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Micro-Mechanical Assessment of the Local Plastic Strain Invoked During a Splined Mandrel Flow Forming Operation

Meysam Haghshenas
The University of Western Ontario

Supervisor
Dr. Robert J Klassen
The University of Western Ontario

Graduate Program in Mechanical and Materials Engineering

A thesis submitted in partial fulfillment of the requirements for the degree in Doctor of Philosophy

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MICRO-MECHANICAL ASSESSMENT OF THE LOCAL PLASTIC STRAIN INVOKED DURING A SPLINED MANDREL FLOW FORMING OPERATION

(Thesis format: Integrated Article)

by

Meysam Haghshenas

Graduate Program in Engineering Science
Department of Mechanical & Materials Engineering

A thesis submitted in partial fulfillment of the requirements for the degree of Doctor of Philosophy

The School of Graduate and Postdoctoral Studies
Western University
London, Ontario, Canada

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Abstract

Splined Mandrel Flow Forming (SMFF) is a metal spinning operation that involves the application of high multiaxial compressive stress states to invoke large plastic flow in the work piece. This allows for essentially one-step fabrication of complex internally-splined shapes. In this research project, the equivalent plastic strain, invoked throughout bcc (1020 steel) and fcc (5052 and 6061 aluminum alloys, pure copper, and 70/30 brass) samples, that were made by SMFF, was measured. The objective of the research were to measure the to obtain data on the effect of microstructure and mechanical parameters on the flow formability of ductile bcc and fcc metal work pieces. To address this objective the equivalent plastic strain in the high strain regions of the flow formed metal parts were measured with the use of Micro/nano-indentation hardness measurements. Also, micro/nano-indentation tests were performed, on the same alloys the metal alloys which were used in the SMFF experiments, to assess the effect of pre-exist plastic strain, strain rate, and deformation volume on the operative deformation mechanisms. These parameters were found to depend upon the microstructure and the associated deformation mechanisms.

Data from indentations tests at constant loading rate and constant strain rates on a variety of ductile metals/alloys were used to determine the effect of dislocation type (i.e. “statistically stored”, and “geometrically necessary”), stacking fault energy, and activation volume. This accounts for the observed strain rate sensitivity and the depth dependence of the indentation stress. It also affects the local strain magnitude and gradient during the SMFF process.

This micro/nano-indentation based test technique allows one to then obtain data from which to validate calculated equivalent plastic strain distributions derived from numerical simulations and, for end users of a metal forming technique, allows one to understand and quantify the mechanical properties of the formed work piece.

Keywords: Splined Mandrel Flow Forming; equivalent plastic strain; micro/nano-indentation; FCC; BCC; Indentation stress; Indentation strain rate; Constant loading rate; Constant strain rate; Thermal activation energy.
Co-Authorship Statement

The research experiments were designed and executed by the candidate. The SMFF tests were performed at TransForm Automotive (TFA) in London, Ontario, while the indentation tests and microstructure studies were performed at Western University. The candidate analysed all the data which involved calculating the equivalent plastic strain, $\varepsilon_p$, and indentation strain rate, $\dot{\varepsilon}_{ind}$, and correlating these parameters to SMFF operating parameters and calculating fundamental deformation parameters like activation volume $V^*$, mechanical work $\Delta W$, and thermal activation energy of dislocations $\Delta G_0$.

The theories explaining the experimental data trends were obtained by discussion with co-authors, L. Wang, J.T. Wood, and R.J. Klassen. Co-authors also edited the articles prior to submission for journal/conference publication.
There are some things that cannot be learned quickly, and time, which is all we have, must be paid heavily for their acquiring. They are very simplest things, and because it takes a man's life to know them, the little new that each man gets from life is very costly and the only heritage he has to leave.

Ernest Hemingway
Dedication

I dedicate this to my father and mother,

_Nowzar Haghshenas and Giti Yoosefi_

and

My Spouse,

_Mehrnoosh_
Acknowledgments

I wish to express my sincere gratitude to my supervisor Professor Robert J. Klassen for his time, support, guidance and ideas provided throughout this project. Without his help, the successful completion of this thesis and the quality of work achieved would not have been possible. His contributions and help are greatly appreciated.

I also wish to thank my past and present colleagues whom I worked with at our Micro Mechanical testing laboratory and Western Machine Shop Services, especially Mr. Brandon Vriens, Dr. Richard O. Oviasuyi, Dr. Vineet Bhakhri, Mr. Chris Vandelaar and all those who have been involved with this project one way or the other for their time and support.

Last but not least, I would like to express my thanks to Ontario Center of Excellence (OCE) and TransForm Automotive (TFA) for supporting this project, financially. Great helps and suggestions by Mr. Robert Thompsons, and Dr. Tom Meier at TFA are truly appreciated.
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<td>Splined Mandrel Flow Forming</td>
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<td>Yield Stress</td>
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<td>Strain hardening rate</td>
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<td>SFE</td>
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<td>UTS</td>
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\[ \tau \quad \text{Shear stress} \]
\[ k_T \quad \text{Hall-Petch slope for twinning} \]
\[ k_S \quad \text{Hall-Petch slope for slipping} \]
\[ c \quad \text{Pile-up or Sink-in factor} \]
\[ A_{\text{ind}} \quad \text{Indentation projected area} \]
\[ H_V \quad \text{Vickers hardness} \]
\[ \varepsilon_r \quad \text{Indentation representative strain} \]
\[ \dot{j}_{\text{ind}} \quad \text{Effective average indentation shear strain rate} \]
\[ ILR \quad \text{Indentation Load Relaxation} \]
\[ CLR \quad \text{Constant Load Rate} \]
\[ CSR \quad \text{Constant Strain Rate} \]
\[ h^* \quad \text{Characteristic length in the Nix/Gao model} \]
\[ H_0 \quad \text{Indentation hardness for a large indentation depth} \]
\[ \mu \quad \text{Elastic shear modulus} \]
\[ \rho_t \quad \text{Total dislocation density} \]
\[ b \quad \text{Burgers vector} \]
\[ \tau^* \quad \text{Thermal component of the stress} \]
\[ \tau_i \quad \text{Athermal component of the stress} \]
\[ \rho_m \quad \text{Mobile dislocations density} \]
\[ v \quad \text{Frequency of atomic vibration} \]
\[ \beta \quad \text{Dimensionless constant} \]
\[ \Delta G_{\text{thermal}}(\tau) \quad \text{Thermal activation energy} \]
\[ T \quad \text{Absolute temperature} \]
\[ TR \quad \text{Thickness reduction} \]
\[ t_i \quad \text{Starting thickness} \]
\[ t_f \quad \text{Final thickness} \]
\[ CR \quad \text{Circumferential-Radial} \]
\[ LR \quad \text{Longitudinal-Radial} \]
\[ \omega \quad \text{Rotational speed} \]
\( \varepsilon_p \)  
von-Mises equivalent plastic true strain

*SEM*  
Scanning Electron Microscope

*PLC*  
Portevin-Le Chatelier Effect

\( d\varepsilon_p(x)/dx \)  
Equivalent plastic strain gradient

\( \varepsilon_{p_{\text{max}}} \)  
Maximum equivalent plastic strain

\( \text{Var}(\varepsilon_p) \)  
Point-to-point variation in the equivalent plastic strain

*MT*  
Mechanical twin

*AT*  
Annealing twin

\( \bar{\varepsilon}_p(x_i) \)  
Predicted value of local equivalent plastic strain from the second-order polynomials that were fitted to the data profiles

\( h_{\text{crit}} \)  
Critical indentation depth

\( A_{\text{actual}} \)  
Actual contact area

\( A_{\text{ideal}} \)  
Ideal projected area

\( E_r \)  
The reduced elastic modulus

\( P_{\text{Hert.}} \)  
Hertzian elastic contact force

\( E \)  
Young modulus

\( \Delta G_0 \)  
Activation strength of the deformation rate controlling obstacles

\( \Delta W' \)  
Apparent mechanical activation work

\( V' \)  
Activation volume

\( 1/\Delta a \)  
Inverse activation area

*ISE*  
Indentation size effect

\( \rho_{\text{SSD}} \)  
Density of geometrically necessary dislocations

\( \rho_{\text{GND}} \)  
Density of statistically stored dislocations

\( l \)  
Intrinsic length scale
Chapter 1
Overview

1.1 Introduction
Parts manufacturers, especially those in the automotive industry, are designing complex metal components and are therefore looking for the fastest and most efficient way to produce complex shapes.

Flow forming has gradually matured as a metal forming process for the production of engineering parts in small to medium batch quantities in last two decades. Advantages such as flexibility, simple tooling and low forming loads, have made the flow forming as a promising process to optimise designs and reduce weight and cost, all of which are vital, especially in the automotive industries.

Flow forming-made parts appear to be an attractive alternative to press formed parts especially with its lower forming load requiring considerable smaller equipment and more flexible tooling as compared to conventional forming processes.

Flow forming is commonly known as a process for transforming flat sheet metal blanks, usually with axi-symmetric profiles, into hollow shapes by a tool which forces a blank onto a mandrel, as illustrated in Fig. 1.1. The blanks are clamped rigidly against the mandrel by means of a tailstock and the shape of the mandrel bears the final profile of the desired product. During the process, both the mandrel and blank are rotated while the spinning tool contacts the blank and progressively induces a change in its shape according to the profile of the mandrel.

As the tool is applied locally on the work piece, the total forming forces are reduced significantly compared to conventional press forming. This not only increases the possibilities in terms of large reductions and change in shape with less complex tooling, but also reduces the required load capacity and cost of the forming machine. In addition, flow forming is also known to produce components with high mechanical properties and smooth surface finish.
For precision flow forming operations, typically three rollers placed with 120° design is used. These rollers have pre-calculated radial and axial offsets between each other to achieve necessary forming conditions. The rollers force the metal blank over a cylindrical mandrel (smooth mandrel or splined mandrel). In the splined mandrel flow forming (SMFF), the metal blank is forced to conform to the shape of the mandrel cylinder including flowing into, and filling, the splines. The components fabricated in such a way are dimensionally accurate replications of splined mandrel that require only minimal secondary machining.

![Figure 1.1: Schematic representation of three roller flow forming process.](image)

As the use of flow forming to create internally-ribbed automotive components is a relatively new field of manufacturing, the interrelation of various flow forming parameters is not completely known. For example, very high local plastic strain occurs in the steel work piece as it flows into, and fills, the mandrel splines during a flow forming operation and this ultimately limits the type of parts that can be manufactured by the
process and the tool life (particularly the life of the splined mandrel). It is essential to optimize the flow forming process parameters in order to ensure that a high quality product is produced without generating unnecessary tool wear.

1.2. Rationale for Research

The production of SMFF’d components with complex geometries is limited by an industry-wide lack of knowledge of the local stress and plastic strain that arises within these parts and within the tooling during fabrication. SMFF process produces a complex and highly variable state of triaxial strain in the work piece with large strain gradients occurring in the complex region of several microns from surface. This results in increased rates of rejected parts and increased tool wear. In this research, the effect of average work piece thickness reduction as a controllable process variable on the local stress state and plastic flow in the vicinity of a splined mandrel of SMFF’d parts made from bcc (1020 steel) and fcc (6061, and 5052 aluminum alloys, pure copper, and 70/30 brass) metal alloys was studied. Strain hardening rate, maximum local equivalent plastic strain, and the grain-to-grain variability in the equivalent plastic strain distribution in these parts is considered in detail by means of micro/nanoindentation testing. The outcome of this investigation is data that will allow the identification of practical operating bounds for the SMFF process.

Manufacturers who plan to use SMFF need to understand the formability bounds of the process. They therefore require empirical relations that describe work piece/mandrel failure rates as a function of the controllable process variables and have the ability to predict failure rates under conditions that are beyond the current conventional practice.

Measurement of the local plastic strain within the interior of thick work pieces that have been deformed into irregular shapes, such as what occurs in SMFF, are made by post-test sectioning of fabricated parts. The local plastic strain is then deduced from either local changes in grain shape or local changes in indentation hardness within the part. Non-destructive techniques such as neutron diffraction can measure the internal elastic strain within such parts however this technique has limited spatial resolution and does not give information on the state of plastic strain.
The aim of the present research is to assess the effects of microstructure, and associated deformation mechanism (\textit{i.e.} dislocation slip and deformation twinning) on the practical flow formability of a metal. These mechanisms are highly dependent upon applied strain, pre-existing plastic strain, and volume of deforming metal. The effect of these variables cannot be assessed directly from a SMFF’d sample. Thus, in this thesis considerable micro/nano-indentation tests at various regions, depths, and strain rates to perform these assessments are performed.

1.3. Objective

My PhD research topic is directed to obtaining a detailed understanding of the global and the local plastic strains and failure criteria that develop during SMFF operations involving complex mandrel shapes and tool paths performed on fcc metal alloy (6061, and 5052 aluminum alloys, brass 70/30, and pure copper) and bcc (1020 steel) work pieces. Micro-level plastic strain measurements performed on these work pieces under different levels of the forming variables (\textit{i.e.} mandrel rotational speed, roller feed rate, and thickness reduction) are identified. The objectives of the project are:

- Assessment of the equivalent plastic strain within the work pieces. Using micro-indentation hardness, plastic deformation is measured at different areas of made samples under different forming conditions.
- Analysis of the effect of microstructure and mechanical parameters (yield stress ($\sigma_y$), rate of work hardening ($\theta$), stacking fault energy, and grain shape) on the local equivalent plastic strain within a SMFF’d parts.
- Consider the effect of deformation mechanisms under constant load rate and constant strain rate during micro/nano-indentation of the metal alloys which were used in the SMFF experiments.

1.4. Structure of Thesis

This thesis has been written following the guidelines of the School of Graduate and Postdoctoral Studies at Western University adopting an integrated-article format. It contains 6 chapters. Chapter 1 of the thesis includes an introduction on the splined mandrel flow forming techniques, the rationale and the objectives of the research.
Chapter 2 contains a review of relevant published literature on the flow forming process and the mechanisms of plastic flow at room temperature of the ductile fcc and bcc materials that will be flow formed in this thesis. This chapter continues a description of the indentation test technique and the interpretation of the plastic strain data arising from such tests.

Chapter 3 is an integration of three published papers on the plastic strain distribution profiles, and work hardening behaviour during splined-mandrel flow forming of bcc alloy (1020 steel), and four fcc alloys (5052, and 6061 aluminum alloys, 70/30 brass, and pure copper). The three papers were published in *Materials & Design* [1] and *Materials Science and Engineering A* [2, 3]. The paper published in *Materials & Design* entitled “Plastic strain distribution during splined-mandrel flow forming” dispute compresses plastic strain distribution of the 1020 steel work pieces subjected to SMFF with different levels of average work piece thickness reduction, from 20 to 60%. A part of this work was presented and published in the Proceedings of International Deep Drawing Research Group (iddrg 2010), Graz, Austria [4]. The two published papers in *Materials Science and Engineering A* are entitled “the investigation of strain-hardening rate on splined mandrel flow forming of 5052 and 6061 aluminum alloys” [2] and “effect of strain-hardening rate on the grain-to-grain variability of local plastic strain in spin-formed fcc metals” [3]. In the first paper, the role of solid solution strengthening additions, *i.e.* Mg in 5052 aluminum alloy, on increasing the average mechanical strength but also increasing the extent of local plastic strain variability in aluminum alloy material subjected to intensive plastic forming operations such as SMFF. The later article on the SMFF of pure copper and 70/30 brass, the deformed microstructure of the formed work pieces indicated that considerably more grain-to-grain variability in the dislocation slip step and deformation twin densities exist in the material with a high strain-hardening rate. These findings are of considerable importance and should be considered when assessing the suitability of high strain forming processes for producing reliable, and homogeneous, parts from fcc metal alloys that display high strain hardening rates.

Chapter 4 is an integration of three papers on the depth dependence and strain rate sensitivity of indentation stress during constant loading rate and constant strain rate of SMFF’d alloys during Nano/micro-indentation. The first paper was published in
Materials Science and Technology [5], the second paper entitled “characterization of depth dependence of indentation stress during constant strain rate nanoindentation of 70/30 brass” was published in Materials Science and Engineering A. The third paper entitled “Microindentation-based assessment of the dependence of the geometrically necessary dislocation upon depth and strain rate” has been accepted in MRS 2013 proceedings.

The paper published in Materials Science and Technology investigates depth dependence and strain rate sensitivity of indentation stress of 6061 aluminium alloy. Constant load rate microindentation on fully annealed, partially aged (T₄), and fully aged (T₆) of 6061 aluminum alloy were investigated in this paper [5].

The second paper reports the results from nanoindentation tests that was performed under constant strain rate conditions to investigate the effect of $\dot{\varepsilon}_{\text{ind}}$ upon the indentation depth dependence of $\sigma_{\text{ind}}$ for annealed and cold worked 70/30 brass [6]. It was observed that parameters, such as the strain rate sensitivity $m$, and the apparent activation energy $\Delta G_0$ showed a clear dependence upon indentation depth but the magnitude of these parameters, which were obtained under constant strain rate conditions, were the same as equivalent values obtained by others under constant load rate conditions. This suggests that valid measurement of thermally-activated parameters can be obtained in common metals by performing micro-indentation tests under constant $P$ conditions. At any given indentation depth, $P$ increases with increasing the strain rate ($0.5\dot{P}/P$).

