximate inverse power law trend exists between SD and $t/d$, i.e. $SD (\mu m) = 29.6(t/d)^{-1.178}$. The CPFE simulations also revealed that the formation of the sidewall height variation occurs in the early stage of the extrusion process. Figure 4.18(c) shows normalized SD as a function of normalized punch penetration depth. Both SD and the punch penetration depth have been normalized by their maximum values in each simulation. It is evident from Fig. 4.18(c) that the sidewall height variation is fully developed within the first 60% of the extrusion process.

It is worth noting that the inverse power law regarding inhomogeneous deformation, i.e., $SD \sim (t/d)^{-1}$, is true on condition that the starting texture of the material being extruded is random. A specimen with a strong initial texture, even with a small starting grain size, is expected to produce extruded cup sidewall height variation that deviates from this inverse power law. To demonstrate this, a fine-grained finite element structure (nominal grain size $\sim 20 \mu m$, with each element representing one grain) with a texture similar to that of the 50 grain model (shown in Fig. 4.2(b), with $d = 375 \mu m$) was created and subjected to reverse extrusion, with $D = 2.4$ mm. The CPFE result for this case is shown as a green “x” with black background in Fig. 4.18(a). As expected, the sidewall height variation for this case is close to that of the $d = 375 \mu m$ case, and clearly deviates from the trend established by the rest of the CPFE simulations assuming an initial random texture.

To further illustrate the influence of texture on the extruded cup quality, the initial texture of the finite element models was analyzed. During axisymmetric reverse extrusion with different punch diameters, the volume of material being extruded, i.e. volume of the “cup sidewalls”, is different. Assuming the same cup sidewall height $H$, the volume, for the quarter model, was estimated as
\[ V_{\text{wall}} = \pi(D_{\text{die}}^2 - D^2)H/16. \] (4)

Therefore, with \( D_{\text{die}} = 3 \) mm, the volumes are \( 0.3H, 0.44H \) and \( 0.64H \) in mm\(^3\) for cases of \( D = 2.73 \) mm, 2.6 mm and 2.4 mm, respectively. The texture index of the orientation distribution function (ODF), \( J_{\text{ODF}} \) [45], for the finite element structures’ initial texture was calculated using the MTEX package [46]. The \( J_{\text{ODF}} \) is the measure of the “severity” of a materials texture, with \( J_{\text{ODF}} = 1 \) indicating a complete random texture and \( J_{\text{ODF}} = \infty \) indicating a single crystal. It is therefore convenient to plot the extruded cup sidewall height variation as a function of \( 1/J_{\text{ODF}} \) of the initial texture of materials making up the cup sidewall. In addition, since SD fully develops in the early stage of the extrusion process, the \( J_{\text{ODF}} \) is calculated for the sidewall materials at only 60\% of the extrusion process. Such a plot is shown in Fig. 4.18(d). Notwithstanding the apparent scatter in the data, a decreasing trend can be seen between SD and \( 1/J_{\text{ODF}} \). Note the outlying data point in Figs. 4.18(a) and 4.18(b), the green “x” with black background, corresponding to a fine grain structure with texture similar to the 50 grain case \( (d = 375 \mu m) \), now follows the overall decreasing trend in Fig. 4.18(d).

One additional example illustrating the influence of initial texture on deformation inhomogeneity in axisymmetric reverse extrusion, involving a hypothetical axially symmetric crystallographic orientation relation (OR). This type of OR can be stated as follows (Fig. 4.19(a)): if a local Cartesian coordinate system \( (x'-y'-z') \) is formed in each grain with \( z' \) parallel to the global \( z \)-axis and \( x' \) perpendicular to \( z' \) through the center of the specimen, then the crystallographic orientation of any grain has a fixed OR with respect to this local coordinate system. A simple case satisfying this OR is such that each grain has orientation: [100]/\( x' \), [010]/\( y' \) and [001]/\( z' \). This OR was implemented to the 50-grain finite element structure, with grain size \( d = 375 \mu m \). Due to the imposed rotational symmetry, the (100) pole figure with respect to the x-y-z
system indicates a strong (100) fiber texture (Fig. 4.19(b)). However, if all the material points (finite elements) have been rotated about z axis to the x-z plane, a (100) pole figure with respect to the x-y-z system would indicate a strong single crystal texture (Fig. 4.19(c)). Such a finite element structure, when subjected to reverse extrusion with punch diameter $D = 2.4$ mm, lead to a relatively smooth cup wall (S.D. of 11 μm, shown in Figs. 4.18(a), 4.18(b), and 4.18(d) as green “+” with black background) despite its large grain size. The cup sidewall height variation generated by this specially case apparently deviates from the general trends observed in prior cases with respect to grain size ($d$, shown in Fig. 4.18(a)), normalized channel width ($t/d$, shown in Fig. 4.18(b)) and inversed texture index ($1/J_{ODF}$, shown in Fig. 4.18(d)).

Fig. 4.19 Illustration of a hypothetical axially symmetric crystallographic orientation relation (a), as well as texture (b)-(c) of a finite element model for a hypothetical sample.