The last paper in chapter 4, accepted to be published at the MRS 2013 proceedings [7], deals with the room–temperature load–controlled pyramidal microindentation tests on the fcc annealed alloys of 70/30 brass, pure copper, and 5052 aluminum alloy samples. In this paper, by means of Nix and Gao model, the size effect during indentation tests was studied and the density of statistically stored dislocations (SSDs) and geometrically necessary dislocations (GNDs) were obtained. GNDs showed an inverse relationship with depth, $h$. Using an Arrhenius law, which describes the successful surmounting of an obstacle by thermal activation of the dislocation segments, as well as the Nix/Gao model, characteristic parameters of dislocation motion such as activation volume and activation energy were calculated.
Chapter 5 of this thesis combines the findings from the published papers in Chapters 3 and 4 into a discussion of the effect of work hardening rate, stacking fault energy (SFE), twinning and/or various dislocation glide mechanisms on the local grain to grain variability in plastic strain during high strain metal forming operation (such as SMFF). In this chapter also the strain rate sensitivity, activation volume and activation energy (measured from Nano/Micro-indentation) of SMFF’d metal alloys are compared discussed in detail.

The thesis ends in Chapter 6 with a short summary of the main findings and contributions of this research and suggestions for future research.
1.5. References


Chapter 2
Review of the relevant literature

In this chapter, at first, the Splined Mandrel Flow Forming (SMFF) process is overviewed (section 2.1). The effect of flow forming variable parameters on the finished part under different conditions is reviewed in the Section 2.2. The concepts of flow formability (spinnability) and analytical/experimental techniques for studying the flow forming are reviewed in the Sections 2.3 and 2.4. In Section 2.5, the mechanisms of plastic deformation of polycrystalline materials \((i.e.\) slipping and twinning) are addressed. Pyramidal indentation testing, the plastic strain resulting during nano/micro-indentation, indentation size effects, and thermal activation of dislocations during the indentation process are covered in Sections 2.6–2.10.

2.1. Process Overview

This section is aimed at reviewing the flow forming fabrication technique. Published literature related to smooth mandrel flow forming is reviewed to provide an important background for my current research on splined mandrel flow forming (SMFF).

Spinning, shear forming, and flow forming (as presented in Fig. 2.1) can all be thought of as “chipless turning” technologies because there is no material removed during the process and, hence, metal shaping occurs exclusively by plastic deformation of the work piece. Any ductile material can, in principal, be formed by these operations and examples exist in the literature of such operations performed on work pieces of various grades of steel, including stainless steel, and non-ferrous metals such as titanium, aluminum, bronze and copper \([1, 2]\).

Flow forming operations performed over cylindrical mandrels produce a seamless cup-like product with very tight dimensional tolerance. A metal blank or preform is formed over a rotating mandrel. The metal blank and the mandrel (which are locked together) rotate, and the forming roller follows the mandrel at a preset. The large compressive forces exerted by the rollers cause the work piece to deform plastically. The deformed metal takes the shape of the mandrel, and proper wall thickness is achieved by control of the gap between the forming rollers and the mandrel.
This forming technique offers significant advantages in comparison with other conventional production techniques. These advantages are particularly pronounced when components are to be produced in small or medium size batches due to lower tooling costs than other process such as deep drawing. Localized deformation of the material under the forming rollers also requires lower forming forces than techniques such as, for example, deep drawing and press forming. The other advantages are [3]:

- Very little wastage of material.
- Excellent surface finishes.
- Accurately dimensioned components.
- Improved strength properties.

Components made by flow forming (tube spinning) include parts for the automotive and aerospace industries, art objects, musical instruments and kitchenware [2]. The process is capable of forming components of diameters ranging from 3 mm to 10 m, and thickness of 0.4 to 25 mm [4].

2.1.1. Splined Mandrel Flow Forming (SMFF) process

SMFF is an effective technique for fabricating internally ribbed cylindrical parts. It involves pressing, with rollers, a spinning metal work piece, over a spinning shaped mandrel such that the work piece replicates the shape of the mandrel (Fig. 2.2). Perhaps the most important problem encountered during SMFF is unexpected mandrel failures resulting from high local forces exerted on the mandrel splines. Indeed, very high local plastic strain occurs in the work piece as it flows into, and fills, the mandrel splines. This ultimately limits the type of parts that can be manufactured by the process and the tool life (particularly the mandrel life time).

2.1.2. Deformation analysis in flow forming

The indentation volume of material that is being deformed plastically during a flow forming operation is very small and hence tremendous strain gradients develop through the thickness of a flow formed part. Figure 2.3 shows the complex force components that are exerted on the work piece by the forming roller during a flow forming operation.
In order to analyze the deformation of flow forming more clearly, the deformation area was classified by Xu et al. [5] according to the different deformation in the radial, tangential and axial directions of the work piece (Fig. 2.4).

Figure 2.1: Classification of spinning processes [2], a) conventional spinning, b) shear spinning, c) flow forming (tube spinning). In each figure the starting blank along with the final formed part is shown schematically.
Figure 2.2: The arrangement of the forming roller, the mandrel and the work piece during a single-roller SMFF.

Figure 2.3: Localized forces imposed by the forming roller during flow forming; $P_t$: tangential forces, $P_r$: radial forces, $P_z$: axial forces [5].
The advantages of the localized deformation during flow forming is that the power required is considerably lower compared to conventional press forming, thus enabling smaller equipment and tools to be used. Since the deformation area is limited to a part of work piece which is in contact to the forming roller, the deformation is strongly constrained by the surrounding work piece material. This results in a highly triaxial stress and plastic strain state to exist in the flow formed work piece.

The forces during flow forming, local plastic strains of magnitude several times greater than the failure strain during uniaxial tension have been observed to occur in the work piece without the onset of work piece failure [6]. This can be attributed to the triaxial stress state that is applied to the work piece.

Findings by Sugarova et al. [7] show that comparison of the strains in directions 0°, 45°, and 90° to the rolling direction of a flow formed work piece indicate that there is a minimal influence of work piece mechanical anisotropy on the flow forming process.

In a SMFF’d part, the material between the ribs is under the compressive strain in the radial direction and under the tensile strain in the axial and tangential direction (Fig 2.5). Based on a study by Shu-yong et al. [8], the material in the ribs is under tensile strain in the radial and axial direction and under the compressive strain in the tangential direction. The radial compressive strain and the tangential tensile strain in the material between the ribs contribute to the flow of the metal into the mandrel splines and ultimately promotes metal filling of the splines.
Even though the plastic strain state is the flow formed work piece is known to be complex and highly triaxial, little in the way of experimental data exist on the magnitude of the strain or of relation to the process parameters. The information that has been reported will be presented in the next sections.

2.2. Flow forming parameters
In any flow forming process there are a series of variables that can be independently controlled and which affect the overall process (Table 2.1). In the present thesis, the effects of thickness reduction as a process variable are considered.

Material thickness reduction can be defined as the overall reduction in work piece thickness that occurs over one complete flow forming pass.

2.3. Flow formability (Spinnability)
The flow formability of a material is defined as the maximum thickness reduction that the work piece can undergo before it fails. The effective parameters that influence the maximum thickness reduction before failure (flow formability) can be divided into two major categories. One category consists of mechanical factors like feed rate, roller tip radius, and roller attack angle. Another category includes the metallurgical factors such as mechanical properties of material, cleanness of alloy, and average grain size [9].
Table 2.1: Flow forming process variables

<table>
<thead>
<tr>
<th>Process</th>
<th>Machine</th>
<th>Working</th>
<th>Tooling</th>
<th>Work piece (Fixed)</th>
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</thead>
<tbody>
<tr>
<td>- Direction of flow</td>
<td>- Power</td>
<td>- Rotational speed</td>
<td>- Roller nose radius</td>
<td>- Starting ID</td>
</tr>
<tr>
<td>- Size of thickness reduction</td>
<td>- Coolant</td>
<td>- Axial feed</td>
<td>- Roller attack angle</td>
<td>- Wall thickness uniformity</td>
</tr>
<tr>
<td>- Number of reduction passes</td>
<td>- Machine stiffness</td>
<td>- Axial roller offset</td>
<td>- Number of forming rollers</td>
<td>- Material mechanical characteristics</td>
</tr>
<tr>
<td>- Working temperatures</td>
<td></td>
<td>- Radial roller offset</td>
<td></td>
<td>- Material flow curve stability</td>
</tr>
<tr>
<td></td>
<td></td>
<td>- Thickness reduction</td>
<td></td>
<td>- Material isotropy /anisotropy</td>
</tr>
</tbody>
</table>

Since flow formability is dependent on the ductility of the work piece, it can be predetermined using a material parameter related to the ductility and toughness.

Kegg et al. [10] proposed a method for deducing the flow formability from the tensile properties of a material as shown in Fig 2.6. Using a half ellipsoidal shaped smooth mandrel, they suggested that for materials having a tensile reduction of area of 80% or less, the maximum thickness reduction (i.e. the flow formability) is equal to or greater than the tensile reduction of area.

Figure 2.7 shows the relationship between the ultimate tensile strength and maximum thickness reduction in forward and backward flow forming operations. The graph indicates clearly that the maximum thickness reduction (i.e. the flow formability) decreases as the ultimate tensile strength of the metal increases.

Kalpakcioglu [11] extended the work of Kegg [10] by providing an analytical and experimental study of the flow formability. He reported that the forming roller corner radius, roller velocity and mandrel speed do not affect the flow formability of metals. On the other hand, the mandrel angle has a great effect on the state of stress in the work piece and thus its flow formability. He concluded that for metals with a true fracture strain greater than about 0.5, there is a upper limit to the maximum thickness reduction during a flow forming operation and further increase in the ductility of the work piece material does not increase the maximum thickness reduction. For metals having a true fracture strain below 0.5, the flow formability depends on ductility of the work piece.
Kalpakjian and Rajagopal [12] performed flow forming studies on a number of different metals with increasing thickness reductions until tensile failure (or failure in the deformation zone) occurred. Sections of these metals illustrating failure are shown in Fig. 2.8.
Figure 2.8: Sections of fractured samples from flow forming tests indicating degree of forward reduction permissible for (A) 2024-T4, (B) 6061-T6, (C) annealed copper, (D) mild steel [12].

Roy et al. [13] studied the flow formability of 1020 steel using a smooth mandrel. They calculated a critical thickness reduction level in the work piece and suggested that there is a maximum thickness reduction level at which the material can be flow formed and still remain defect free. This was found to occur at a reduction level between 51.8 and 52.9%. This is shown in Fig. 2.9, where the maximum strain at the roller interface versus reduction level shows an abrupt shift as the thickness reduction level passed between these two values. At reduction values beyond 51.8%, defects, in the form of localized cracking on the roller-side surface of the formed part were observed.
Figure 2.9: Maximum equivalent plastic strain incurred at the roller interface found from fitted relationships versus thickness reduction level. The strain increases substantially at a critical reduction level between 51.8 and 52.9% indicated by the vertical bar [13].

In this chapter measuring the flow-formability of the internally splined flow formed work piece under different flow forming conditions (parameters) is of interest. This will be done by monitoring the curves which show the relationship between maximum thickness reduction (formability) and equivalent plastic strain at different conditions.

2.4. Analytical techniques for studying the flow forming process

Theoretical techniques can provide insights into the evolution of stress and strain within a flow formed work piece. Four different analytical approaches have been identified in studies of flow forming: force prediction using the deformation energy method [14, 15] the upper bound method [16, 17], prediction of failure by wrinkling [18], and prediction of strains [13, 19-22]. In what follows recent findings related to experimental measurements of plastic strain invoked by flow forming operation are discussed.

Experimental techniques have been applied to study the mechanism of deformation and evolution of strains, to investigate failure mechanisms and predict failure, to predict forming forces and surface quality, and to optimise product geometry. However, other experimental techniques which have been applied elsewhere could be applied to obtain more information about flow forming, specifically about the stresses and strains...
generated in the process. Examples of such an application to other processes are a study by Chaudhri [23] who employed Vickers micro-indentation techniques to map the local equivalent plastic strain in the deformed region around a large spherical indentation made in copper, and that by Tseng et al. [24] who used a similar technique to map the equivalent plastic strain through the roll-bite region of cold rolled steel.

Most of the work, both early and more recent, has concentrated on prediction of forces in flow forming. This is an important issue in the design of flow forming machines. However the study of forces on its own does not produce knowledge which would lead to understanding of the process mechanics. The prediction of metal flow and tool forces ideally leads to a prediction of stresses, strains and therefore potential damage in the formed part.

To study the mechanism of deformation and the evolution of strains in spinning, three experimental approaches have been identified (surface etching pattern method [25], plugged hole method [26]) and hardness testing (micro-indentation) method [13, 19-22].

The plugged holes method consists of drilling holes in a spiral and radial pattern in the sheet blank and filling them with scrap material. After the sheet is spun, the holes are revealed by cutting the sheet, and examined to construct a three-dimensional picture of sheet deformation. A slightly different method was proposed by Kalpakcioglu [27] who applied the grid line technique to spinning to study the deformation through the sheet thickness. In this method, the sheet blank is cut in two, grid lines are inscribed on the exposed surface and the two parts are soldered together. After forming, the two parts are separated by melting and the two surfaces cleaned to study deformation through the sheet thickness. The third approach is that of Quigley and Monaghan [25] who studied strains in spinning by etching a pattern of circles of known size on the sheet surface before forming. The deformed circles are measured after forming with an optical projector to determine surface strains.

Roy et al. [13] employed micro-indentation hardness testing to map the true equivalent plastic strain through the thickness on the smooth-mandrel flow forming of 1020 steel. The work piece experienced increased plastic strain in subsequent forming passes with material near the mandrel and the roller displaying elevated equivalent plastic strain,
which was dependent upon thickness reduction, during the final forming stage. It was also observed that as reduction increased, the local plastic strain increased more rapidly at the roller interface than at the mandrel interface.

Hu et al. [19] recently have used micro-hardness testing (Vickers) to map the mechanical properties distribution through the thickness of Al–Cu–Mg alloy tube fabricated by friction stir welding and flow forming. They showed the same hardness distribution trend through the thickness of their welded flow formed aluminum tube as Roy et al. [13] showed in 1020 steel.

In chapter three of this thesis, microindentation technique are used to examine the effect of thickness reduction on the distribution of equivalent plastic strain within the internally ribbed regions of 1020 steel, 5052, and 6061 aluminum alloys, pure copper and 70/30 brass work pieces subjected to a SMFF operation. The plastic equivalent strain profile in the different regions around the internal ribs in the work piece are plotted to predict the critical strained areas in the flow formed samples. The maximum thickness reduction possible is ultimately decided by the formability of the work piece material.

In the present project, the mechanism of deformation and evaluation of equivalent plastic strain in the flow formed worked pieces using the microindentation testing are studied. With the proper micro-indentation hardness testing technique, successful translation of hardness values to equivalent plastic strain will allow me to plot the local equivalent plastic strain through SMFF’d work pieces made of fcc and bcc alloys and thereby assess the effect of material type on the formability. The difference of formability will of course be related to the operative mechanisms of plastic deformation in these metals. These various deformation mechanisms are described next.
2.5. Plastic deformation of polycrystalline metals

In this section the mechanisms of plastic deformation of SMFF’d polycrystalline metals/alloys –bcc (1020 steel) and fcc (5052, and 6061 aluminum alloys, pure copper, and 70/30 brass)– are reviewed. Since plastic deformation most often occurs by collective motion of line defects, dislocations, within the crystalline material, this section discusses the characteristics of dislocations and their involvement in plastic deformation.

2.5.1. Plastic deformation by dislocation glide

Plastics deformation, or plastic flow, can be envisaged as sliding, or successive displacements of one plane of atoms over another on so-called slip planes. This occurs by the motion of dislocations within the crystal when it is subjected to a shear stress.

The number of independent slip systems represents the different possible combinations of slip planes, upon which a dislocation can “easily” glide and energetically favourable directions upon these planes in which dislocations can move. The possible slip systems for bcc and fcc crystal structures are listed in Table 2.2.

Polycrystals are composed of many grains with different relative crystallographic orientation. When the bulk material is deformed, each individual grain undergoes slip. The stress at which slip begins in each grain depends on the orientation of the possible slip systems with respect to the tensile axis. The shape change in a plastically deforming grain may be constrained by neighbouring grains that have not yet reached their yield point. In addition, the grain boundaries, being regions of considerable atomic misfit, act as strong barriers to dislocation motion.

A fine-grained material (one that has small grains) is harder and stronger than one that is coarse grained, since the former has a greater total grain boundary area to impede dislocation motion. The yield strength $\sigma_y$ varies with grain size according to:

$$\sigma_y = \sigma_0 + k_y d^{-1/2}$$  \hspace{1cm} (2.1)

In this expression, termed the Hall-Petch equation, $d$ is the average grain diameter, and $\sigma_0$ and $k_y$ are constants for a particular material.
It should also be mentioned that grain size reduction improves not only strength, but also the toughness of many alloys [28]. Before deformation the grains are equiaxed, however, after deformation the grains become elongated along the direction in which the specimen was deformed. Also, a grain in a polycrystal is not free to deform plastically like a single crystal, since it must remain in contact with, and accommodate the shape changes of its neighbour grain. The result of all these factors is that the stress-strain curve for a polycrystalline material is different than the one for a single crystal. So that, depending on the grain orientation respect to the direction of the load applied to the material, some grains yield first, just when the resolved shear stress reach a critical value, and then other grains will follow progressively as the applied load increases.

Table 2.2: Slip Systems for Face-Centered Cubic and Body-Centered Cubic metals [28]

<table>
<thead>
<tr>
<th>Metals</th>
<th>Slip Plane</th>
<th>Slip Direction</th>
<th>Number of Slip Systems</th>
</tr>
</thead>
<tbody>
<tr>
<td>Face Centered Cubic</td>
<td>{111}</td>
<td>〈110〉</td>
<td>12</td>
</tr>
<tr>
<td>Cu, Au, Ni, Ag, Au</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Body Centered Cubic</td>
<td>{110}</td>
<td>〈111〉</td>
<td>12</td>
</tr>
<tr>
<td>α-Fe, W, Mo</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>α-Fe, W</td>
<td>{211}</td>
<td>〈111〉</td>
<td>12</td>
</tr>
<tr>
<td>α-Fe, K</td>
<td>{321}</td>
<td>〈111〉</td>
<td>24</td>
</tr>
</tbody>
</table>

2.5.2. Strain hardening

Strain hardening is often utilized commercially to enhance the mechanical properties of metals during fabrication procedures. Work (strain) hardening is the phenomenon whereby a ductile metal becomes harder and stronger as it is plastically deformed. Sometimes it is also called work hardening, or, because the temperature at which deformation takes place is “cold” relative to the absolute melting temperature of the metal, cold working.

Strengthening during deformation of a metallic material is obtained by increasing the number of dislocations. Before deformation, the dislocation density is about 10⁶ cm of dislocation line per cubic centimeter of metal [29]. By applying a stress greater that the yield stress, dislocations begin to slip. Upon dislocation slip, the strain hardening phenomenon is explained on the basis of dislocation–dislocation strain field interaction.
Eventually, a dislocation moving on its slip plane encounters obstacles that pin the ends of the dislocation line. By continuously applying the stress, a dislocation attempts to move by bowing in the center. The dislocation may move so far that a loop is produced. When the dislocation loop finally touches itself, a new dislocation is created. The original dislocation is still pinned and can create additional dislocation loops (Frank–Read source). Due to dislocation multiplication or the formation of new dislocations, the dislocation density may increase to about $10^{12}$ cm of dislocation line per cubic centimeter of metal during strain hardening.

Dislocation motion is, indeed, the cause for plastic flow that occurs in metallic materials. However, we have too many dislocations, they are positioned closer to each other and interfere with their own motion. As the dislocation density increases, the resistance to dislocation motion by other dislocations becomes more pronounced. Thus the imposed stress necessary to deform a metal increases with increasing plastic forming.

Figures 2.10 and 2.11 show the variation in yield and tensile strength with increasing the amount of cold work in 1040 steel, copper and brass. Since in most cold working processes one or two dimensions of the metal are reduced at the expense of an increase in the other dimensions, cold work produces elongation of the grains in the principal direction of working. Ductility, in percent elongation, experiences a reduction with increasing percent cold work.
Figure 2.10: The variation in a) yield strength, and b) tensile strength with cold work percent [28].

Figure 2.11: The decrease in ductility (%EL) with percent cold work for 1040 steel, copper and brass [28].
2.5.3. Plastic deformation by twinning

Plastic deformation in some metals can occur by the formation of mechanical twins, or twinning. During twinning, a shear force can produce atomic displacements such that on one side of a plane (the twin boundary), atoms are located in mirror image positions to the atoms on the other side (Fig. 2.12) [28]. Twinning occurs on definite crystallographic planes and directions that depend on the crystal structure.

Figure 2.13 shows a comparison between slip and twinning deformations for a single crystal that is subjected to a shear stress. Twining and slipping are different in several aspects. Firstly, for slip, the crystallographic orientation above and below the slip plane is the same both before and after the deformation; whereas for twinning, there will be a reorientation across the twin plane. Also, slip occurs in distinct atomic spacing multiples, whereas the atomic displacement during twinning is continuous (Figs. 2.12 and 2.13).

The amount of bulk plastic deformation from twinning is normally small relative to that resulting from dislocation slip. However, the real importance of twinning lies with the accompanying crystallographic reorientations; twinning may place new slip systems in orientations that are favorable relative to the stress axis such that the dislocation slip process can now take place. This will then result in marked decrease in the work-hardening rate.

![Figure 2.12: Twin formation by progressive shear of the parent lattice [30].](image-url)
Figure 2.13: Deformation in a single crystal subjected to a shear stress $\tau$ a) deformation by slip, b) deformation by twinning in a single crystal [28].

Twinning and dislocation slip are competitive deformation processes with, generally, slip dominating. However, at high strain rates (such as those encountered during shock loading) and/or at low temperatures, twinning can contribute significantly to the plastic deformation [31].

Based on studies by Meyers *et al.* [32], the experimentally observed *Hall–Petch* slope for the yield stress of a polycrystalline metal deforming primarily by dislocation slip is much less than that for a metal deforming by a twinning mechanism. Indeed, for coarse-grained metals, the stress required for activating twinning increases much faster with decreasing grain size than the stress for perfect dislocation slip. Figure 2.14 shows the larger grain size dependence of the twinning stress, as compared with the slip stress. For most cases, a *Hall–Petch* relationship is obeyed in twinning, but with a slope, $k_T$, that is higher than the one for slip, $k_S$. 
Figure 2.14: The schematic of Hall–Petch relationship for twinning and full dislocation slip in coarse-grained metals and alloys. $\tau$ is the shear stress and $d$ is the grain size. The higher slope for twinning indicates that twinning is more difficult than the slip of full dislocations in smaller grains [33].

2.5.3.1. Stacking Fault in fcc structures

The atomic arrangement on the \{111\} plane of an fcc structure could be obtained by the stacking of close-packed planes of spheres. For the fcc structure, the stacking sequence of the planes of atoms is given by $ABC ABC ABC$ (Fig. 2.15).

By applying plastic deformation “faults” in the stacking sequence can be produced [34]. Slip on the \{111\} plane in an fcc lattice produces deformation stacking fault. Indeed, slip occurs in the fcc lattice on the \{111\} plane in the \langle110\rangle direction and with a Burgers vector $a/2[110]$ which defines one of the observed slip direction, which can favourably energetically decomposed into two partial dislocations. That is, the same shear displacement produced by $b$ can be accomplished by the two step path $b_2+b_3$ (dashed arrows in Fig. 2.16a). The latter displacement is more energetically favourable but it causes the perfect dislocation to decompose into two partial dislocations. Slip by this two-step process creates a stacking fault $ABC AB AB ABC$ which is called extrinsic or
twin, stacking fault (Fig. 2.17). The \( AB \) \( AB \) constitute twin which correspond \( hcp \) stacking type. Therefore, stacking fault is \( fcc \) metals can be considered as submicroscopic twins of nearly atomic distance.

![Diagram of stacking sequence](image1)

**Figure 2.15:** Close-packed stacking sequence for \( fcc \) in a) 2D, b) 3D [35].

The difference in deformation behaviour of \( fcc \) metals are due to differences in stacking fault behaviour. A stacking fault in a \( fcc \) metal, when viewed from dislocation theory, is an extended dislocation consisting of a thin region bounded by partial dislocations (Fig 2.16b).

![Diagram of dislocation](image2)

**Figure 2.16:** a) Slip in a close-packed (111) plane in an \( fcc \) lattice [34], b) dissociation of a dislocation into two partial dislocations.
The nearly parallel partial dislocations (Fig. 2.16b) tend to repel each other, but this is counterbalanced by the surface tension of stacking fault pulling them together. The lower the stacking fault energy the greater the separation between partial dislocations and the wider the stacking fault.

![Diagram of stacking sequence with and without fault in the (111) plane of fcc structure.](image)

**Figure 2.17: Stacking sequence with and without fault in the (111) plane of fcc structure.**

Dissociation of unit dislocations is independent of the character (edge, screw, or mixed) of dislocation. However, unlike the un-extended screw dislocation, the extended screw dislocation defines a specific slip plane, the \{111\} plane of the fault and it will be constrained to move in this plane. The partial dislocations move as a unit maintaining the equilibrium width of the faulted region. Because of this restriction to a specific slip plane, an extended screw dislocation cannot cross slip unless the partial dislocations recombine into a perfect dislocation. Constrictions in the stacking fault ribbon which permit cross slip are possible, but this requires energy. The greater the width of stacking fault (or the lower the SFE) the more difficult it is to produce constrictions in the stacking faults. This explains why cross slip is quite prevalent in high SFE metals, (*i.e.* Aluminum which has a very narrow stacking fault ribbon), while it is not observed usually in low SFE metals (*i.e.* copper which has a wide stacking fault ribbon).

The plastic deformation mechanisms (slipping, twinning) are dependent on both SFE and critical resolved shear stresses required for slipping and twinning which, in turn, depends on the orientation relationship of favored slip and twin systems in a polycrystalline metal with the external loads (ratio of Schmid factor for slip versus that for twin) [36]. In fcc
family, aluminum and aluminum alloys have the highest SFE, while copper and copper alloys possess the lowest SFE-values.

2.5.3.2. SFE and strain hardening rate
The SFE has considerable influence on strain hardening as a consequence of its role on grain refinement, twin formation and defect accumulation. The significant enhancement of strain hardening observed in low SFE alloys is due to the inhibition of dynamic recovery/cross slip and deformation twinning.

The effects of twin boundaries are equivalent to conventional grain boundaries with respect to the Hall–Petch effect [33, 37], i.e. the same Hall–Petch slope for both twin boundaries and grain boundaries because twin boundaries are strong barriers for dislocation motion. Basinski [38] suggested that that, as a result of twinning shear transformation, some glissile dislocations are converted into sessile configurations and other dislocations become less glissile after twinning. Whatever the case, the consequence is identical: the twinning development leads to a work-hardening rate higher than that measured in the absence of twinning. This will conceivably have an effect on the flow formability (i.e. increase the grain–to–grain variability in the local equivalent plastic strain). This will be studied in chapters 3 and 4.

2.5.3.3. Twinning, Strength and ductility
Since deformation twinning acts as strong barriers to dislocation motion, it consequently affects the tensile ductility of a material [39-44]. Figure 2.18 shows the engineering stress–strain curves of three samples of Cu–30Zn, Cu–10Zn, and Cu in which $SFE_{Cu–30Zn} < SFE_{Cu–10Zn} < SFE_{Cu}$. With decreasing the stacking fault energy, the strength increases. However, the ductility first increases (Cu–10Zn sample) and then decreases, indicating an optimum stacking fault energy for the best ductility. In fact, the Cu–30Zn sample has the highest densities of twins and dislocations, which are already saturated before the tensile testing, making it impossible to further accumulate these crystalline defects during the tensile testing [33]. This leads to very high strain hardening rate and consequently low ductility.
Figure 2.18: The engineering stress–strain curves Cu, Cu–10Zn, and Cu–30Zn samples [33].

2.5.3.4. SFE and strain rate sensitivity (m)

Strain rate sensitivity is defined as $m = \frac{\partial \ln \sigma}{\partial \ln \dot{\varepsilon}}$ and describes how fast strain hardening occurs in response to plastic deformation. A positive value of $m$ implies that the material can resist necking. High values of $m$ and $n$ (strain hardening exponent) means the material can exhibit a better formability in stretching. For a material that obeys the power law of strain hardening, the $m$ value is numerically identical to the post necking strain and $n$ value is identical to uniform (pre-necking) strain. Therefore, a material with high $m$ value exhibits more deformation before final failure (fracture). Tensile strain rate jump tests on fine grained copper have shown that twinned structures significantly increase the strain rate sensitivity.

2.5.4. Twinability

In face-centered-cubic (fcc) materials, twinning and slip occur through dislocation processes operating on the same set of crystallographic slip system and are thus in direct competition [26]. The competition can be described by “twinability”, the ease with which a metal deforms by twinning in competing with dislocation mediated slip [45, 46]. The
commercial Al alloys can, to a first approximation, be ordered in terms of twinability as follows [47]:

\[1.xxx < 4.xxx < 2.xxx < 6.xxx < 7.xxx < 5.xxx\]

Indeed, alloying with Mg is expected to bring about significant increase in the tendency for formation of partial dislocations and for mechanical twinning.

Considering the SFE of SMFF’d alloys in the present research, the twinnability can be ordered as follows:

\[70/30\ brass > Copper > 5052\ aluminum > 6061\ aluminum\]

In this PhD research, metals that are known to deform by different slip/twin ratios are tested to be able to investigate this factor on the flow formability. Since previous studies (Roy et al. [13]) have already shown that flow forming involves large plastic strain gradients and largely different strain gradients, specialized micro/nano-indentation tests are performed to investigate the effect of strain rate and indentation depth on the slip/twin deformation mechanisms. These tests will allow a more detailed assessment of the operative deformation mechanisms at work piece during SMFF of these materials.
2.6. Pyramidal indentation testing

The aim of this research is to improve our understanding of the local plastic deformation during a single-roller SMFF operation and the responsible plastic deformation mechanisms in the fcc and bcc metals/alloys. This is accomplished through experimentally mapping the local equivalent plastic strain through the thickness of a SMFF’d part using micro/nano-indentation hardness measurements. Micro-indentation techniques have been used by others to infer the local equivalent plastic strain of highly deformed materials. Chaudhri [23, 48] employed Vickers microindentation techniques to map the local equivalent plastic strain in the deformed region around a large spherical indentation made in copper while Tseng et al. [24] also used Vickers micro-indentation hardness to map the equivalent strain through the roll-bite region in cold rolling of 1018 steel. Roy et al. [13] recently used this technique to map the local plastic strain during smooth mandrel flow forming. Therefore, it is necessary to review the indentation technique principals and indentation platform and the way it operates.

2.6.1. Berkovich indenter characteristics

The micro/nano-indentation tests carried out in this study were performed on a NanoTest indentation machine (Fig. 2.19) which is a nano/micro-mechanical property testing platform designed and manufactured by Micro Materials Ltd. Wrexham, United Kingdom. The machine consists of three separate modules that include: indentation, scanning and impact testing. All three modules are designed to work in conjunction with the low load head (0.1-500 mN) or the high load head (0.1-20 N). In all tests, diamond Berkovich indenters with a pyramidal shape were used (Fig. 2.20).

2.6.2. Indentation platform

As shown in Fig. 2.19, the NanoTest platform is made up of a pendulum that can rotate about a friction-less pivot. At the bottom end the pendulum is attached the indenter and at the top end is mounted a copper coil. This copper coil is attracted to a permanent magnet once an electric current is applied to the coil. This attraction of the coil causes the indenter to move toward the sample. The resulting displacement of the indenter is measured by a parallel plate aluminum capacitor that has one of the plates attached to the
indenter holder. Any change in the distance between the capacitor plates as a result of the movement of the indenter, translates into a measurable change in the capacitance.

The sample to be tested is attached to a stage whose motion can be manipulated in the X–Y–Z directions by means of three DC motor driven micrometers.

The Micro Materials Nanotest indenter works by measuring the indentation force and the indenter motion when it is in contact with the test material. The sequence begins by applying an increasing amount of force to the indenter to indent a polished sample to a predetermined force/depth at a defined loading rate. The indentation force, depth and time data are recorded by the NanoTest software.

To run an accurate indentation test, it is necessary to perform a number of test calibrations of which the two most important are the load and depth calibrations. Load calibrations are performed by hanging weights of known mass, to the pendulum while the machine records the voltage that must be applied to the actuator coil to balance the pendulum in a vertical position. Depth calibration is performed using a sample of known mechanical properties (fused silica). No force is applied to the indenter during this test; rather the DC motor-driven micrometer is used to move the sample into contact with the indenter. As the micrometer moves the sample, the signal amplitude of the capacitance indentation displacement is continually monitored to correlate the depth of motion to the capacitance of the gauge.
Figure 2.19: Schematic illustration of the hardware components of a NaoTest platform [49].

Figure 2.20: 2D and 3D schematic illustrations of a Berkovich indenter typical of the one used in this study [50].
2.6.3. Contact Area Changes

Hardness measurements rely heavily on accurate determination of projected indentation area, at both the macro- and the nanoscale. Even traditional harness tests have to account for material characteristics that might adversely affect the measured area.

At a particular depth of penetration the indentation contact area is dependent not only upon the indenter tip shape, but also the elastic properties of the indented material. Materials with limited elasticity accommodate the volume of the indenter by plastic flow of the material, eventually causing the indented material to piling-up around the indenter tip. Elastic materials accommodate the indenter by longer range elastic deformation. In such cases the material appears to sink-in around the tip. Sink-in is often associated with well annealed samples. Figure 2.21 is a schematic of these two situations shown in cross section and top view. These figures demonstrate that the extent of elastic deformation can dramatically change the contact area from that predicted by the total displacement of the probe. Bolshakov and Pharr [51] did extensive finite element analysis on the influence of material properties on the shape of the deformation zone during indentation testing. In systems that demonstrated pile-up they measured differences of up to 60% between the calculated contact area and the actual contact area. They found that only materials that did not work-harden (pre-work hardened materials for example) experienced pile-up. In order to quantify the amount of elastic recovery in a system they utilized the ratio of the final depth ($h_f$) over the maximum depth ($h_{max}$). For entirely elastic material the ratio would be equal to zero, and for a purely plastic response the ratio would equal one. All materials with a ratio below 0.7 experience sink-in, regardless of their ability to work-harden. Materials with an $h_f/h_{max}$ value above 0.7 experienced pile-up [52, 53]. The amount of pile-up corresponds to the size of the plastic zone, or the load applied.

A parameter describing the ratio of the indentation contact area to the triangular area was then determined for each set of indentations:

$$c = \frac{A_i}{A_c}$$

where $A_i$ refers to the actual indentation projected contact area, and $A_c$ to the ideal cross-sectional area of the indenter, the area of the triangle marked by the corners of the
indentation. For an indentation where pile-up has occurred, \( c < 1 \); for sink-in \( c > 1 \) [54]. If indenter geometry is self-similar at all depths, \( c \) should be independent of depth for a homogeneous material [54].

Figure 2.22 shows a plot of \( c \) as a function of depth for the strain-hardened and the annealed copper samples [54]. As is shown in Fig. 2.22, derived by McElhaney et al. [54], for the annealed material, \( c \) is considered to be 0.9 and for work hardened material \( c = 1.2 \).

Figure 2.21: Schematic representation of pile-up and sink-in. Top picture is a cross-section of the indenter at maximum load. The radius of the projected area of contact (a) based upon displacement is an overestimate in the case of sink-in and an over estimate for materials that pile-up. This is easily visualized by the overhead view, where the assumed contact area is indicated by dotted lines while the actual contact area is indicated by the solid lines [55].
Figure 2.22: The correction parameter, $c$, as a function of contact depth for the strain-hardened and annealed copper samples [54].