4.3.5 Additional remarks

The present experiments yielded no information on friction between the punch and the extruded material or between the extruded material and the die wall. While acknowledging this
limitation, friction and its potential influence deserve some comment. To reiterate the experimental facts, it is worth noting first that all extrusion runs were conducted under “dry” conditions, without the use of any lubricant. Second, the characteristic relative velocity between the punch and the extruded material or between the extruded material and the die wall is ~1 µm/s for the present extrusion experiments, at a typical extruded cup sidewall height of ~1000 µm and a typical extrusion run time of ~1000 s. Change in friction between the extruded material and the punch or the die is expected to come largely from the change in contact pressure, which is expected to increase as the punch diameter $D$ increases or the extruded cup sidewall thickness $t$ decreases [44]. The difference in sliding velocity between different parts of the extruded cup structure is small, and thus the velocity influence on friction is not expected to be significant. However, because of potential additional material strengthening as $t$ decreases, the effects of friction and material strengthening at small length scales are intertwined. A clear separation of these effects await future study.

We suggest that the decreased number of grains across the extruded cup sidewall in combination with the increased plastic strain as the sidewall thickness decreases leads to the observed change from homogeneous to inhomogeneous deformation. As shown by Eq. (4), the axisymmetric reverse extrusion process with a smaller (larger) punch diameter would involve a larger (smaller) number of grains in the plastic deformation, if the starting grain size is maintained constant. As the extruded cup sidewall thickness decreases to tens of microns, only a small number of grains are involved in the formation of the cup sidewall, and the difference in the degree of deformation in each grain becomes significant. The effect of this inhomogeneous deformation is further accentuated as the average strain of each grain increases. Such effects become much diminished when a large number of grains are involved in the deformation process: either at a fixed
initial grain size but increased characteristic forming dimension; or at a fixed characteristic forming dimension but decreased average grain size.

The present simulation results serve to illustrate the benefit of combining CPFE with experimentation. Figure 4.12(b) shows reasonable agreement between the CPFE generated principal logarithmic total strain within the extruded cup sidewall and the experimental estimate of the characteristic strain of reverse extrusion. This agreement, both in terms of the strain magnitude and the dependence of strain on the extruded cup sidewall thickness, offers partial validation of the present CPFE model. In the analysis of the influence of texture of the starting material on the extruded part quality, as shown in Fig. 4.18, especially Fig. 4.18(d), a distinct advantage is found for CPFE. An analogous feat would be extremely difficult, if not impossible, for experimentation. The influence of initial texture of the materials being extruded on the shape of extruded parts has not been much discussed in the literature, and the present CPFE results will help shed some light on this issue.

However, it should also be recognized that some physics elements may be missing from the present CPFE model for it to be capable of fully capturing the essence of metal forming at characteristic dimensions of 100-200 microns and smaller. The present experiments indicate a breakdown of one-parameter scaling of the extrusion mechanical response with the scaling parameter being the macroscale flow stress, as shown in Fig. 4.13 for both Al and Cu, and especially for Cu as shown in Fig. 4.13(a). In contrast, the present CPFE simulations on Al are unable to capture the additional stiffening of the extrusion response at the largest grain size, and similar output is expected if simulation on Cu were to be conducted. In addition, the grain-size dependent strain-hardening behavior observed experimentally through uniaxial compression of the rod samples could not be captured by the CPFE simulations.
Physically, while the impediments on dislocation motion by grain boundaries give rise to Hall-Petch scaling [45], the presence of strain gradients within grains and the consequent generation of GNDs can give rise to additional material hardening [46]. In the present axisymmetric reverse extrusion experiments, the deformation geometry imposes strong strain gradients on the extruded material. If large grains are more capable of accommodating strain gradients within as compared to small grains, then an additional hardening component may be present within large grain materials and absent from the small grain counterparts. Such strain gradient hardening effects are distinct from dislocation motion impediment by grain boundaries, and may be responsible for the stiffer extrusion response presently observed for large grain materials, especially Cu, as compared to what is expected from Hall-Petch scaling alone. The twin boundaries, such as the annealing-induced ones shown in Figs. 4.6(a)-4.6(c), may serve as effective dislocation barriers similar to grain boundaries, hence twinning in Cu may also contribute to the scaling anomaly observed in Fig. 4.13(a). Under severe plastic deformation conditions such as during extrusion, twinning can be activated in Cu at room temperature and low strain rates [47, 48]. In addition, under deformation, larger grains tend to develop higher volume fraction of twins [48]. Therefore, Cu samples with larger grain sizes may develop higher densities of twin boundaries, thus contributing to their stiffer extrusion response. Exploring and quantifying such possibilities in experimentation and simulation remain a task for the future. Strain gradient plasticity theory states that the effective plastic strain contains a contribution from plastic strain and a contribution from plastic strain gradient, $E_p = \sqrt{2\varepsilon_{ij}^p \varepsilon_{ij}^p / 3 + l^2 2\varepsilon_{ijk}^p \varepsilon_{ijk}^p / 3}$, where $\varepsilon_{ij}^p$ and $\varepsilon_{ijk}^p$ are respectively components for plastic strain and plastic strain gradient, and $l$ is a material length scale parameter [49]. The formal plastic strain gradient contribution is absent in the present CPFE model formulation. Determining whether inclusion of such effects into updated CPFE
models would result in better representation of experimental situations in microscale metal forming likewise remains a task for the future.