2.7. Interpretation of the plastic strain resulting from nano/micro-indentation

Figure 2.23 shows a schematic diagram of the expanding cavity model of the elastic plastic deformation induced by indentation. For an indent of penetration depth $h$ and projected width $a$, a hydrostatic core surrounds the indenter. The material within this core is constrained by the high hydrostatic pressure and, therefore, undergoes only elastic deformation.

Early observations of the plastically deformed zone around conventional pyramidal indentations indicated that its shape is approximately hemispherical [56, 57]. Marsh [57] therefore suggested that the deformation zone could be represented as half of that produced by a spherical cavity expanding under internal hydrostatic pressure. Johnson [58] took this description further by equating the expanding hydrostatic volume to the region of material under the indenter (Fig. 2.23). Although not a perfect description of indentation (for example, the lack of constraint can lead to differences near the surface, particularly if there is significant pile-up or sink-in), this picture of indentation is the
basis of several analyses, including those leading to equations commonly used for indentation creep analysis.

2.7.1. Representative stress
The stress under an indentation varies from high levels in the vicinity of the tip to vanishingly small values in remote regions. Moreover, the stress state is highly multiaxial in nature and its degree of tri-axially varies with position. However, a characteristic (or effective) indentation stress is commonly expressed. This characteristic stress $\sigma_{ind}$ is taken as the applied load $P$, divided by the indentation contact area $A_{ind}$.

For a Berkovich indenter (Fig. 2.20):

$$A_{ind} = 3\sqrt{3}h^2 \tan^2 \theta$$  \hspace{1cm} (2.5)

where $h$ is the depth of indent and $\theta = 65.3^\circ$ is half of the included angle of the indenter. Therefore, one can write the indentation stress $\sigma_{ind}$ as:

$$\sigma_{ind} = \frac{P}{cA_{ind}} = \frac{P}{24.5ch^2}$$  \hspace{1cm} (2.6)

where $c$ is the correction factor when considering pile-up and/or sink-in.

2.7.2. Representative strain
Many researchers focused their effort on finding a relation between standard hardness criteria (Brinell and Vickers) and effective strain. Tabor [59] first established an empirical relation between pyramidal Vickers hardness and the flow stress of the material based on measured values of hardness on specimens compressed by specific ratios, therefore with known flow stress. Tabor found that for pyramidal indentations, the average plastic strain resulting from the indentation process was 8-10%. More importantly, due to the fact that pyramidal indentations are geometrically self-similar, meaning that the ratio of their projected diagonal length to their depth remains constant, all pyramidal indents have an equivalent average plastic strain of 8-10%. Tabor also correlated the Vickers hardness number to yield stress. He proposed the following equation:
\[ H_v = c \sigma_y \]  

where \( H_v \) is the Vickers hardness, \( c \) is a constraint factor between 2.9 to 3.0 and \( \sigma_y \) is the uniaxial yield strength of the indented material.

![Diagram of deformation field](image)

**Figure 2.23:** Representation of the deformation field under an indent, as an expanding hemispherical volume subjected to hydrostatic pressure, as developed by Marsh [57] and Johnson [58]. As the hydrostatic volume increases in size, more material undergoes plastic deformation and the elastic–plastic boundary advances [60].

Following Tabor’s work, Chaudhri [23] investigated the strains surrounding spherical indentation in copper. In order to do so, a large spherical indent was made and then the strain surrounding the indentation site was mapped using much smaller Vickers micro-indentations. This was accomplished via developing a relationship for equivalent plastic strain as a function of indentation hardness. Chaudhri [23] deformed several samples of annealed copper to plastic strains of 0.1 to 0.97 and then performed repeated hardness tests on them. Chaudhri [48] later found that the Vickers hardness could be fit to a power law function of equivalent plastic strain. Since this technique was successfully employed to develop strain maps around a spherical indentation, the same process can be used to investigate the equivalent strain of materials subjected to other cold forming operations, such as flow forming.
Kim et al. [61] performed an upsetting experiment, and then measured the hardness at various locations in the part. They also calculated effective strain distribution in the upset part through a FE analysis of the upsetting process. By correlating the measured hardness and the numerically found strains, they found a relation between Vickers hardness and effective strain (Fig. 2.24). Gouveia et al. [62, 63] performed a similar study for cold forward extrusion. They obtained a relation between Vickers hardness and effective strain by measuring the hardness at the center of cylindrical specimens compressed at specific ratios. Petruska and Janicek [64] also conducted a similar study. They carried out compression and extrusion tests on steel and copper specimens. After cutting the specimens through their symmetry axes, they measured hardness at various points. By relating the measured values of Brinell or Vickers hardness to numerically obtained strains, they obtained empirical equations. Ruminski et al. [65] obtained an empirical relation between Vickers hardness and effective strain to determine the mechanical property distribution in cold drawn tubes.

Tseng et al. [24] did exactly this by investigating both strain and strain rate in the roll-bite region during cold rolling of 1017 steel. The same techniques were employed as by Chaudhri [23]. Tseng et al. [24] were able to establish a relationship between equivalent plastic strain and hardness by fitting their data to a 2nd order polynomial relationship.

Once this relationship was established (Fig. 2.25), Tseng et al. made a large number of indents through the roll-bite region of cold-rolled 1017 steel. Then, applying the above relationship, they were able to create a strain map of the region between the rolls. In the preceding works of Tabor [59], Chaudhri [23, 48] and Tseng et al. [24], it is clear that there is a relationship between indentation hardness and equivalent flow stress/strain.

Based on the hardness measurements made on a material that had been compressed by various amounts, Tabor [59] concluded that the initial uniaxial strain, $\varepsilon_0$, is additive to the representative strain, $\varepsilon_r$. $H_v$ may then be expressed that

$$H_v = A(\varepsilon_0 + \varepsilon_r)^B$$

2.8
With the proper micro-indentation hardness testing technique, the applicability of this equation to multi-axially deformed (i.e. SMFF) materials (i.e. 5052 and 6061 aluminum alloys, pure copper, and 70/30 brass) will be investigated in this thesis.

![Graph showing the relationship between hardness and effective strain in 1010 steel.](image)

**Figure 2.24:** Relationship between hardness obtained and effective strain in 1010 steel [61].

![Graph showing the relationship between true strain and Vickers hardness for 1017 steel.](image)

**Figure 2.25:** Relationship between true strain and Vickers hardness for 1017 steel [24].
2.7.3. Representative strain rate

Following the argument described in the previous section for the effective indentation stress, Atkins et al. [66] derived a relationship for the effective average indentation shear strain rate \( \dot{\gamma}_{\text{ind}} \), as a function of the distance \( r \) from the center of an indentation, the diameter of the hydrostatic volume of deformed material beneath the indentation \( d \), and the rate of change of this diameter with respect to time \( \dot{d} \):

\[
\dot{\gamma}_{\text{ind}} = \frac{3}{2} \frac{d^2}{r^3} \dot{d}
\]

This relationship was used by Pollock et al. [67], who selected the maximum strain rate (located at the boundary between the hydrostatic volume and the region deforming plastically) as the characteristic value of the indentation strain rate. These workers also noted that, for a conical indenter, the value of \( d \) and the indentation depth \( h \) are linearly related, leading to the following equation:

\[
\dot{\varepsilon} = k \frac{\dot{h}}{h}
\]

where \( k \) is constant.

2.8. Indentation strain rate and stress

There are four types of tests that have been employed using depth-sensing indentation systems to gain insight into the relationship between indentation strain rate and indentation stress [68]: indentation load relaxation (ILR) tests [69] constant load rate (CLR) tests [70, 71] constant-load indentation creep tests [72-74] and constant strain rate (CSR) tests [75, 76]. In the following sections CLR and CSR test mechanisms are discussed in details.

2.8.1. Constant load rate tests (CLR)

In a CLR test, the indenter is loaded at a constant loading rate until the indenter has reached a prescribed depth in the material. A complete series of experiments would involve this procedure utilizing a different loading rate for each indent made and
calculating the indentation stress for that loading rate from the applied load and the achieved depth.

Researchers [77-80] have used CLR indentation testing, at a wide range of loading rates and indentation depths, in order to measure the rate sensitivity of materials. Indentation is, indeed, a preferable method, since the tested volume of material is scale-able with respect to the microstructure, to measure the strain rate sensitivity of metals and alloys.

By taking the indentation stress, $\sigma_{\text{ind}}$, and indentation strain rate, $\dot{\varepsilon}_{\text{ind}}$, proportional to flow stress and flow strain rate, one can write:

$$\sigma_{\text{ind}} = D\dot{\varepsilon}_{\text{ind}}^m$$  \hspace{1cm}  (2.11)

taking the logarithm of both sides of Eq. 2.11 and simply yields

$$m = \frac{d\ln\sigma_{\text{ind}}}{d\ln\dot{\varepsilon}_{\text{ind}}}$$  \hspace{1cm}  (2.12)

The challenge encountered in previous indentation approaches was the scatter of the data of the nominal indentation hardness [81], which often leads to a low resolution of indentation strain rate sensitivity exponent (especially for materials with low indentation strain rate sensitivity exponents).

### 2.8.2. Constant strain rate (CSR) tests

In a CLR indentation test, the strain rate is not constant \textit{(i.e.} strain rate non-linearly diminishing with indentation load or depth). It is, therefore, hard to directly convert a constant loading rate to a representative strain rate that might be a more useful parameter than loading rate for analyzing the inhomogeneous deformation. However, most of the indentation experiments in the previous works were made under constant load rate condition possibly due to the instrumental limitations.

Lucas and Oliver [80], for the first time, developed the means of achieving constant average strain rate (CSR) indentation by controlling the indentation load rate, $\dot{P}$ such that the ratio of $\frac{\dot{P}}{P}$ was held constant and found a good agreement with uniaxial test data.

From the definition of indentation stress:
\[
\sigma_{\text{ind}} = \frac{P}{A_{\text{ind}}} = \frac{P}{Bh^2} \tag{2.13}
\]

therefore,

\[
\dot{P} = \frac{\partial P}{\partial t} = \frac{\partial P}{\partial h} \frac{\partial h}{\partial t} = \frac{\partial P}{\partial h} \dot{h} \tag{2.14}
\]

where,

\[
\frac{\partial P}{\partial h} = 2h\sigma_{\text{ind}} \tag{2.15}
\]

therefore,

\[
\dot{P} = 2h\dot{h}\sigma_{\text{ind}} \tag{2.16}
\]

by substituting Eq. 2.13 in Eq. 2.16, one can write:

\[
\dot{P} = \frac{2P\dot{h}}{h} \tag{2.17}
\]

and therefore,

\[
\frac{\dot{P}}{P} = 2\frac{\dot{h}}{h} = 2\dot{\varepsilon} \tag{2.18}
\]

then,

\[
\dot{\varepsilon} = \frac{1}{2} \frac{\dot{P}}{P} \tag{2.19}
\]

2.9. Indentation size effect

The existence of a material size effect (or length scale) for plasticity is now firmly supported by direct dislocation simulations [82, 83] and by four kinds of laboratory experiments: micro-torsion [84], micro-bending [85], particle-reinforced metal-matrix composites [86] and micro-indentation hardness tests [87-90]. These experiments have repeatedly shown that metallic materials display significant size effects when the volume of the deforming metal is in the order of a cubic micrometer. This is clearly evident during nano/micro-indentation tests when the measured indentation stress \( \sigma_{\text{ind}} \) is clearly
greater at small indentation depths \((h < 1.6 \mu m)\) than at deep indentation depths. The fact that the flow stress of a material increases when the volume of the deformed material decreases is of particular importance to consider when analysing mechanical forming operations such as SMFF where very strong plastic strain gradients are invoked in the work piece.

Size effects in the indentation hardness may arise from either the strong gradients of plastic strain that are naturally created in micrometer scale indentations or from dislocation starvation effects for nanometer scale indentations \([90]\) that may arise when intense plastic deformation is forced to occur across a very small volume of an initially defect–free crystalline material.

The process of making a pyramidal shaped indentation requires the lateral displacement of the indented material. This displacement results in the generation of a strain gradient around the indentation and is accomplished by the motion of line defects, dislocations, in the region beneath the indentation (Fig. 2.26). In the case of deep indentations (i.e. \(h > 1 \mu m\)) the required plastic deformation occurs over a relatively large area and hence it can be accommodated by conventional dislocation motion occurring by glide on easy-slip crystallographic systems. Such a population of dislocations is referred to as a “statistically stored” dislocation (SSD) density and their properties, i.e. their mobility and the way they interact with each other, are the same as dislocations that contribute to the plastic strain during the deformation of large–size samples of material.

If, on the other hand, the indentation is very shallow (i.e. \(h < 1 \mu m\)) the plastic deformation is forced to occur over a very small volume containing a limited number of easy slip systems. In this case dislocations are forced to initiate and glide upon other crystallographic systems. This required increased applied stress and hence the indentation stress is observed to increase when \(h < 1 \mu m\). Such a population of dislocations is referred to as a “geometrically necessary” dislocation (GND) density and, since they may need to be initiated and glide on non–easy slip systems, may have quite different mobility and interaction characteristics than SSDs.

Nix and Gao \([91]\), using the Taylor dislocation model, argued that the geometrically necessary dislocations, whose density is proportional to the inverse of the indentation
depth. Based on their model the following relation between the micro-indentation hardness \( H \) and the indentation depth \( h \) was proposed:

\[
\left( \frac{H}{H_0} \right)^2 = 1 + \frac{h^*}{h}
\]

where \( h^* \) is a characteristic length on the order of microns given by Nix and Gao [91] that depends on the properties of indented material and the indenter angle, and \( H_0 \) is the indentation hardness for a large indentation depth (i.e. \( h \gg h^* \)). The above relation predicts a linear relation between \( H^2 \) and \( 1/h \).

![Figure 2.26: Geometrically necessary dislocations underneath an indenter. (a) The material originally occupying the region of the plastic indent has been pushed into the substrate material as extra storage of defects. (b) A schematic view of the atomic steps on the indented surface and the associated geometrically necessary dislocations [92].](image)

The theoretical argument for the hypothesis that indentation tests performed, with a pyramidal indenter, under conditions of constant \( \frac{\dot{P}}{P} \) will be performed under conditions of essentially constant \( \dot{\varepsilon}_{\text{ind}} \) regardless of indentation depth \( h \) is presented in the following proof. By assuming that \( \dot{\varepsilon}_{\text{ind}} = \frac{\dot{h}}{h} \) and \( \sigma_{\text{ind}} = \frac{P}{Bh^2} \), where \( B \) is a geometrical constant. Therefore,

\[
P = \sigma_{\text{ind}} Bh^2
\]
invoking the Nix and Gao equation for the indentation depth dependence of $\sigma_{\text{ind}}$ [91]:

$$\sigma_{\text{ind}} = \sigma_0 \sqrt{1 + \frac{h^*}{h}}$$

2.22

substituting Eq. 2.21 into Eq. 2.22:

$$\sigma_{\text{ind}} = Dh^2 \sigma_0 \sqrt{1 + \frac{h^*}{h}}$$

2.23

the derivative of $P$ with respect to time $t$ can be expressed as:

$$\dot{P} = \frac{\partial P}{\partial t} = \frac{\partial P}{\partial h} \frac{\partial h}{\partial t}$$

2.24

where $\frac{\partial h}{\partial t} = \dot{h}$; therefore,

$$\dot{P} = \frac{\dot{h} \left( 2h \sqrt{1 + \frac{h^*}{h}} - \frac{h^*}{2\sqrt{1 + \frac{h^*}{h}}} \right)}{h^2 \sqrt{1 + \frac{h^*}{h}}} = 2 \frac{\dot{h}}{h} - \frac{\dot{h} h^*}{2h^2 \left( \frac{\sigma_0}{\sigma_{\text{ind}}} \right)^2}$$

2.25

$$\dot{P} = \frac{\dot{h}}{h} \left( 2 - \frac{1}{2} \frac{h^*}{h} \left( \frac{\sigma_0}{\sigma_{\text{ind}}} \right)^2 \right)$$

2.26

By expanding and simplifying Eq. 2.26 based on the Nix/Gao equation and considering $\sigma_{\text{ind}} \geq \sigma_0$ one can write:

$$\dot{P} = \frac{\dot{h}}{h} \left( 2 - \frac{1}{2} \left( \frac{\sigma_0}{\sigma_{\text{ind}}} \right)^2 \right) \approx 2 \frac{\dot{h}}{h}$$

2.27

or

$$\dot{\varepsilon}_{\text{ind}} \approx 0.5 \frac{\dot{\varepsilon}}{P}$$

This is in agreement with the predictions presented by Alkorta et al. [93].
2.10. Characterising thermally activated dislocation mechanisms

During plastic deformation in certain ranges of temperature and strain rate, different micromechanisms may play important roles and, therefore, one must consider the operative dislocation–based deformation mechanisms in order to explain the deformation behaviour. The motion of dislocations through fields of discrete sub-micrometer sized particles was studied since the 1970s in connection with the plastic deformation of metals and alloys.