4.4 Summary

A detailed study of axisymmetric reverse extrusion of Cu 110 and Al 1100 alloy rod specimens was carried out. At a characteristic extrusion dimension of 100-200 µm, the study was focused on the influence of the initial average grain size on the mechanical response and homogeneity of plastic deformation. Accompanying crystal plasticity finite element simulations were carried out. Reasonable agreement was obtained between the experimentally determined characteristic plastic strain for extrusion and the CPFE generated logarithmic total strain, partially validating the current CPFE model. The mechanical response of extrusion showed deviations from continuum plasticity scaling behavior as the characteristics extrusion dimension became small as compared to the initial grain size, an effect indicated by the present experimental data but not captured by the current CPFE model. The initial grain size of the material being extruded was shown to strongly influence the final shape of extruded parts, while CPFE simulations provided additional insights regarding the influence of the initial texture of material being extruded on the extruded part shape. The present study serves as a baseline for further studies of metal forming at the meso to micro scales, and demonstrates that a comprehensive understanding of meso – micro scale metal forming, inclusive of an understanding of the mechanical response and the influence of grain size and texture, remains to be developed in the future.

Clarification: The CPFEA simulation in this chapter was conducted by Dr. Shao and his students, Shahrior Ahmed and Mohammad Dodaran.
4.5 References


CHAPTER 5. MECHANICAL RESPONSE AND INCOMPLETE FILLING IN COMPRESSION MOLDING ON AL WITH MICROSCALE SINGLE PUNCHES AND DOUBLE-PUNCH SETS

5.1 Introduction

Fabricating surface patterns at nano and micro dimensions offers a means for achieving structures with potential usage in wide ranging technological applications, including photonic devices [1], electronic devices [2], micro chemical reactions [3], and micro heat exchangers [4]. Fabricating nano scale surface patterns by nanoimprinting lithography (NIL) has been extensively studied [5]. The NIL protocol involves an imprinting step in which a nanostructured hard die is pressed into a thin layer of polymeric resist on a substrate, followed by a pattern transfer step in which the imprinted pattern onto the resist is transferred to the substrate surface, typically by reactive ion etching (RIE). Prior to the development of the NIL technique in the 1990s, microscale imprinting with electrodeposited Ni dies has been used to impart microscale surface patterns onto polymeric materials, as in the molding step of the the LiGA microfabrication protocol (Lithographie, Galvanoformung, Abformung) [6]. Common to both NIL and LiGA, the pattern creation process proceeds by pressing a structured hard die into polymeric materials above their glass transition temperatures. These imprinting processes involve viscous flow of polymeric liquids into die recesses, filling these recesses and thus forming nano or micro scale patterns after cooling followed by demolding. Besides pattern replication in polymeric materials, direct microscale pattern transfer onto metals by molding with structured hard dies has been demonstrated over a decade ago, examples include pattern replication by micro molding in Al [7], Cu [8], and Ni [9]. The key difference between the physical mechanisms responsible for micro/nano pattern replication in polymers and in metals is that one involves viscous flow of polymeric liquids and the other involves plastic flow of metals into die recesses under compression.
Plastic deformation at small length scales, from ~1000 µm to sub-µm, has been intensely researched in the past two decades. Significant and non-monotonic dependence of the flow stress of metals have been observed on specimens with characteristic external sizes ranging from ~1000 µm and above to ~1 µm and below [10]. In the ~100 µm to ~10 µm scale, strengthening has been observed when strain gradients are imposed by the deformation geometry, such as in torsion of thin wires [11] and bending of thin foils [12]. With no strain gradients, significant strengthening has also been observed in axial compression of samples when their external dimensions decrease from ~40 µm down to ~0.2 µm [13]. In contrast, softening was observed in uniaxial tests on specimens with external dimensions from ~3000 µm down to ~200 µm[10, 14-16]. Plasticity at small scales is directly relevant to pattern replication by compression molding in metals at such scales. For example, quantitative analysis of the mechanical response of a single crystalline Al specimen to molding by a single, long, rectangular strip punch showed a significant size effect, i.e., the characteristic molding pressure increases sharply as the punch width decreased from ~5 µm to ~0.55 µm [15].

One particular concern in pattern replication by molding is whether the material being molded is able to completely fill the die recesses during the molding process, especially when the recesses are of different dimensions. One case in point is shown in Fig. 5.1, which displays a typical result of elemental Al after compression molding with a Si die. The Si die contains a one-dimensional periodic array of long rectangular punches. The width of the individual Si punch, w, is ~9 µm, and the spacing between two adjacent punches in the array, s, is also ~9 µm. The planview scanning electron microscopy (SEM) image shown in Fig. 5.1(a) gives the impression that the compression molding process created a periodic array of trenches on the Al surface. However, a closer examination shown in the tilted SEM image of Fig. 5.1(b) shows that Al flow
into the recesses between individual Si punches is far less than the total punch displacement into
the molded Al piece, as indicated by the depth of the feature created at the outer most edge of the
Si die. Stated in the extreme, the entire Si die appeared to have sunk into the molded Al as if a flat
piece, without any recesses present. Such a result of compression molding is clearly not
satisfactory when the goal is to imprint multiple smaller geometrical features onto the molded
piece. To our knowledge, this phenomenon, although clearly relevant to high-throughput and low-
cost fabrication of micro/nano scale surface patterns, has not been studied in detail in the literature.

Fig. 5.1 Problems with compression-based pattern replication with molds containing multiple
features: (a) a planview SEM image, showing a bulk piece of Al after compression molding with
a Si mold insert, consisting of an one-dimensional periodic array of rectangular Si protrusions.
The width of each Si protrusion is ~9µm, and the pitch of the array is ~18µm; (b) a 50° tilt view
of the same molded Al piece, showing incomplete filling of Al between each Si protrusion.

To generalize, we aim in this chapter to better understand the material’s response to small
scale compression molding by a hard die containing protrusions/recesses with different dimensions,
including the degree of die filling achieved. A single crystalline Al specimen was compression
molded by tool steel dies in the form of two parallel rectangular punches of nominally the same
dimensions separated by a spacing in between them, dubbed “double punches” of different widths,
w, and spacing between them, s. In what follows, Section 2 describes the procedures for
experimentation. Section 3 presents the major results and a discussion, and Section 4 gives a summary.

5.2 Procedures for experimentation and simulation

5.2.1 Fabrication of single and double rectangular strip punches

A FEI Quanta 3D FEG Dual-Beam scanning electron microscope/focused ion beam (SEM/FIB) instrument was used to fabricate two series of rectangular strip punches in a tool steel, with a bulk hardness of HRC60. Commercially available 1 mm diameter tool steel rods were mechanically polished at one end to reduce its diameter to ~50 to ~100 µm. The final cutting step was accomplished on the FIB instrument, using the same Ga\(^+\) ion beam current of 1 nA to polish the top and sidewall surfaces of each punch.

![SEM images of the FIB-cut rectangular strip punches](image)

Fig. 5.2 SEM images of the FIB-cut rectangular strip punches, (a) a single rectangular strip punch with length ~22 µm and width ~4 µm; (b) a double-punch set, each single rectangular punch has length of 62 µm, width of ~22.5 µm. The spacing between two punches is ~4.5 µm.

A series of single, rectangular strip punches were first cut, with values of their widths, w, of ~13 µm, ~8 µm, ~4 µm, ~2 µm, and ~1 µm. The length of the punches was at least three times of the punch width, such that the compression molding can be considered to occur under approximately plane strain conditions. Then, three different single rectangular punches with the same length of ~22 µm, but varying w values of ~22 µm, ~12 µm, and ~4 µm, were fabricated.
These three punches with different ratios of length to width of 1, 1.8, and 5.5, were used to test the effect of punch in-plane aspect ratio on the molding response. Figure 5.2(a) shows the SEM image of one single rectangular punch with a length ~ 22 µm and a width of ~ 4 µm.

Table 5.1 dimensions of double-punches with fixed $s$~2 µm

<table>
<thead>
<tr>
<th>$s$ (µm)</th>
<th>2.0</th>
<th>2.0</th>
<th>2.0</th>
<th>2.0</th>
<th>2.0</th>
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<tr>
<td>$w$ (µm)</td>
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<td>13.0</td>
<td>10.2</td>
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<td>4.2</td>
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<tr>
<td>$\lambda$</td>
<td>0.12</td>
<td>0.15</td>
<td>0.20</td>
<td>0.31</td>
<td>0.48</td>
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</tbody>
</table>

Table 5.2 dimensions of double-punches with fixed $\lambda$~0.20

<table>
<thead>
<tr>
<th>$s$ (µm)</th>
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<th>2.0</th>
<th>3.2</th>
<th>4.5</th>
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<tbody>
<tr>
<td>$w$ (µm)</td>
<td>5.0</td>
<td>10.2</td>
<td>16.0</td>
<td>22.5</td>
</tr>
<tr>
<td>$\lambda$</td>
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<td>0.20</td>
<td>0.20</td>
<td>0.20</td>
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Table 5.3 dimensions of double-punches with fixed $w$~5.0 µm

<table>
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<th>3.5</th>
<th>5.2</th>
<th>7.5</th>
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<td>$w$ (µm)</td>
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<td>5.2</td>
<td>5.0</td>
<td>5.2</td>
<td>5.2</td>
<td>5.0</td>
</tr>
<tr>
<td>$\lambda$</td>
<td>0.20</td>
<td>0.29</td>
<td>0.48</td>
<td>0.67</td>
<td>1.0</td>
<td>1.5</td>
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Table 5.4 dimensions of double-punches with varying $s$ and $w$