Considering ambient temperature plastic flow the fcc alloys, the stress necessary for dislocation motion can be divided into two components; athermal (called also internal stress) and thermally activated (called also effective stress) [94-96]. The athermal stress is related to long range obstacles and the creation of new dislocations leading to strain hardening. The athermal stress is only weakly depending upon temperature through the effect of temperature on the elastic modulus of the material. Athermal stress is strain rate independent and depends on the microstructure (grain size, precipitates, and dislocation–dislocation interactions). The athermal component can be written as:

$$\tau_i = \alpha \mu b \sqrt{\rho_i}$$  \hspace{1cm} 2.28

where $\mu$ is the elastic shear modulus, $\alpha$ is a constant describing the interaction between dislocations taken by some to be in the order of 0.3 [97, 98], $b$ is the Burgers vector of dislocations, and $\rho_i$ is the total dislocation density. If assuming that all the dislocations are of the same configuration and mobility, one can express $\rho_i$ as the summation of the density of GNDs and SSDs as:

$$\rho_i = \rho_{GND} + \rho_{SSD}$$  \hspace{1cm} 2.29

Following the Nix and Gao equation (Eq. 2.22), the $H_0$, the independent hardness for a large indentation depth, where $\rho_{SSD} \gg \rho_{GND}$, can be expressed as:

$$H_0 = 3\sqrt{3} \alpha \mu b \sqrt{\rho_{SSD}}$$  \hspace{1cm} 2.30
where \( \mu \) is the shear modulus, \( b \) is Burgers vector, \( \alpha \) is a constant (0.3 [97, 98]). Based on Eq. 6, the density of SSDs is independent of indentation depth and is given as:

\[
\rho_{SSD} = \frac{H_0^2}{27(\alpha \mu b)^2}
\]

On the other hand, the density of geometrically necessary dislocations, \( \rho_{GND} \), is expressed as a function of the indentation depth, \( h \), and the angle \( \theta \) between the surface of the indenter and the plane of the surface (19.3° for Berkovich indenter) as [98-101]:

\[
\rho_{GND} = \frac{\int_0^a 2\pi (h_c/ba) r dr}{(2/3)\pi a^3} = \frac{3}{2bh_c} \tan^2 \theta
\]

where \( r \) is the radius of an indentation of depth \( h_c \), and \( a \) is the radius of the plastic deformation zone around the indenter.

The thermal effective stress component is strain rate and temperature dependent (it increases when the strain rate increases or the temperature decreases). It is related to the interaction of the mobile dislocations with small discrete obstacles within the microstructure. The dislocation can only continue to move if they are able to overcome these obstacles with the combined help of the applied stress and thermal vibration.

The general expression for the applied flow stress is therefore given as the sum of the athermal and thermal components of the stress required to move a dislocation through a field of micro-structural scale obstacles as:

\[
\tau = \tau_i + \tau^*(T, \dot{\gamma})
\]

where \( \tau_i \) is the athermal, and \( \tau^* \) is thermal component of the stress. The shear strain rate \( \dot{\gamma} \) is directly related to the mean velocity \( \bar{v} \) of the dislocations by the Orowan equation:

\[
\dot{\gamma} = \rho_m b \bar{v}
\]

where \( \rho_m \) is the mobile dislocations density. The dislocation velocity is controlled by obstacles (their strength and inter-obstacle spacing), the temperature, and the effective applied shear stress as [102-104]:

\[
\tau = \tau_i + \tau^*(T, \dot{\gamma})
\]
\[
\bar{v} = \beta bv \exp \left[ -\frac{\Delta G_{\text{thermal}}(\tau)}{kT} \right]
\]

where \( \beta \) is a dimensionless constant, \( \nu \) is the frequency of atomic vibration, and \( \Delta G_{\text{thermal}}(\tau) \) is the thermal activation energy required for a dislocation, subjected to an applied shear stress \( \tau \), to overcome the deformation-rate limiting obstacle.

The resulting shear strain rate, \( \dot{\gamma} \), for such an obstacle-limited dislocation glide deformation process can be written in terms of an Arrhenius-type rate equation as:

\[
\dot{\gamma} = \dot{\gamma}_0 \exp \left[ -\frac{\Delta G_{\text{thermal}}(\tau)}{kT} \right]
\]

where the pre-exponent term \( \dot{\gamma}_0 \) is related to the total dislocation density, which can be expressed by \( \rho_m = \alpha \left( \frac{\tau}{\mu b} \right)^2 \), as:

\[
\dot{\gamma}_0 = \dot{\gamma}_p \left( \frac{\tau}{\mu} \right)^2
\]

\[
\dot{\gamma}_{\text{ind}} = \dot{\gamma}_p \left( \frac{\tau_{\text{ind}}}{\mu} \right)^2 \exp \left[ -\frac{\Delta G_{\text{thermal}}(\tau)}{kT} \right] = \dot{\gamma}_p \left( \frac{\tau_{\text{ind}}}{\mu b} \right)^2 \exp \left[ -\frac{\Delta G_{\text{thermal}}(\tau)}{kT} \right]
\]

\[
\dot{\gamma}_{\text{ind}} = \dot{\gamma}_p \rho_m b^2 \exp \left[ -\frac{\Delta G_{\text{thermal}}(\tau)}{kT} \right]
\]

### 2.11. Conclusion

The primary objective of this research is to apply pyramidal indentation testing to assess the plastic strain profiles invoked in SMFF’d work pieces made of bcc (1020 steel) and fcc (5052 and 6061 aluminum alloys, pure copper, and 70/30 brass) metal alloys. Also, the theoretical background of the mechanisms of plastic flow at room temperature (slip and twinning) during micro/nanoindentation of SMFF’d metal alloys will be assessed in detail. Descriptions of the theories, mechanism, and techniques which were applied to achieve these objectives were presented in this chapter.
2.12. References


Chapter 3

Assessment of plastic strain distribution and work hardening rate during SMFF operations on bcc (1020 steel) and fcc (pure copper, 70/30 brass, 6061 and 5052 aluminum) alloys

This chapter consists of three published, peer reviewed journal papers from my PhD research. Sections 3.1, 3.2, and 3.3 consists of published articles on the splined mandrel flow forming of 1020 steel (Section 3.1), 5052 and 6061 aluminum alloys (Section 3.2), as well as pure copper and 70/30 brass (Section 3.3). The materials subjected to SMFF operations possess different crystal structures (i.e. bcc and fcc), work hardening behaviour and stacking fault energies. It is, therefore, expected that the deformation responses of the tested materials during SMFF operations to be different.

Since the SMFF principals in the “experimental procedure” in the sections 3.1–3.3 is almost identical, although the materials and hardness/strain equations are different, the details of flow forming operation in the experimental procedure of Sections 3.2 and 3.3 have been taken out.

3.1. Plastic strain distribution during splined-mandrel flow forming*

This section provides the local variation in the von-Mises equivalent plastic true strain within a 1020 steel work piece that was fabricated by single-roller flow forming over a splined-mandrel. The largest equivalent plastic true strain occurs near the work piece /mandrel interface directly in front of the nose of the internal ribs and reaches approximately 170% when the work piece thickness reduction is 51%. This represents the forming limit for 1020 steel when subjected to the flow forming parameters used in this study. High levels of grain elongation were also observed in the work piece at the work

* A version of this section was published in the Materials and Design.  
3.1.1. Introduction

Splined-mandrel flow forming is a manufacturing process that uses rollers to form a spinning metal disc over a cylindrical mandrel containing longitudinal splines (Fig. 3.1.1). This allows for an internally ribbed thin-walled cylindrical metal part to be created in a single forming process. It is for this reason that splined-mandrel flow forming is a very cost effective fabrication method for a wide variety of parts. Although the technique is now used commercially there remains questions about how the magnitude of the local plastic strain near the region of the ribs of these flow formed parts is affected by process parameters such as, for example, the work piece thickness reduction ratio. Understanding these dependencies is critical to optimizing the usefulness of the splined-mandrel flow forming process.

In a previous study Roy et al. [1] measured the distribution of the local von-Mises equivalent plastic true strain through the thickness of a low carbon steel work piece that was flow formed, with different thickness reduction ratios, over a smooth mandrel. They determined the equivalent plastic strain by performing micro-indentation hardness tests and relating the measured hardness to the equivalent true plastic strain through calibration equations developed from similar hardness tests performed on samples, of the same alloy, deformed to known levels of true plastic strain. Roy et al. [1] found that the von-Mises equivalent plastic true strain followed a highly non-linear dependence upon position through the thickness of the flow formed part. The maximum plastic strain occurred at the surface of the work piece over which the roller contacted while the minimum strain occurred near the mid-thickness of the work piece. This method of using micro-indentation hardness to determine equivalent plastic strain will be use in this study to assess the through thickness variation in plastic strain in various regions of an internally ribbed part made by a splined-mandrel flow forming process.

There exists to date only a limited amount of published work on the local stress and strain within a work piece that has been flow formed over a splined mandrel. Ma et al. [2] investigated the local stress and strain in an aluminum alloy work piece subjected to spin
forming over a conical mandrel containing a circumferential spline. They proposed a range of processing parameters that are suitable for fabricating internally ribbed conical parts from the 3003 aluminum alloy. Jiang et al. [3-5] have reported the results of experiments and finite element simulations of the local plastic strain in an aluminum alloy work piece formed by a ball spinning operation over a longitudinally splined mandrel. They reported significant grain elongation occurring in the work piece as it enters into, and fills, the mandrel splines. It was observed that the extent of grain elongation, and hence the degree of local plastic strain, varied with location within the work piece ribs. No data have yet been reported on the effect of process variables on the magnitude of the local plastic strain in various regions of a metal work piece made by splined-mandrel flow forming. Zhang et al. [6] studied folding defect and plastic strain distribution during ball spinning of inner grooved copper tubes using two-dimensional FE simulations. Li et al. [7] conducted the finite element simulation of the cross-section forming of axially inner grooved copper tube. They analyzed the stress–strain distribution, metal flow rule, and contact force based on the simulation and experimental results. Their results show that gaps in the groove walls are caused not only by the diametric clearance between the inner wall of the copper tube and the mandrel but also the bending deformation of the copper tube.

In this research the results of an investigation of the through-thickness variation in the von-Mises equivalent plastic true strain in a low carbon steel work piece that is flow formed over a splined-mandrel by a single-roller flow forming process are presented. The objective of the study is to quantify the equivalent plastic true strain in the region near the ribs of the work piece and to understand its dependence upon the process parameter of thickness reduction.

3.1.2. Experimental procedure

Flat discs, 210 mm diameter and 8.5 mm thickness, were cut from 1020 steel plate. This plain carbon steel alloy has a ferrite/pearlite microstructure with an average ferrite grain size of 7.0 ± 0.5 µm. The ferrite grain shape is equiaxed with respect to the three orthogonal planes of the plate (RD refers to the Rolling Direction). The pearlite constituent appears as dark regions at, and in between, the ferrite grain boundaries (Fig.
The discs were each subjected to a multi-pass single-roller flow forming operation over a cylindrical mandrel containing longitudinal splines of 9.4 mm length, 3.0 mm depth and 2.5 mm width (Fig. 3.1.1a). Each disc was subjected to three flow forming passes with the work piece and mandrel rotating together at 300 rpm. The work piece was cooled and lubricated with an aqueous-based lubricant during each pass. The first two passes were identical for each sample and caused the flat disc to become formed around the cylindrical shape of the mandrel (Fig. 3.1.1b). The final flow forming pass pressed the work piece against the mandrel and caused it to fill the mandrel splines and endure a net change in thickness. The amount of deformation imparted during the final pass was controlled to produce parts with average thickness reductions (TR %) of 19%, 25%, 39%, and 51% (Fig. 3.1.3). The average thickness reduction refers to the percentage difference in thickness of the work piece before and after the multi-pass flow forming operation (Equation 3.1.1).

\[
TR\% = \frac{t_f - t_i}{t_i} \times 100
\]

where \( t_i \) is the initial thickness of the disc and \( t_f \) is the final thickness of the flow formed work piece.

All other forming parameters were kept constant for all the tests. It was found that thickness reductions greater than 51% resulted in the work piece tearing on a circumferential plane directly ahead of the longitudinal ribs (Fig. 3.1.4).

The local plastic deformation in the flow formed parts was assessed by sectioning the work piece to reveal the Longitudinal-Radial (LR) mid-plane through one of the ribs and the Circumferential-Radial (CR) plane across two of the ribs (Fig. 3.1.5). Both sectioned planes were mechanically ground with successively finer grits of SiC paper and finally polished with 1 \( \mu \)m diamond paste.

Micro-indentation hardness tests were performed, with 7 \( \mu \)m deep Berkovich pyramidal indentations spaced 100 \( \mu \)m apart, along lines extending from the mandrel- and the roller-sides of the work piece on the polished LR and CR planes (Fig. 3.1.5).
Figure 3.1.1: (a) Geometry of the single-roller splined-mandrel flow forming process used in this investigation; (b) flow forming sequence showing the starting work piece (Initial), the ‘cupped’ work piece after forming passes I and II, and the fully formed work piece after forming pass III [this figure is an adaptation of Fig. 3 from Ref. [1].
Figure 3.1.2: The equiaxed ferrite/pearlite microstructure of the 1020 steel plate from which the discs used in this flow forming study were cut.
Figure 3.1.3: Flow formed parts made with different amount of thickness reductions (TR). The small internal ribs that are studied in this investigation can be seen in all the parts.

Figure 3.1.4: The location of tearing in the wall of a flow formed part that was made with a thickness reduction greater than 51%. The tearing occurs on the Circumferential-Radial (CR) plane directly ahead of the nose of the internal ribs.
Figure 3.1.5: Illustration of the internally ribbed flow formed part. The two planes of section; namely, the Longitudinal-Radial (LR) mid-plane of a rib and the Circumferential-Radial (CR) plane across two ribs are shown. Measurements of the indentation hardness were made in the shaded Regions I–IV and the extent of grain elongation was assessed in the Regions I–V.

The micro-hardness was observed to change with position in the flow formed samples due to differences in the amount of local plastic strain (i.e. strain hardening). A relationship between the Berkovich indentation hardness $H$ (GPa) and the von-Mises equivalent plastic true strain ($\varepsilon_p = \sqrt[3]{\frac{2}{3} \varepsilon_{ij} \varepsilon_{ij}}$) of the indented material was obtained by subjecting samples of the same 1020 steel to plane-strain cold-rolling up to thickness reductions of $TR = 90\%$ ($\varepsilon_p = -\frac{2}{\sqrt{3}} \ln(1 - TR) = 266\%$) and measuring the indentation hardness of the samples. Figure 3.1.6 shows the resulting plot of $H$ versus $\varepsilon_p$. The
following expression for $\varepsilon_p(H)$ was obtained, by non-linear least squares regression, from the data in Fig. 3.1.6:

$$\varepsilon_p(H) = \left( \frac{H}{276.7} \right)^{7.571} - 0.08$$  \hspace{1cm} 3.1.2

In this study Eq. (3.1.2) is used to calculate $\varepsilon_p$ from the measured hardness data.

After micro-indentation testing, the polished samples were etched in a 2% Nital solution to reveal the ferrite grain boundaries and the pearlite constituent of the microstructure. Optical and scanning electron microscopy was then used to assess the change in ferrite grain size and shape in different regions of the flow formed parts. The change in ferrite grain shape reflects the magnitude of the local plastic strain.

Figure 3.1.6: von-Mises equivalent plastic true strain $\varepsilon_p$ versus the Berkovich indentation hardness for 1020 steel deformed plastically to various thickness reduction levels by plane-strain rolling.
3.1.3. Results and discussion

3.1.3.1. Variation in the equivalent plastic true strain within the flow formed part

Figures 3.1.7 and 3.1.8 show $\varepsilon_p$ (Eq. 3.1.2) versus distance from the mandrel/work piece interface at the top of the rib, near the nose, and directly in front of the rib on the LR plane (Regions I and II in Fig. 3.1.5) in parts made with thickness reductions of 19%, 25%, 39%, and 51%. In both locations $\varepsilon_p$ is highest at the mandrel/work piece interface and decreases with increasing distance through the thickness of the part. $\varepsilon_p$ also increases, at any given location, with increasing thickness reduction.

Figure 3.1.9 shows a plot of $\varepsilon_p$ versus distance from the mandrel/work piece interface on the LR plane at a position along the rib well away from the nose (Region III in Fig. 3.1.5). While $\varepsilon_p$ in this region increases with increasing thickness reduction it does not show as large a strain gradient nor does it display as high magnitude as do the data nearer to the nose (Figs. 3.1.7 and 3.1.8).
Figure 3.1.7: von-Mises equivalent plastic true strain, $\varepsilon_p$, versus distance from the workpiece/mandrel interface for different thickness reductions levels (TR). The strain was calculated from the indentation hardness measured along a line on the LR plane extending a distance of 1.7 mm from the top of an internal rib (Region I, Fig. 3.1.5).
Previously reported investigation of $\varepsilon_p$ in a 1020 steel work piece that was flow formed over a smooth-mandrel indicate that $\varepsilon_p$ is highest at the work piece /roller interface [1]. The $\varepsilon_p$ at the region of the work piece /roller interface was also measured in this study (Fig. 3.1.10). While $\varepsilon_p$ increases near the work piece /roller interface it does not reach the same magnitude as what occurs at the work piece /mandrel interface directly in front of the internal rib (Fig. 3.1.8).
Figure 3.1.9: von-Mises equivalent plastic true strain, $\varepsilon_p$ versus distance from the work piece/mandrel surface at the top of an internal rib, well away from the nose, along a line on the LR plane extending a distance of about 2.5 mm (Region III, Fig. 3.1.5).

Figure 3.1.11 shows $\varepsilon_p$ versus position across the complete thickness of 1020 steel parts made by splined-mandrel and smooth-mandrel flow forming under similar thickness reduction levels (TR = 51%). The $\varepsilon_p$ imparted to the work piece during splined-mandrel flow forming is significantly higher than that imparted during smooth-mandrel flow forming.
Figure 3.1.10: von-Mises equivalent plastic true strain, $\varepsilon_p$ versus distance from the work piece/mandrel surface along a line extending a distance on the LR plane a distance of about 1.2 mm (Region IV, Fig. 3.1.5). Plots are shown for samples that were flow formed to work piece thickness reduction levels of: 19%, 25%, 39%, and 51%.