<table>
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<th>8</th>
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<tbody>
<tr>
<td>$w$ (µm)</td>
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<td>4.3</td>
<td>3.6</td>
</tr>
<tr>
<td>$\lambda$</td>
<td>1.0</td>
<td>1.8</td>
<td>2.2</td>
</tr>
</tbody>
</table>

A number of tool steel double-punches were cut. The dimensions of each double-punch are shown in Tables 5.1 through 5.4, where $w$ is the width of one single rectangular punch and $s$ is the spacing between two rectangular punches of nominally the same dimension. We define $\lambda$ as the ratio $s/w$. Table 5.1 shows the dimensions of five double-punches. Each double-punch has the same $s$ of ~2 µm, and $w$ decreases from ~17.0 µm to ~4.2 µm, such that $\lambda$ varies from 0.12 to 0.48. Table 5.2 shows the dimensions of another four double-punches, keeping $\lambda$ fixed at ~0.20, while varying
the absolute dimensions. As $s$ increases from 1.0 $\mu$m to 4.5 $\mu$m, correspondingly, each single punch width increases from 5.0 $\mu$m to 22.5 $\mu$m. Table 5.3 shows the dimensions of six double-punches, keeping $w$ fixed at $\sim$5 $\mu$m and varying $s$ from 1.0 $\mu$m to 7.5 $\mu$m. Therefore, the ratio $\lambda$ increases from 0.20 to 1.5. Table 5.4 shows the dimensions of another three double-punches, with varying $w$ and $s$ values. The ratio $\lambda$ increases from 1.0 to 2.2. Figure 5.2(b) shows the SEM image of such a double-punch, with $w \sim 22.5$ $\mu$m and $s \sim 4.5$ $\mu$m.

### 5.2.2 Compression molding experiments

The so-fabricated single punches and double-punches were then used to conduct instrumented compression molding on one commercial, $<110>$ oriented, single crystalline Al specimen, polished to a mirror finish. All compression molding experiments were carried out using the same Nanointenter XP system (MTS Systems Corp., Knoxville, TN) and repeated at least four times at room temperature. In Fig. 5.3(a), the black curve shows a typical total compression force, $P$, vs. total punch displacement, $\Delta_{\text{total}}$, curve using the single rectangular strip punch shown in Fig. 5.2(a). This raw $P$-$\Delta_{\text{total}}$ curve exhibits an initially rapidly rising and approximately linear portion, followed by a bend-over. After the bend-over, another approximately linear portion is observed with a much smaller slope as compared to the initial portion. Qualitatively similar $P$-$\Delta_{\text{total}}$ curves were obtained at other $w$ values, consistent with results in the literature [15]. The raw $P$-$\Delta_{\text{total}}$ curves include a load frame stiffness contribution. To determine the load frame stiffness contribution at each punch width, a method as described in literature was followed [15]. Briefly, separate $P$-$\Delta_{\text{total}}$ curves were collected using the same punch, with a minimal load applied such that little actual specimen penetration occurred on the surface of the Al specimen under SEM examination. These small-load curves were then averaged (e.g., shown as the red curve in Fig. 5.3(a)), and then fitted with a straight line (e.g., the blue curve in Fig. 5.3(a)). The slop of this
fitted line was taken as the average load frame stiffness at this punch width. Then the actual punch penetration depth, \( \Delta \), was obtained by subtracting the average load frame stiffness from the recorded total punch displacement, \( \Delta_{\text{total}} \). Figure 5.3(b) shows such a \( P-\Delta \) curve obtained from the raw \( P-\Delta_{\text{total}} \) curve shown in Fig 5.3(a).

Fig. 5.3 (a) The black curve shows a typical \( P-\Delta_{\text{total}} \) curve obtained from the single rectangular strip punch shown in Fig. 5.2 (a), the red curve shows the averaged load frame stiffness curve, and the blue curve is the linear fit of the red curve; (b) the obtained \( P-\Delta \) curve after subtracting the load frame stiffness

Repeat \( P-\Delta \) curves obtained at each punch width were averaged together. The nominal contact pressure, \( p \), was obtained by normalizing the total force \( P \) by the nominal contact area, \( A \), i.e., punch width×length. The punch indentation depth was normalized by the punch width, \( w \). This normalization of the indentation depth was shown to enable quantitative comparison of molding response data obtained from punches of different dimensions [17]. All the molding response curves obtained using single punch compression and double-punch compression were processes following the same data processing procedure outlined above. It is noted that, as each double-punch data set includes two identical single rectangular punches, the total force \( P \) was normalized by the sum of the contact areas from both punches to obtain the nominal contact pressure. Likewise, the punch indentation depth was normalized by \( 2w \) for double-punch data sets.
5.3 Results and discussion