The maximum $\varepsilon_p$ during splined-mandrel flow forming occurs near the mandrel/work piece interface directly ahead of the nose of the internal ribs and reaches a value of about 160%. Both parts show increased $\varepsilon_p$ at the work piece / roller interface however $\varepsilon_p$ is also slightly greater in this region for the splined-mandrel flow formed part than for the smooth-mandrel part. The creation of internal ribs therefore results in considerably higher equivalent plastic true strain in the region of the work piece at the mandrel surface.
Figure 3.1.11: von-Mises equivalent plastic true strain, $\varepsilon_p$ versus distance across the thickness on the LR plane of two parts made by single-roller flow forming at about 51% thickness reduction. The closed circles represent data from the present study of an internally ribbed work piece made by splined-mandrel flow forming while the closed triangles represent previously reported data of a work piece that was made by smooth-mandrel flow forming (Roy et al. [1]).

The magnitude of the $\varepsilon_p$ through the thickness of a flow formed work piece (TR = 51%) on the CR plane in the region between two ribs (Region IV, Fig. 3.1.5) is shown in Fig. 3.1.12. In this region $\varepsilon_p$ is very high ($\approx 170\%$) near the work piece /mandrel interface and is of approximately the same magnitude as $\varepsilon_p$ directly ahead of the ribs (Fig. 3.1.7). Therefore, it is concluded that the maximum equivalent plastic true strain occurs at the region directly in front of the internal ribs of the splined-mandrel flow formed part investigated here.

It was observed that in the flow forming process performed in this study, thickness reductions greater than 51% resulted in tearing of the work piece on the CR plane directly in front of the ribs (Fig. 3.1.4). Our micro-indentation hardness studies indicate that the largest equivalent plastic true strain, $\varepsilon_p \approx 170\%$, also occurs on this plane near the work
piece /mandrel surface. It is suggested, therefore, that the maximum von-Mises equivalent plastic true strain that the 1020 steel can endure during this single roller splined-mandrel flow forming procedure is about 170%. It is recognized that this magnitude of strain does not necessarily represent an absolute forming limit for 1020 steel since it is likely to be a function of the depth and spacing of the splines on the particular mandrel used for this study.

![Diagram showing von-Mises equivalent plastic true strain vs. distance from the mandrel surface](image)

**Figure 3.1.12: von-Mises equivalent plastic true strain, $\varepsilon_p$, versus distance across the thickness on the CR plane at a location equi-distant between two internal ribs. The part was made by single-roller flow forming process involving a 51% thickness reduction.**

3.1.3.2. Analysis of the grain shape in flow formed parts

To assess the local deformation occurring during the process of metal filling of the splines, the changes in ferrite grain shape on etched LR and CR sections in the regions around the ribs of the flow formed parts (Regions I to V, Fig. 3.1.5) were assessed. Figures 3.1.13 and 3.1.14 show optical images of the etched microstructure in the rib region of a work piece that was flow formed with a thickness reduction of 51%.
Figure 3.1.13: The ferrite/pearlite microstructure in different regions on the LR plane of a flow formed part (51% thickness reduction); (a) directly ahead of the rib, (b) directly at the nose of the internal rib and (c) at the top of the ribs away from the nose region.
Figure 3.1.14: The ferrite/pearlite microstructure on the CR plane in the different regions of adjoining ribs of a flow formed part (51% thickness reduction). (a and c) indicate the microstructure at the leading edge while (b and d) indicate the microstructure at the trailing edge of a rib (with respect to the forming direction).

While the microstructure of the starting 1020 steel disc consists of equiaxed ferrite grains with small colonies of pearlite located at the grain boundaries (Fig. 3.1.2), the flow
formed work piece contains highly stretched ferrite grains with the degree of stretching varying with location in the work piece. The region on the LR plane around the front of the internal rib, Fig. 3.1.13b, shows considerable grain elongation. This corresponds to a region of the mandrel were a small radius curvature leads into the recessed spline. This results in very high local plastic strain and strain gradients. The thin wall region of the flow formed work piece directly ahead of the rib also shows highly elongated ferrite grains (Fig. 3.1.13a). This corresponds to the region displaying the highest $\varepsilon_p$ as determined from the hardness measurements (Figs. 3.1.8, 3.1.12) and the location of the plane of fracture when thickness reductions greater than 51% are attempted (Fig. 3.1.4).

Figure 3.1.13c indicates that the ferrite grains of the work piece material at the top of the ribs, away from the nose region, are quite equiaxed and are similar to the grain shape of the starting material. This suggests that the work piece material that ends up in the bottom of the longitudinal splines is not heavily plastically deformed. This is confirmed by the relatively low measured $\varepsilon_p$ at the work piece /mandrel interface in the rib well away from the nose (Fig. 3.1.9).

The etched microstructure of the CR plane across two adjoining ribs is shown in Fig. 3.1.14. The ferrite grains are highly stretched in both the leading and the trailing edges of the ribs. Jiang et al. [5] reported that during ball rolling of an aluminum alloy work piece against a deeply splined-mandrel a difference in the extent of grain elongation could be observed in the leading and trailing edges of the resulting internal ribs. While extensive grain elongation clearly occurs near the leading and the trailing edges of the flow formed ribs in the present study, differences in the extent of grain elongation between the leading and the trailing edges were not observed. This may be due to the differences in the splined mandrel geometry and the work piece material in this study compared to that in Ref. [5].

Electron microscopy was used to record the local grain shape around indentations in various regions of the flow formed parts to determine if the variation in $\varepsilon_p$, determined from the measured $H$, coincides with the observed change in local aspect ratio of the ferrite grains. Figure 3.1.15 shows scanning electron micrographs of indentations made in various regions on the LR midplane of a flow formed sample made from high strained
(mandrel side) and low strained (roller side) regions of flow formed parts made with highest (51%) and lowest (19%) thickness reductions. The images indicate that the extent of grain elongation (i.e. the aspect ratio) increases with increasing thickness reduction and is also increased in the regions near the work piece /mandrel interface where the $\varepsilon_p$ is larger.

### 3.1.4. Conclusions

This study presents an assessment of the local von-Mises equivalent plastic true strain within a 1020 steel work piece that was fabricated by multi-pass single-roller flow forming over a splined-mandrel. The effect of thickness reduction on the equivalent plastic strain was investigated by flow forming samples at thickness reductions of 19%, 25%, 39%, and 51%. Particular attention was given to assessing the local plastic strain in the regions of the internal ribs of the work piece.

The following conclusions can be drawn from this investigation:

1. The measured equivalent plastic true strain was highest on the work piece /mandrel interface in the thin wall region directly in front of the nose of the internal ribs. This is different than what occurs during a smooth-mandrel flow forming process where the maximum equivalent plastic strain occurs at the work piece /roller interface. This difference reflects the increased local plastic strain necessary for the work piece to fill the recessed splines of the mandrel.

2. The maximum equivalent plastic true strain in the region of the work piece near the nose of the internal ribs increases with increasing work piece thickness reduction and reaches $\varepsilon_p \approx 170\%$ when the thickness reduction is 51%. Since the work piece is observed to fail at this region when the thickness reduction exceeds 51% it is concluded that $\varepsilon_p \approx 170\%$ represents the forming limit for 1020 steel when subjected to the flow forming parameters used in this study. It is recognized however that this forming limit will be sensitive to the geometry (depth, length, and spacing) of the mandrel splines.

3. Elongation of the ferrite grains of the work piece was observed to occur in regions that endured the highest plastic strain and was highest at the work piece /mandrel
interface near the nose of the internal ribs and along the leading and trailing edges of the rib.

Figure 3.1.15: Scanning electron micrographs of micro-indentations made on the polished LR sections of flow formed work pieces made at thickness reductions of: (a) 19%, and (b) 51%. The images on the left side are from regions near the work piece/mandrel interface while the images on the right side are from regions near the work piece/roller interface.

3.1.5. Acknowledgements
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3.1.6. References


3.2. Investigation of strain-hardening rate on splined mandrel flow forming of 5052 and 6061 aluminum alloys

In the preceding section, the effect of the strain-hardening rate on the plastic strain distribution in parts made from aluminum alloys by the Splined Mandrel Flow Forming (SMFF) process was investigated. Parts were made from annealed 5052 and 6061 aluminum alloys by SMFF with different levels of average work piece thickness reduction, from 20 to 60%. The average yield stress for the 5052 and 6061 aluminum alloys increased by 69% and 58% after SMFF operation (60% thickness reduction). The larger increase in yield stress of the 5052 aluminum work piece is attributed to its higher strain-hardening coefficient resulting from it containing higher levels of solid-solution Mg compared to the 6061 aluminum alloy. While the magnitude of the average von-Mises equivalent plastic strain through the thickness of the SMFF work piece was essentially the same for both alloys, the local equivalent plastic strain near the surface of the work piece and the point-to-point scatter in the measured plastic strain was greater in the 5052 aluminum alloy. This is attributed to the effect of the increased solid-solution Mg content localizing plastic flow. These findings illustrate the role of solid solution strengthening additions, in this case Mg, on increasing the average mechanical strength but also increasing the extent of local plastic strain variability in aluminum alloy material subjected to intensive plastic forming operations such as SMFF.

3.2.1. Introduction

Aluminum alloys have long been used as lightweight alternatives to low carbon steel for many structural applications. The Mg rich wrought aluminum alloys, such as the 5052 and 6061 aluminum alloys in their fully annealed, O-temper condition, are particularly attractive for certain applications because of their combined high yield stress and high room-temperature formability limits. These two alloys display significantly different strain-hardening characteristics owing to their different amounts of Mg present in solid

solution. The 5052 aluminum alloy contains up to 2.80 weight percent Mg while the 6061 aluminum alloy contains up to only 1.00 weight percent Mg [8]. This difference, in addition to the fact that much of the Mg in the 6061 aluminum alloy is present as Mg-Si rich precipitates (i.e. Mg$_2$Si), results in the 5052 aluminum alloy having a considerably higher concentration of Mg in solid solution than the 6061 aluminum alloy. The large amount of solid solution Mg in the 5052 aluminum alloy has a large affect on its plastic flow behaviour and increases its strain-hardening rate [9, 10]. While both alloys have sufficient ductility, when in the O-temper condition, to be shaped by a variety of room-temperature forming processes, the increased strain-hardening rate of the 5052 aluminum alloy offers the possibility for creating parts, via plastic forming, that have considerably higher yield stress compared to similarly formed 6061 aluminum alloy parts.

In this chapter the response of the 5052 and 6061 aluminum alloys to a particularly high-strain spin-forming process referred to as Splined Mandrel Flow Forming (SMFF) is studied. The SMFF process involves using rollers to press a spinning work piece over a similarly spinning splined mandrel. The pressure induced by the forming rollers as they travel down the work piece cause it to conform to the outer surface, and flow into the recessed splines, of the mandrel. The final product is therefore a thin-wall cylindrical part containing internal ribs. It was reported that when a 1020 steel work piece is subjected to a SMFF process involving a 50% average work piece thickness reduction the local equivalent plastic true strain directly ahead of an internal rib in the work piece is up to 170% [11]. This substantial level of plastic strain offers the possibility of producing high strength, heavily strain-hardened, internally-ribbed parts from relatively soft material provided that the work piece material possesses a sufficiently high strain-hardening rate. Aluminum alloys such as the 5xxx series which possess high strain-hardening rates due to their high level of solid solution Mg are therefore good potential candidates for SMFF processes. While very high local plastic strain and strain gradients are a characteristic feature of the internal rib regions of all SMFF parts [11-14] the effect of the strain-hardening properties of the work piece upon these gradients has not been reported. Such an assessment is presented in this research for the 5052 and 6061 aluminum alloys.
3.2.2. Experimental procedure

The 5052 and 6061 aluminium alloys test material was supplied in the form of 8.5 mm thick plates. The chemical composition of the two alloys is given in Table 3.2.1. The chemically etched microstructure of the as-received 6061 aluminum test material is shown in Fig. 3.2.1. The material has relatively large grains that are slightly elongated in the rolling direction of the plate. The average grain size in the rolling direction is between 100 and 200 µm while the grain size normal to the rolling direction is between 50 and 100 µm.

![Chemically etched microstructure of the as-received 6061 aluminum plate material.](image)

Figure 3.2.1: Optical micrograph of the chemically etched (Keller’s reagent) microstructure of the as-received 6061 aluminum plate material.

Figure 3.2.2 indicates $H$ versus $\varepsilon_p$ for the 5052 and 6061 aluminum alloys. Equations of the following form were fitted to the data to express $H(\varepsilon_p)$:

$$H(\varepsilon_p) = A(\varepsilon_p + \varepsilon_{ind})^B$$  \hspace{1cm} 3.2.1

In this equation $A$ and $B$ are material constants, calculated to best fit the experimental data, and $\varepsilon_{ind} = 0.08$ is the additional average equivalent plastic strain associated with the
pyramidal indentation process [11, 15-17]. Rearranging Eq. 3.2.1 and substituting the fitted values of $A$ and $B$ gives the following expressions for the $\varepsilon_p$ as a function of $H$ (in units of GPa) for the 5052 and 6061 aluminum alloys.

$$
\varepsilon_p(H)_{5052O} = \left(\frac{H}{1.402}\right)^{7.1} - 0.08 \quad 3.2.2a
$$

$$
\varepsilon_p(H)_{6061O} = \left(\frac{H}{1.113}\right)^{4.4} - 0.08 \quad 3.2.2b
$$

Tensile samples were cut from the starting plate material and from both the axial and the circumferential directions of the SMFF samples (60% thickness reduction) of each alloy. The tensile tests were performed under constant cross-head speed and the engineering stress ($\sigma$) and strain ($\varepsilon$) were calculated from the recorded load and cross-head displacement data.

![Figure 3.2.2: Indentation hardness $H$ versus von-Mises equivalent plastic strain, $\varepsilon_p$, for both the 5052 and 6061 aluminum alloys.](image-url)
3.2.3. Results

3.2.3.1. Effect of SMFF on the $\sigma - \varepsilon$ response

Figure 3.2.3 shows the stress versus strain response of the 6061 and 5052 aluminum alloys in both the as-received condition and after SMFF (60% thickness reduction). Both alloys show similar initial yield stress in the as-received condition however the 5052 aluminum alloy displays significantly larger levels of uniform strain prior to neck formation than the 6061 aluminum alloy (Table 3.2.1).

5052 aluminum alloy in the as-received condition also displays considerable serration, Portevin-Le Chatelier (PLC) effect, in the stress-strain response. Al–Mg alloys typically show the PLC effect at room temperature for a wide range of strain rates [18-20]. The PLC effect denotes a plastic instability, which is related to the discontinuous dislocation glide from the interaction of the moving dislocations with fast diffusing solute atoms (i.e. Mg) [21, 22]. Mobile solute atoms diffuse to the stress field generated by the mobile dislocations and tend to cluster at dislocations. Clustering leads to an enhanced resistance to dislocation motion. When the dislocation breaks free of the solute cluster, the resistance to its subsequent glide is reduced until a solute cluster is re-established via diffusion. The successive trapping and breaking free of the gliding dislocations as a result of this solute diffusion mechanism results in the serrated flow curve displayed in the as-received 5052 aluminum alloy. The higher uniform strain and the serrations both reflect the fact that the 5052 aluminum alloy contains more Mg in solid solution than does the 6061 aluminum alloy [23, 24]. Since the tensile tests in this study were performed under constant cross-head speed, and not constant specimen strain-rate, conditions they do not allow an accurate determination to be made of the strain-hardening coefficient. Table 3.2.1 therefore lists strain hardening coefficients reported in the literature [25-27] for annealed 5xxx and 6xxx class of aluminium alloys.
Figure 3.2.3: Engineering stress versus strain plots for the as-received and the SMFF (circumferential and axial) material: a) the 6061 aluminum alloy and b) the 5052 aluminum alloy.
The 6061 and 5052 aluminum alloys both display significant hardening after SMFF (Fig. 3.2.3). The increase in the initial yield stress is 70 MPa for the 6061 aluminum alloy and 140 MPa for the 5052 aluminum alloy. Since the SMFF process was performed under the same conditions both materials experience essentially the same level of average plastic strain during the SMFF process. The increased yield stress of the SMFF 5052 aluminum alloy compared to the 6061 aluminum alloy is therefore related to its increased strain-hardening rate. Figure 3.2.3 and Table 3.2.1 indicates that the both alloys have considerably higher yield stress in the axial direction compared to the circumferential direction after SMFF. This reflects the deformation path characteristic of the particular SMFF process used in this study.