5.3.1 Single punch molding and size effect

Figure 5.4(a) summarizes the averaged compression molding response curves, $p$-$\Delta/w$, for the single crystalline Al specimen indented with single rectangular strip punches with widths $w$ decreasing from ~13 µm to ~1 µm. These $p$-$\Delta/w$ curves are qualitatively similar, possessing an initial almost vertical section, where $p$ increases sharply with increasing $\Delta/w$, followed by a “knee” after which $p$ increases more slowly in an approximately linear fashion as $\Delta/w$ increases further. It is clear that the molding response curves at $w$ values of 13 µm, 8 µm, and 4 µm are almost on top of each other. As $w$ further decreases from 4 µm to 1 µm, the molding response curves become stiffer, indicating that the characteristic molding pressure has an explicit and significant dependence on $w$, contrary to continuum mechanics expectations and indicative of a significant mechanical size effect. To better quantify this size effect, the nominal contact pressure at each punch width was taken and averaged at normalized indentation depth of 0.25. This averaged contact pressure was taken as the characteristic molding pressure and then plotted as a function of $w$, as shown in Fig. 5.4(b). The so-obtained characteristic molding pressure remains relatively flat when $w$ is greater than 4 µm, and increases sharply as $w$ decreases below 4 µm. The finding summarized in Fig. 5.4 is consistent with previous experimental observations [15].

To eliminate possible complications associated with this mechanical size effect in single punch molding, in subsequent molding runs, both single punch and double-punch were fabricated with $w > 4$ µm.
5.3.2 Dependence of single punch molding response on the specimen in-plane orientation

Fig. 5.4 (a) averaged nominal contact pressure vs. normalized indentation depth at each single punch width. (b) Characteristic molding pressure as a function of single punch width.

Molding runs were carried out using the single rectangular strip punch shown in Fig 5.2(a), with a length ~22 µm and a width ~4 µm. After one set of repeat compression moldings was completed, additional molding runs were conducted with the <110> oriented single crystalline Al specimen rotated respectively by 21°, 45°, and 84°, keeping all other testing parameters unchanged. Figure 5.5(a) shows the SEM image of the indentation imprints made on the Al specimen, with varying sample rotation angles. Figure 5.5(b) plots the nominal contact pressure vs. normalized indentation depth curves at different sample rotation angles. It is evident that nearly identical
molding response curves were obtained, indicating that the in-plane sample rotation angle has minimal effect on the molding response of the single crystalline Al specimen.  

5.3.3 Dependence of the single punch molding response on aspect-ratio

Fig. 5.6 (a)(b)(c) SEM image of indentations made from the 22×22 μm punch, the 22×12 μm punch and the 22×4 μm punch, with aspect ratios of 1.0, 1.8 and 5.5, respectively. (d) The nominal contact pressure vs. normalized indentation depth curves at each aspect ratio.

Three single rectangular punches, with length×width of 22×22 μm, 22×12 μm, and 22×4 μm, were used to test if the punch aspect ratio has significant effect on the molding responses. Figure 5.6 (a) (b) (c) show the corresponding indentations made from the above three single rectangular punches with aspect ratios of 1.0, 1.8 and 5.5. All the molded indentations have flat bottoms, straight sidewalls, sharp corners and sharp sidewall to bottom transitions, mimicking the features from the single rectangular punches. Figure 5.6 (d) shows the collected molding response curves and it is clear that these response curves are almost identical.
5.3.4 Double punch molding: molding response curves and incomplete filling

Figures 5.7(a), 5.7(b), 5.7(c), 5.7(d), and 5.7(e) summarize the double-punch molding response curves, measured using double-punches listed in Tables 5.1, 5.2, 5.3, and 5.4, respectively. All the molding response curves are qualitatively similar to those obtained from single punch molding, i.e., each curve includes an initial section where $p$ increases rapidly with increasing indentation depth, followed by an abrupt turn-over after which $p$ increases slowly with increasing depth at a much smaller slope. Specifically, Fig. 5.7(a) summarizes the molding response curves obtained using a series of double-punches with a constant spacing of ~2 $\mu$m, while the width of the individual rectangular punch varied from 17 $\mu$m to 4.2 $\mu$m. Therefore, $\lambda$ varied from 0.12 to 0.48. Figure 5.7(a) shows that nearly identical molding response curves are obtained at $\lambda$ of 0.12 and 0.15, while as $\lambda$ further increases, the molding response curves become stiffer, i.e., a clear increase in molding pressure can be observed. Figure 5.7(b) shows the molding response curves obtained from four double-punches with a constant $\lambda$ of 0.2, while the spacing between the two rectangular punches changed from 1.0 $\mu$m to 2.0 $\mu$m, 3.2 $\mu$m, and 4.5 $\mu$m. The molding response curves is seen to depend on the spacing $s$, i.e., the nominal contact pressure $p$ increases as $s$ decreases from ~ 4.5 $\mu$m to ~2 $\mu$m. The value of $p$ at $s$ values of 1.0 $\mu$m and 2.0 $\mu$m are nearly identical. Figure 5.7(c) shows the molding response curves obtained from the three double-punches with a constant punch width $w$ of ~5 $\mu$m, while the $s$ values changed from 1.0 $\mu$m to 1.5 $\mu$m and 2.4 $\mu$m, with the corresponding $\lambda$ values of 0.20, 0.29 and 0.48. It is evident the response curves exhibit a similar trend as shown in Fig. 5.7(a), namely, $p$ increases with increasing $\lambda$ values. Figure 5.7(d) shows the molding response curves using the last three double-punches with a constant $w$ of ~5 $\mu$m, with varying $s$ values of 3.50 $\mu$m, 5.2 $\mu$m, and 7.5 $\mu$m, and corresponding $\lambda$ values of 0.67, 1.0, and 1.50, respectively. Now a clear and opposite trend can be observed, that the contact pressure begins to decrease as $\lambda$ increases. Figure 5.7(e) summarizes the molding
response curves obtained from another three double-punches with $\lambda$ varying from 1.0 to 2.2. A similar trend as shown in Fig. 5.7(d) was observed, in that the measured $p$ decreases as $\lambda$ increases, and the molding response curves of $\lambda$ being 1.8 and 2.2 are almost on top of each other.