**Table 3.2.1: Listing of the chemical composition, uniaxial yield stress, ductility, and strain-hardening coefficient of the 5052 and 6061 aluminum alloys**

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Chemical composition (Wt%) [28]</th>
<th>Yield stress (MPa)</th>
<th>Strain hardening exponent (n)</th>
<th>Elongation (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5052 aluminum alloy</td>
<td>Mg 2.2–2.8, Mn 0.10, Si 0.25, Fe 0.40, Cr 0.15–0.35, Cu 0.10, Zn 0.10, Al Balance</td>
<td>≈80</td>
<td>0.31 [26]</td>
<td>15</td>
</tr>
<tr>
<td>6061 aluminum alloy</td>
<td>Mg 0.8–1.2, Mn Max. 0.15, Si 0.4–0.8, Fe Max. 0.7, Cr 0.04–0.35, Cu 0.15–0.40, Ti Max. 0.15, Zn Max. 0.25, Al Balance</td>
<td>≈80</td>
<td>0.19-0.22 [25, 27]</td>
<td>10</td>
</tr>
</tbody>
</table>

3.2.3.2. Local plastic strain resulting from SMFF

The variations in $\varepsilon_p$ in Zones I–III of the mid-plane of an internal rib in a 6061 aluminum alloy part made by SMFF (60% thickness reduction) are illustrated by the degree of grain elongation shown in Fig. 3.2.4. The grains in Zone III, the area directly ahead of the internal rib and the area of highest plastic strain in the SMFF sample, are approximately three times longer in the axial direction than the as-received grain size.
(Fig. 3.2.1) and are correspondingly contracted in the radial direction of the work piece. This suggests that the average $\varepsilon_p$ is in the order of 120 to 140% in this region of the sample. Figures 3.2.5 to 3.2.8 show $\varepsilon_p$, determined from the measured $H$ via Eqs. 3.2.2(a,b), plotted versus through-thickness position in Zones I to IV of SMFF work pieces made from the 5052 and 6061 aluminum alloys. The $\varepsilon_p$ varies considerably with position in the sample and with the degree of thickness reduction. In all zones, $\varepsilon_p$ is higher at the edges of the sample than in the interior. The maximum $\varepsilon_p$ occurs in Zone III. These trends are similar to what was previously reported for $\varepsilon_p$ profiles in SMFF work pieces made of 1020 steel [4]. Comparison of the profiles in Figs. 3.2.5 to 3.2.8 indicates that the average $\varepsilon_p$ across a given zone in the work piece is essentially independent of the work piece alloy. This is illustrated in Fig. 3.2.9 which shows a histogram of the average $\varepsilon_p$ for each of the four zones for SMFF (60% thickness reduction) samples. This observation is intuitively correct since the average $\varepsilon_p$ within the internal rib region of a SMFF part must be determined by the dimensions of the mandrel spline, into which the work piece metal flows and fills, and is not dependent upon the properties of the work piece material. Figures 3.2.5 to 3.2.8 indicate however that differences exist between the two alloys in the profile of $\varepsilon_p$ across any of the zones and also in the scatter of the $\varepsilon_p$ data. These two factors are a function of the mechanical properties of the work piece material and are discussed below.
Figure 3.2.4: Optical images of the etched microstructure of Zones I-III of the 6061 aluminum alloy after SMFF (60% thickness reduction). The images indicate that the maximum grain elongation, and hence the maximum plastic strain, occurs in Zone III directly in front of the internal rib of the work piece.
Figure 3.2.5: von-Mises equivalent plastic true strain, $\varepsilon_p$, versus position $x$ through the thickness of Zone I of samples of a) the 5052 aluminum alloy and b) the 6061 aluminum alloy made by SMFF under various thickness reductions. The work piece/mandrel surface is at $x=0$. The trends end at decreasing $x$ values because the final thickness of the work piece is reduced when the thickness reduction increases.
Figure 3.2.6: von-Mises equivalent plastic true strain, $\varepsilon_p$ versus position $x$ through the thickness of Zone II of samples of a) the 5052 aluminum alloy and b) the 6061 aluminium alloy made by SMFF under various thickness reductions. The work piece/mandrel surface is at $x=0$. The trends end at decreasing $x$ values because the final thickness of the work piece is reduced when the thickness reduction increases.
Figure 3.2.7: von-Mises equivalent plastic true strain, $\varepsilon_p$ versus position $x$ through the thickness of Zone III of samples of a) the 5052 aluminum alloy and b) the 6061 aluminium alloy made by SMFF under various thickness reductions. The work piece /mandrel surface is at $x = 0$. The trends end at decreasing $x$ values because the final thickness of the work piece is reduced when the thickness reduction increases.
Figure 3.2.8: von-Mises equivalent plastic true strain, $\varepsilon_p$ versus position $x$ through the thickness of Zone IV of samples of a) the 5052 aluminum alloy and b) the 6061 aluminium alloy made by SMFF under various thickness reductions. The work piece/mandrel surface is at $x = 0$. The trends end at decreasing $x$ values because the final thickness of the work piece is reduced when the thickness reduction increases.
3.2.4. Discussion

The data presented here indicate, first, that an internally-ribbed SMFF part made with the 5052 aluminum alloy has a significantly increased yield stress compared to one made, under identical conditions, from the 6061 aluminum alloy. Since the initial yield stress of both materials is essentially the same, the increased yield stress of the SMFF part is attributed to the increased strain-hardening rate of the 5052 aluminum alloy (Table 3.2.1). While it is demonstrated that the average equivalent plastic strain across the thickness of the SMFF part is independent of the work piece alloy, the magnitudes of the maximum local $\varepsilon_{p}$, the maximum local $d\varepsilon_{p}(x)/dx$, and the point-to-point scatter in $\varepsilon_{p}$ displayed in Figs. 3.2.5 to 3.2.8 may be dependent upon strain-hardening characteristics of the work piece.

3.2.4.1. Local plastic strain and plastic strain gradients

Second order polynomial functions $\varepsilon_{p}(x)$, where $X$ is the distance, from the mandrel surface, were obtained by least squares regression curve fitting to the $\varepsilon_{p}$ versus $x$ data in
Figs. 3.2.5 to 3.2.8. The maximum $\varepsilon_p$ occurs, in all the SMFF samples, at the work piece/mandrel surface ($x = 0$) in the Zone III region directly in front of the internal rib. Figure 3.2.10 shows $\varepsilon_{p_{\text{max}}}$ versus percentage thickness reduction. The maximum equivalent plastic strain gradient $d\varepsilon_p(x)/dx$ also occurs at $x = 0$ of Zone III in all the SMFF samples tested. Figure 3.2.11 shows $d\varepsilon_p/dx\big|_{\text{max}}$ versus percentage thickness reduction. The 5052 aluminum alloy displays higher $\varepsilon_{p_{\text{max}}}$ and $d\varepsilon_p/dx\big|_{\text{max}}$ than the 6061 aluminum alloy when subjected to SMFF under identical conditions. These figures suggest that, for a given SMFF condition, both the maximum local equivalent plastic strain and strain gradient increase with increasing strain hardening coefficient of the work piece material.

3.2.4.2. Grain-to-grain variations in the local plastic strain

Figures 3.2.5 to 3.2.8 also indicate that the point-to-point variability in $\varepsilon_p$ increases with increasing thickness reduction during SMFF. The average point-to-point variation in $\varepsilon_p$ can be quantified by the average variance of $\varepsilon_p$ expressed as:

$$\text{Var}(\varepsilon_p) = \frac{\sum_{i=1}^{n} (\varepsilon_p - \varepsilon_p(x_i))^2}{n}$$

where $n$ is the number of positions, within a specific zone of the sample, where $\varepsilon_p$ was measured and $\varepsilon_p(x_i)$ is the second order polynomial function obtained by fitting to the $\varepsilon_p$ versus $x$ data. Figure 3.2.12 shows $\text{Var}(\varepsilon_p)$ versus percentage thickness reduction for the 5052 and 6061 aluminum alloys. While $\text{Var}(\varepsilon_p)$ clearly increases with increasing thickness reduction for both alloys, the average variance, and hence the average point-to-point scatter in $\varepsilon_p$, is larger for the 5052 aluminum alloy.
Figure 3.2.10: Maximum von-Mises equivalent plastic true strain, $\varepsilon_P$, in Zone III versus thickness reduction for the 5052 and 6061 aluminum alloys.

Figure 3.2.11: Maximum von-Mises equivalent plastic true strain gradient, $d\varepsilon_P/dx \big|_{\text{max}}$, in Zone III versus thickness reduction for the 5052 and 6061 aluminum alloys.
Figure 3.2.12: Average variance of the von-Mises equivalent plastic true strain, $\epsilon_p$ in Zone III versus thickness reduction for the 5052 and 6061 aluminum alloys.

Figure 3.2.13 shows a high magnification optical image of the first sixteen indentations at the work piece/mandrel surface in Zone I of a SMFF (60% thickness reduction) sample of the 6061 aluminum alloy. The size of the indentations is less than the grain size. The plastic deformation that occurs during indentation therefore occurs locally within only one or two grains. The point-to-point scatter in $\epsilon_p$ therefore reflects grain-to-grain variability in the plastic flow stress. Figure 3.2.13 indicates that grain-to-grain variability in the flow stress, under any given SMFF condition, is greater in the 5052 aluminum alloy than the 6061 aluminum alloy. Since the primary difference between these two alloys is the amount of Mg in solid solution, it can be concluded that the high concentration of solid solution Mg in the 5052 aluminum alloy contributes not only to an increased overall strain hardening coefficient, and this produces SMFF parts of higher yield stress, but also contributes to increased grain-to-grain variations in the flow stress within the SMFF work piece.
3.2.5. Conclusion
The increase in the yield stress and the local plastic strain due to Splined Mandrel Flow Forming (SMFF) to thickness reduction levels from 20% to 60% was studied for two wrought aluminum alloys; 5052 and 6061 aluminum alloys. While both alloys had essentially the same initial yield stress the yield stress of the 5052 aluminum alloy after SMFF (60% thickness reduction) was 47% higher than the similarly flow formed 6061 aluminum alloy. This is attributed to the higher strain-hardening coefficient of the 5052 aluminum alloy.

While the average equivalent plastic strain across the thickness of the SMFF samples was essentially independent of the work piece alloy, the maximum local equivalent plastic strain, the maximum plastic strain gradient, and the point-to-point variance in the local plastic strain were all found to be higher in the 5052 aluminum alloy than in the 6061 aluminum alloy. Since the depth of the micro-indentations used to determine the local equivalent plastic strain were smaller than the grain size, the differences are attributed to be due to higher grain-to-grain variability in the plastic strain on the SMFF 5052 aluminum alloy.
Figure 3.2.13: High magnification optical image of the etched microstructure near the work piece/mandrel edge in Zone II of an indented 6061 aluminum SMFF sample. The indentations are of a size less than the grain size indicating that variations in the measured $\varepsilon_p$ may be due to grain-to-grain variation in the plastic flow of the alloy.
3.2.6. References


3.3. Effect of strain-hardening rate on the grain-to-grain variability of local plastic strain in spin-formed fcc metals*

In the Section 3 of Chapter 3, the effect of strain-hardening rate on the grain-to-grain variability of local plastic strain in spin-formed fcc are investigated. To study the effect of work hardening rate and stacking fault energy (SFE) two fcc materials, pure copper and 70/30 brass, with completely different SFEs than 5052 and 6061 aluminum alloys were subjected to the SMFF operations. Micro-indentation hardness was used to assess the local equivalent plastic strain distribution in four fcc metals subjected to identical splined mandrel flow forming processes. Both the maximum local equivalent plastic strain and the grain-to-grain variability in the equivalent plastic strain increased with increasing strain-hardening rate of the work piece material. The deformed microstructure of the formed work pieces indicated that considerably more grain-to-grain variability in the dislocation slip step and deformation twin densities exist in the material with a high strain-hardening rate (i.e. 70/30 brass). These findings are of considerable importance and should be considered when assessing the suitability of high strain forming processes for producing reliable, and homogeneous, parts from fcc metal alloys that display high strain hardening rates.

3.3.1. Introduction

Spin-forming a metal work piece over a splined mandrel is an effective method for fabricating a variety of internally-ribbed cylindrical parts [2, 3, 6, 7, 11, 29-32]. This technique, referred to as Splined Mandrel Flow Forming (SMFF), invokes very high local plastic strain in regions where the work piece is forced to flow around, and over, the protruding mandrel splines. In past studies micro-indentation hardness testing have been used to measure the variation in the local equivalent plastic strain $\varepsilon_p$ in SMFF parts made from steel and aluminum alloys [11, 29, 30]. This technique allows one to assess

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the local variation in $\varepsilon_p$ with a spatial resolution similar to the grain size of the workpiece material. While the average through-thickness $\varepsilon_p$ is primarily a function of the mandrel shape and the degree of thickness reduction invoked upon the workpiece by the particular SMFF process, the magnitude of the local $\varepsilon_p$ and the degree of grain-to-grain scatter in $\varepsilon_p$ is highly dependent upon the microstructure of the workpiece. Our past study of the local $\varepsilon_p$ in identically formed SMFF workpieces made from the 5052 and 6061 aluminum alloys indicated that the grain-to-grain variability in $\varepsilon_p$ was greater for the 5052 aluminum alloy. It is suggested that this was due to the increased strain-hardening rate of the 5052 aluminum alloy however extensive data on the dependence of the grain-to-grain variability of $\varepsilon_p$ for a range of fcc metals, displaying a wide range of strain-hardening coefficients, has yet to be done.

In this study the local $\varepsilon_p$ profiles are measured in SMFF workpieces made from 70/30 brass and pure copper and then compare these profiles with those previously reported for the 5052 and 6061 aluminum alloys to assess the effect of workpiece strain-hardening rate on the magnitude and the variability of the local $\varepsilon_p$.

3.3.2. Experimental procedure
Splined mandrel flow forming tests were performed on multiple flat discs, each 210 mm diameter and 8.5 mm thickness, cut from plates of commercially pure copper and 70/30 brass that was annealed for 3–4 hours at 500°C.

3.3.2.1. Correlation of indentation hardness with equivalent plastic strain
Figure 3.3.1 indicates $H$ versus $\varepsilon_p$ for the 70/30 brass and the pure copper. The following equations for the $\varepsilon_p$ as a function of $H$ (in units of GPa) were obtained by fitting to the data in Figure 3.3.1.

$$\bar{\varepsilon}_p(H)_{\text{Copper}} = \left(\frac{H}{1.638}\right)^{6.3} - 0.08$$
$$\bar{\varepsilon}_p (H)_{70/30\ Brass} = \left( \frac{H}{2.771} \right)^{7.1} - 0.08$$

3.3.1b

Figure 3.3.1: Indentation hardness $H$ versus von-Mises equivalent plastic strain, $\varepsilon_p$ for both the pure copper and the 70/30 brass (solid-fill data points) and the 5052 and 6061 aluminum alloys.

3.3.2.2. Assessing the strain-hardening rate

In this study, correlating the local $\varepsilon_p$ invoked by SMFF with the strain hardening rate of the work piece material is of particular interest. Uniaxial tensile coupons were therefore cut from the starting copper and 70/30 brass material. Similar tensile coupons were also cut from the as-received 5052 and 6061 aluminum alloys that were used in the previously reported SMFF study [30]. The tensile tests were performed on a servo-hydraulic test
frame at a constant strain rate of $10^{-3}$ sec$^{-1}$. Specimen elongation was measured with a clip-on extensometer.

3.3.3. Results

3.3.3.1. Grain shape in the as received and the flow formed work piece

After micro-indentation testing, the sectioned copper and 70/30 brass samples were chemically etched to reveal the deformed grain shape, dislocation slip steps, and twins. The grain size, and shape, of the copper and 70/30 brass material before- and after-SMFF are shown in Figures 3.3.2–3.3.4. The average grain size of the as received, annealed, pure copper was about 25 $\mu$m while that of the 70/30 brass was about 75 $\mu$m (Table 3.3.1). The size and areal density of annealing/mechanical twins are considerably larger for the 70/30 brass than for the copper (Figure 3.3.5). The grain shape of both materials after SMFF is highly elongated indicating the extensive plastic deformation invoked by the forming process.
Figure 3.3.2: Optical micrographs of the chemically etched microstructure of the annealed a) pure copper, and b) the 70/30 brass. The size of the grains and the annealing twins are clearly larger in the 70/30 brass.
Figure 3.3.3: SMFF (60% thickness reduction) copper microstructure in different regions of the mid-plane plane of an internal rib (a) at the top of the rib away from the nose region, (b) directly at the nose of the internal rib and (c) directly ahead of the rib.
Figure 3.3.4: SMFF (60% thickness reduction) 70/30 brass microstructure in different regions of the mid-plane plane of an internal rib (a) at the top of the rib away from the nose region, (b) directly at the nose of the internal rib and (c) directly ahead of the rib.
Table 3.3.1: Chemical composition, stacking fault energy, and grain size of the four fcc metals studied in this investigation

<table>
<thead>
<tr>
<th>Test material</th>
<th>Composition</th>
<th>Stacking fault energy (mJ/m²)</th>
<th>Average grain size (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>70/30 brass</td>
<td>70% Cu, 30% Zn</td>
<td>7 [33, 34]</td>
<td>75 [Present work]</td>
</tr>
<tr>
<td>pure copper</td>
<td>99.9% Cu</td>
<td>78 [35-37]</td>
<td>25 [Present work]</td>
</tr>
<tr>
<td>5052 aluminum alloy</td>
<td>2.2% Mg, 0.40% Fe, 0.25% Si, 0.17% Cr, 0.1% Mn, 0.1% Cu, 0.1% Zn, Bal. Al [28]</td>
<td>approx. 145 [39]</td>
<td>52 [38]</td>
</tr>
<tr>
<td>6061 aluminum alloy</td>
<td>1.0% Mg, 0.6% Si, 0.4% Fe, 0.4% Cu, 0.3% Cr, 0.3% Zn, 0.1% Mn, 0.1% Ti, Bal. Al [28]</td>
<td>150 [40]</td>
<td>100 [Present work]</td>
</tr>
</tbody>
</table>

a: before indentation
b: after indentation

a: before indentation
b: after indentation

Figure 3.3.5: Optical micrographs of the deformed microstructure in Zone III at the mid-thickness of SMFF parts (60% thickness reduction) before and after indentation of a) 70/30 brass, showing both deformation twins and dislocation slip steps with considerable grain-to-grain variability, and b) pure copper, showing no deformation twins and extensive dislocation slip with much less grain-to-grain variability.