![Graphs showing double-punch molding response curves](image)

Fig. 5.7 (a) (b) (c) (d) (e): double-punch molding response curves, measured using double-punches listed in Tables 5.1, 5.2, 5.3, and 5.4, respectively.
The characteristic molding pressure was obtained from all the double-punch molding runs and plotted as a function of $\lambda$, as shown in Fig. 5.8. The error bar on the characteristic pressure stems from averaging results from several repeated molding runs. For comparison with the single rectangular punch molding, the averaged characteristic compression pressure, $\sim$330 MPa, obtained from single punch molding runs with $w$ being 4, 8, and 13 $\mu$m, is plotted as a base line. The legend in the graph corresponds to the double-punch sets listed in Tables 5.1-5.4.

![Graph showing characteristic molding pressure vs. $\lambda$](image)

Fig. 5.8 The characteristic molding pressure vs. $\lambda$

The five black data points are obtained from the response curves in Fig. 5.7(a). It shows clearly that the characteristic pressures at $\lambda \sim$0.12 is $\sim$370 MPa, $\sim$12% higher than the base line. The characteristic pressure changes little as $\lambda$ increases from 0.12 to 0.15, and then jumps to $\sim$395 MPa at $\lambda \sim$0.20, about $\sim$20% higher than the base line, and keeps increasing to $\sim$423 MPa at $\lambda \sim$0.31, $\sim$28% higher than the base line, and reaches to $\sim$442 MPa at $\lambda \sim$0.48, $\sim$34% higher than the base pressure.

The five red data points stem from the response curves shown in Figs. 5.7(c) and 5.7(d). It is evident that the measured characteristic pressure increases from $\sim$395 MPa to $\sim$450 MPa as $\lambda$ increases from 0.20 to 0.48. Moreover, at each $\lambda$, the measured pressure is very close to their black counterparts. This characteristic pressure tends to decrease after reaching the maximum value of
~450 MPa, and goes down to ~400 MPa at $\lambda = 1.5$. The data points obtained from Fig. 5.7(e) are plotted in the right side in Fig. 5.8. The characteristic pressure reduces gradually as $\lambda$ increases from 1.0 to 2.2, showing a trend that $p$ would approach the base line once the $\lambda$ value is sufficiently large such that a double-punch molding could be seen as molding with two individual punches, without any interaction between them.

If we focus on the four data points at $\lambda \approx 0.20$, taken from the response curves shown in Fig. 5.7(b), the black and red points are almost on top of each other, while the two blue points lie below. Because each single punch in all the double-punch sets possesses a width greater than 4 $\mu$m, a $w$ value deliberately chosen to eliminate possible size effects as exhibited in Fig. 5.4(b), and $\lambda$ is fixed to be $\sim 0.20$, therefore, the discrepancy in the measured characteristic pressure can only be attributed to the absolute size of the spacing between the two punches, $s$. In other words, the measured characteristic pressure is not only $\lambda$ dependent, but also $s$ dependent when the value of $s$ becomes sufficiently small.

Figures 5.9(a), 5.9(b), and 5.9(c) show the SEM images of three molded features with punch penetration depths ~3.5 $\mu$m, ~8.8 $\mu$m, and ~11.8 $\mu$m, using the same double-punch with $w = 22.5$ $\mu$m and $s = 4.5$ $\mu$m, as shown in Fig. 5.2(b). All three indentations have flat bottoms, sharp corners, and clean sidewalls. In Fig. 5.9(a), the molded Al didn’t flow into the recess between the two punches, and only a shallow bump was formed with sharp edges. This incomplete filling persisted as the punch penetration depth increased, as shown in Figs. 5.9(b) and 5.9(c). Though the overall bump height increased somewhat with increasing penetration depth, the bump height at the center of the indent is still only $\sim 10\%$ of the total molding depth. Similar phenomena can be observed using double-punches with $\lambda < 0.20$, indicating that the Al specimen molded by the
two rectangular punches separated by a spacing in between behaves almost as if it is compressed by a single punch, with a width of \(2w+s\).

![SEM images of morphology of three molded features](image)

Fig 5.9 SEM images of morphology of three molded features with penetration depth ~3.5 µm, ~8.8 µm and 11.8 µm, using the same double-punch set as shown in Fig. 5.2(b).

![SEM images of double-punch sets and corresponding molded features](image)

Fig 5.10 SEM images of double-punch sets and corresponding molded features. (a) \(w=5\) µm, \(s=1.0\) µm (b) \(w=5.2\) µm, \(s=3.5\) µm (c) \(w=5.2\) µm, \(s=5.2\) µm. (d) (e) (f): Molding features from (a) (b) (c).

Figures 5.10(a) – 5.10(f) show the SEM images of three double-punches with nearly the same punch width of 5 µm, and varying spacing of 1.0 µm, 3.5 µm, and 5.2 µm, as well as typical morphologies of features molded by these double-punches. As shown in Fig. 5.10(d), at \(s \sim 1.0\) µm, the molded Al did not fill the gap between the two individual punches, forming only a bump.
in between. This phenomenon is consistent with the results shown in Figs. 5.9(a)-5.9(c), and the bump height at the feature center is less than 10% of the total punch penetration depth. This incomplete filling remains as \( s \) increases to 3.5 \( \mu \text{m} \). In this case, however, the height of the bump increased, reaches \( \sim 60\% \) of the total penetration depth, as shown in Fig. 5.10(e). As \( s \) increases to 5.2 \( \mu \text{m} \), this incomplete filling almost vanished, as shown in Fig. 5.10(f).

5.4 Summary

Single punch molding and double punches molding were conducted on single crystal Al. The measured single punch molding response was consistent with the results in literature, i.e., the characteristic pressure exhibited a significant dependence on the punch width as width values decreased from \( \sim 4 \mu \text{m} \) to \( \sim 1 \mu \text{m} \). Therefore, to avoid the complication caused by such a mechanical size effect, all the double punches were cut with width greater than \( \sim 4 \mu \text{m} \). For the double-punch experiments, measured characteristic molding pressure exhibited a significant dependence on the spacing to punch width ratio, \( \lambda = s/w \), as well as a significant dependence on \( s \) when \( \lambda \) was fixed. The molded features were examined and the phenomenon of incomplete filling was observed to occur at \( \lambda < 0.5 \). These results shed light on general design considerations for pattern replication by molding, and illustrate the peculiarities of materials’ response when molding replication is pushed to micron dimensions.

5.5 References


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CHAPTER 6. SUMMARY

This dissertation focuses on meso/micro scale mechanical size effects in micro metal forming operations, including microscale reverse extrusion and compression molding. The experimentation offered new and basic data for micro metal forming technologies. The combination of experimentation and finite element analysis simulations offered new insight into physical factors controlling metal forming at the meso to micro scales, and present test cases for further development of micron scale plasticity theories and models.

Uniaxial compression testing on meso/micro scale Al ring and pillar specimens verified the existence of a softening effect as the specimen external dimension decreases to the sub-mm scale, qualitatively consistent with the surface layer model. In contrast to the surface layer model, however, it is shown that the thickness of the surface layer is not necessarily coupled to the average grain size, and rather exhibited a significant dependence on the external specimen size. This fact calls for a more definitive way to distinguish the surface layer from the bulk. Together with the data generated from nano/micro scale pillar compression tests, the compression flow stress of Al was plotted as a function of characteristic specimen dimensions in a master curve fashion. We believe similar master curves exist for other metallic materials, including BCC, FCC and HCP metals. This provides a baseline for delineating the deformation mechanisms that are truly important to metal forming operations.

Different from the uniaxial compression or tensile testing in which most of the specimens’ surfaces are free of constraints, many micro forming operations involve more constraints to the surfaces of the specimens, a fact that will complicate the prediction of the mechanical response and deformation behaviors during metal forming processes. The microscale reverse extrusion testing conducted on both Al and Cu specimens with varying average grain sizes serves as a
convincing example. During micro reverse extrusion, though the texture evolution of Cu with a fixed average grain size showed good agreement with that corresponding to macroscale deformation, the mechanical response of Cu and Al exhibited a different trend with respect to grain size variations, inconsistent with conventional Hall-Petch scaling. These observations point to deviations from continuum mechanics/plasticity in meso/micro metal forming, and need to be better understood before adequate models guiding micro metal forming operations can be achieved.

Compression molding is a promising approach to fabricate nano/micro scale surface features. The compression molding experiments using double-punch sets with varying punch widths and spacings revealed several important issues that were not addressed before. The phenomena of incomplete filling was observed, and could be diminished by reducing the ratio of spacing to width. These experiments gives guidance in design and operation of the micro molding processes.
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VITA

Bin Zhang obtained his B.S. degree in 2010 from the Department of Materials Science and Engineering of Shandong University, China. He obtained his M.S. degree in 2013 from the Department of Materials Science and Engineering at Shanghai Jiao Tong University, China. In January 2014 he joined the Mechanical Engineering Program at LSU as a Ph.D. candidate. His research mainly focuses on microscale metal forming, including meso/micro scale mechanical size effects, micro reverse extrusion, compression molding and roll molding.