3.3.3.2. Strain-hardening rate of the four fcc metals
Figure 3.3.6 shows the a logarithmic plot of the uniaxial tensile true stress versus true strain of the as-received annealed pure copper, 70/30 brass, 5052, and 6061 aluminum alloys. The strain-hardening coefficients $n$ calculated as the slope of the data trends in Figure 3.3.6, was $n = 0.48$, 0.36, 0.29, and 0.22 for the 70/30 brass, pure copper, 5052, and 6061 aluminum alloys respectively. The significantly higher strain hardening coefficient displayed by the 70/30 brass and the pure copper is due to the inhibition of dynamic recovery/cross slip and the increased deformation twinning that occurs during plastic deformation of these low stacking fault energy metals.
3.3.3.3. Local plastic strain resulting from SMFF of the different fcc metals

Figures 3.3.7 to 3.3.10 show $\varepsilon_p$, determined from the measured $H$ and Eqs. 3.3.1(a, b), plotted versus through-thickness position $x$ in Zones I to IV of the pure copper and the 70/30 brass SMFF work pieces formed with 60% thickness reduction. The local $\varepsilon_p$ varies considerably with position in the work piece and, in all zones, $\varepsilon_p$ is higher at the edges of the sample than in the interior. Second-order polynomial functions of $\bar{\varepsilon}_p(x)$, where $X$ is the distance from the mandrel surface, were obtained by least squares regression curve fitting to the $\varepsilon_p$ versus $x$ data in Figs. 3.3.7 to 3.3.10. The maximum $\varepsilon_p$ occurs, in all the SMFF samples, at the work piece/mandrel surface ($x = 0$) in the Zone III region directly in front of the internal rib. This is similar to what was previously reported for $\varepsilon_p$ profiles in SMFF work pieces made of 1020 steel, 5052, and 6061 aluminum alloys [11, 29, 30]. Comparison of the $\varepsilon_p$ profiles through the thickness of Zone III (60% thickness reduction) between the four fcc alloys (70/30 brass, copper, 5052, and
6061 aluminum alloys) indicates that the magnitude of the maximum local \( \varepsilon_p \) at the mandrel/work piece interface, and the point-to-point scatter in the \( \varepsilon_p \) data, are greatest for the 70/30 brass.

### 3.3.4. Discussion

The objective of this research is to determine the dependence of the maximum local \( \varepsilon_p \), and the grain-to-grain variability in \( \varepsilon_p \), upon the strain-hardening rate of a range of fcc metal work pieces deformed by an identical SMFF process.

#### 3.3.4.1. Effect of work-hardening rate on the maximum local \( \varepsilon_p \)

Figure 3.3.11 shows a plot of maximum local \( \varepsilon_p \) in Zone III versus percentage thickness reduction of the SMFF work pieces. At all thickness reductions the maximum local \( \varepsilon_p \) occurs in the work piece near the surface where it contacts the mandrel during the forming process and the magnitude is greatest for the 70/30 brass, followed by the pure copper, 5052, and 6061 aluminum alloys. This order of material corresponds to decreasing work-hardening rate. This is clearly illustrated in Figure 3.3.12 where the maximum local \( \varepsilon_p \), in Zone III, is plotted against the strain-hardening coefficient, \( n \).
Figure 3.3.7: von-Mises equivalent plastic true strain, $\varepsilon_p$, versus position $x$ through the thickness of Zone I of samples of (a) pure copper and (b) 70/30 brass made by SMFF under various thickness reductions. The work piece mandrel surface is at $x=0$. The trends end at decreasing $x$ values because the final thickness of the work piece is reduced when the thickness reduction increases.
Figure 3.3.8: von-Mises equivalent plastic true strain, $\varepsilon_p$ versus position $x$ through the thickness of Zone II of samples of (a) pure copper and (b) 70/30 brass made by SMFF under various thickness reductions.
Figure 3.3.9: von-Mises equivalent plastic true strain, $\varepsilon_P$ versus position $x$ through the thickness of Zone III of samples of (a) pure copper and (b) 70/30 brass made by SMFF under various thickness reductions.
Figure 3.3.10: von-Mises equivalent plastic true strain, $\varepsilon_p$ versus position $x$ through the thickness of Zone IV of samples of (a) pure copper and (b) 70/30 brass made by SMFF under various thickness reductions.
Figure 3.3.11: Maximum von-Mises equivalent plastic true strain, $\varepsilon_P$ in Zone III versus thickness reduction for the 70/30 brass, pure copper, 5052 and 6061 aluminium alloys.

Figure 3.3.12: Maximum local plastic strain in Zone III, plotted versus the strain-hardening coefficient, $n$ for work pieces of the four fcc metals deformed by SMFF to different levels of thickness reduction.
3.3.4.2. Effect of work-hardening rate on the grain-to-grain variation in equivalent plastic strain

Figures 3.3.7 to 3.3.10 indicate that the point-to-point variability in $\varepsilon_p$ is a function not only of the thickness reduction during SMFF but also on the work piece material. For example, Fig. 3.3.5 indicates that, for SMFF under the same conditions, the 70/30 brass material displays greater point-to-point variability in $\varepsilon_p$ than the other three alloys. The average point-to-point variation in $\varepsilon_p$ can be expressed in terms of the average variance of $\varepsilon_p$ expressed by:

$$
Var(\varepsilon_p) = \frac{1}{k} k \sum_{i=1}^{k} \left( \varepsilon_p - \bar{\varepsilon}_p(x_i) \right)^2
$$

where $k$ is the number of positions, within a specific zone of the sample, where $\varepsilon_p$ was measured and $\bar{\varepsilon}_p(x_i)$ is the predicted value of local equivalent plastic strain from the second-order polynomials that were fitted to the data profiles in Figs. 3.3.7 to 3.3.10.

Figure 3.3.13 shows $Var(\varepsilon_p)$ versus percentage thickness reduction for the Zone III region of the 70/30 brass, copper, 5052, and 6061 aluminum alloys. While $Var(\varepsilon_p)$ increases with increasing thickness reduction for all the materials, the largest average variance, and hence the largest point-to-point scatter in $\varepsilon_p$, occurs in the 70/30 brass.

Figure 3.3.14 shows $Var(\varepsilon_p)$, obtained across Zone III (60% thickness reduction), plotted against the strain hardening coefficient $n$. The point-to-point variability in $\varepsilon_p$ clearly increases with increasing $n$. 
3.3.4.3 Understanding the effect of strain-hardening rate on the local equivalent plastic strain

The data in Figures 3.3.13 and 3.3.14 indicate that both the magnitude and the point-to-point variability in $\bar{\varepsilon}_p$ are increased when the SMFF work piece material displays a high work-hardening rate. In this study we have concentrated solely upon close-packed fcc metals. The strain-hardening rate of these materials is inversely related to the ease at which dislocations can cross-slip which, in turn, is related to the Stacking Fault Energy (SFE) of the material. Materials with low SFE tend to have extended dislocation and have difficulty cross-slip which, hence, display high work-hardening rate and large stacking faults which appear as twins in the microstructure. The 70/30 brass alloy which consistently displays the largest $\bar{\varepsilon}_p$, $\text{Var}(\bar{\varepsilon}_p)$, and $n$, is a low SFE material.
Close examination of the deformed microstructure in Zone III (60% thickness reduction) of the 70/30 brass and the 6061 aluminum alloy, the materials with the highest and the lowest strain-hardening rates, indicate that considerably more grain-to-grain variability in the dislocation slip step and twin densities exist in the 70/30 brass compared to the 6061-O aluminum alloy.

![Graph showing the relationship between \(\text{Var}(\varepsilon_p)\) and the strain hardening coefficient \(n\) for different materials.](image)

**Figure 3.3.14:** \(\text{Var}(\varepsilon_p)\), obtained across Zone III (60% thickness reduction), plotted against the strain hardening coefficient \(n\) of the fcc work piece.

### 3.3.5. Summary

In this study the dependence of the maximum local \(\varepsilon_p\), and the grain-to-grain variability in \(\varepsilon_p\), were characterized upon the strain-hardening rate of four fcc metal work pieces, having a strain-hardening coefficient from \(n = 0.22\) to 0.48, deformed by identical Splined Mandrel Flow Forming processes. Both the magnitude and the variability of \(\varepsilon_p\) increased with increasing strain-hardening rate. Close examination of the deformed microstructure indicated that considerably more grain-to-grain variability in the dislocation slip step and twin densities exist in the deformed material with a high strain-
hardening rate. This suggests that the inability of certain grain orientations to undergo the required plastic deformation, due presumably to their inability to cross-slip sufficiently, results in the neighbouring grains being required to deform more to accommodate the large displacements invoked during the SMFF process. This ultimately causes the high maximum local $\bar{\varepsilon}_p$ and grain-to-grain variability in $\bar{\varepsilon}_p$.

These findings are of considerable importance and should be considered when assessing the suitability of high strain forming processes, such as the SMFF process, for producing reliable, and homogeneous, parts from fcc metal alloys that display high strain hardening rates. Our findings indicate clearly the role of the strain hardening rate of fcc work pieces on the degree of grain-to-grain variability of the local plastic strain after an SMFF process. The use of micro-indentation hardness testing to map the grain-to-grain variability in the equivalent plastic strain is therefore a very useful technique for assessing the susceptibility of a material to premature local fracture in the regions of high plastic strain. This micro-indentation based test technique allows one to then obtain data from which to validate calculated equivalent plastic strain distributions derived from numerical simulations and, for end users of a metal forming technique, allows one to understand and quantify the mechanical properties of the formed work piece.
3.3.6. References


Chapter 4

Micro/nano-indentation-based assessment of the depth dependence of indentation stress, dislocations’ type, SFE, and strain rate sensitivity of the fcc metals/alloys

As discussed in Chapter 3 of this thesis, the SMFF samples of the bcc and fcc metals/alloys displayed significant strain gradients over a distance of several micrometers in the largest strain regions (Zone III in Figs. 3.1.5 and 3.2.4) of the work piece. This behaviour is contributed to the size effect which cannot be explained in such a large plastic deformation process (*i.e.* SMFF) by using continuum mechanics based analysis. Also, the large plastic strains were applied at very high deformation (strain) rate during the SMFF process. Considering these findings, the localized and severe plastic deformation invoked during the SMFF process would be very similar to that invoked during the micro/nano-indentation tests on the same fcc metals.

Nano/micro-indentation test is one of the most developed techniques which can provide information about the material behaviour when it is being deformed at the micrometer and sub-micrometer scales. Therefore, considerable nano/micro-indentation tests at various depths (from 0.2 μm to 10 μm) and strain rates were performed on the materials subjected to SMFF operations in this research.

The following chapter consists of three articles on the micro/nano-indentation assessments of the 6061 and 5052 aluminum alloys, pure copper, and 70/30.

Section 4.1 consists of a published article in “Materials Science and Technology” in which the depth dependence and strain rate sensitivity of the indentation stress of the 6061 aluminium alloy (in annealed, partially aged, and fully aged) is considered in details. Section 4.2 consists of an article published in “Materials Science and Engineering A” entitled “Assessment of the depth dependence of the indentation stress during constant strain rate nanoindentation of 70/30 brass”. In this section, the results of constant strain rate nanoindentation tests performed on annealed and 80% cold-worked 70/30 brass are presented which address the effect of indentation strain rate and indentation depth upon the indentation stress and the kinetics of the deformation process.
Section 4.3 includes an accepted article (MRS 2013 proceedings) entitled “Microindentation-based assessment of the dependence of the geometrically necessary dislocation upon depth and strain rate” in which the model of Nix and Gao was applied to calculate the density of statistically stored dislocations (SSDs) and geometrically necessary dislocations (GNDs) in fcc alloys (5052 aluminum alloy, pure copper, and 70/30 brass) under constant loading rate.

4.1. Depth dependence and strain rate sensitivity of the indentation stress of the 6061 aluminium alloy

In this research, the depth dependence and strain rate sensitivity of the indentation stress of the 6061 aluminum alloy is considered in details. Indentation tests were performed on samples of the 6061 aluminium alloy in the annealed, T₄, and T₆ temper conditions. The tests were performed over a range of loading rates to study the effect of indentation strain rate, \( \dot{\varepsilon}_{\text{ind}} \), on the indentation depth dependence of the average indentation stress \( \sigma_{\text{ind}} \). While \( \dot{\varepsilon}_{\text{ind}} \) changes by several orders of magnitude during the constant loading rate nano/micro-scale indentation tests it was observed that the strain rate sensitivity of \( \sigma_{\text{ind}} \) increases with decreasing indentation depth for all the samples tested. By applying an obstacle-limited dislocation glide description of the deformation process one is able to demonstrate that the apparent activation energy of the obstacles to dislocation glide increases with decreasing indentation depth and is also dependent upon the heat treatment condition of the 6061 aluminum test material. This suggests that, based upon the assumption of the operative deformation mechanism chosen, the strength of the dislocation-obstacle interactions that limit the rate of deformation is significantly increased in indentations of depth less than about 4 \( \mu \)m.

* A version of this chapter was published in the Materials Science and Technology. M. Haghshenas, L. Wang, R.J. Klassen, Depth dependence and strain rate sensitivity of indentation stress of 6061 aluminium alloy, Materials Science and Technology 28, 2012, 1135-1140.
4.1.1 Introduction
It is usually observed during nano/micro-scale indentation testing that the measured average indentation stress $\sigma_{\text{ind}}$ is significantly increased when the indentation depth, $h$, is less than several micrometers [1-10]. This has been explained in terms of the increased applied stress necessary for a dislocation to glide through the high density of “geometrically necessary” dislocations required to accommodate the large localized strain gradients around sub-micrometer deep indentations [2, 6, 9, 11-13] or in terms of the increased stress necessary to nucleate dislocations from a small volume of metal beneath the indentation, which may have no easy dislocation-nucleation sources. Either of these “glide limited” or “nucleation limited” mechanisms may affect the strain rate sensitivity of the indentation stress of shallow indentations compared to deep indentations. No investigation has been performed to date on the role that indentation strain rate plays on the observed indentation depth dependence of $\sigma_{\text{ind}}$. Simple dimensional analysis of a geometrically self-similar indentation, such as a pyramidal indentation, indicates that the average indentation strain rate $\dot{\varepsilon}_{\text{ind}}$ must be directly dependent upon the ratio of the indentation velocity and the indent depth $\dot{h}/h$ [8, 9, 14-20]. One would, therefore, expect that $\dot{\varepsilon}_{\text{ind}}$ would become quite large when $h$ is small. This could account, at least in part, for the observed indentation depth dependence of $\sigma_{\text{ind}}$. The objective of this research is therefore to assess the influence of indentation strain rate on the dependence of $\sigma_{\text{ind}}$ upon indentation depth and to determine if there exists a depth dependence of the strain rate sensitivity of $\sigma_{\text{ind}}$.

4.1.2. Experimental procedure
4.1.2.1. Test material
This study was performed on 6061 aluminum alloy in three standard heat treatment conditions: Annealed (O), $T_4$, and $T_6$ tempered. The annealed condition was obtained by heating the samples at 413°C for 2-3 hours. The $T_4$ temper was performed by solution annealing for 3-4 hours at 520°C followed by water quenching and then aging at room temperature for over 18 hours. The $T_6$ temper was performed with a similar
solution/quenching treatment followed by aging at 160-200°C for 6 hours. The T6 temper produced a peak-aged 6061 aluminum alloy.

The test material contained large equiaxed grains of average size from 50 to 200 μm. Cube samples, 5 mm on edge, were prepared from each thermal condition and one surface of each sample was further ground and polished to a final surface roughness of 0.05 μm.

4.1.2.2. Indentation tests
Room-temperature micro-indentation tests were performed with a diamond Berkovich indenter on a NanoTest indentation testing platform made by Micro Materials Ltd. (Wrexham, UK). Indentations were made at loading rates of 10, 100, 500, 1000, and 2000 mN/sec to a maximum load of 2000 mN in order to obtain $\sigma_{\text{ind}}$ data as a function of indentation depth at different values of $\varepsilon_{\text{ind}}$. During each test the indentation depth $h$, corrected for both thermal drift and elastic compliance of the test frame, was recorded at intervals of 100 msec. Between 3 and 5 indentation tests were performed at each loading rate on each of the three thermal conditions.

4.1.2.3. Calculation of the projected area function of the indentation
The projected area of an ideal three-sided Berkovich pyramidal indentation is expressed, in terms of the indentation depth $h$, as:

$$A_{\text{ideal}}(h) = 24.56h^2$$  \hspace{1cm} (4.1.1)

All Berkovich indenters, including the one used in this study, are not perfectly pyramidal and have a certain amount of rounding at the indenter tip (Fig. 4.1.1). The effect of this spherical tip upon the actual projected area function $A(h)$ of a pyramidal indentation has been studied extensively [21-26]. The profile of the pyramidal indenter used in this study was imaged with a scanning electron microscope and the radius $R$ of the rounded tip was found to be 500 nm (Fig. 4.1.2). When $h$ is very small, much less than $R$, a spherical indentation is created and the projected area function is

$$A(h) = \pi h(2R - h)$$  \hspace{1cm} (4.1.2)
At larger depths the indentation assumes a transitional, conical, shape between that of a sphere and a three-sided pyramid (Fig. 4.1.1). In this transitional region the projected area function is [23, 26]:

\[ A(h) = 24.56(h + 0.062R)^2 \]  

Equations 4.1.2 and 4.1.3 allow one to calculate the critical indentation depth \( h_{\text{crit}} = 0.058R \) above which the indentation transitions from spherical to conical. Since \( R = 500 \) nm for the indenter used in this study \( h_{\text{crit}} = 29 \) nm. In this research the data from indentations of depth \( h \geq 500 \) nm are analyzed. Since this depth is much larger than \( h_{\text{crit}} \), the projected area function of the indentation is given by Equation 4.1.3 and the average indentation stress is then

\[ \sigma_{\text{ind}} = \frac{P}{CA(h)} = \frac{P}{24.56C(h + 0.06R)^2} \]  

In this equation the constant \( C \) represents the effect of metal pile-up or sink-in around the indentation. Analytical and finite element simulations have shown that the magnitude of \( C \) is strongly correlated to the ratio of the indentation depth upon unloading to the indentation depth at maximum load (\( h_f/h_{\text{max}} \)) [27-31]. The natural limits for this parameter are \( 0 \leq h_f/h_{\text{max}} \leq 1 \). Metal pile-up occurs around the indenter when \( 0.875 < h_f/h_{\text{max}} \leq 1 \) while metal sink-in occurs when \( 0 \leq h_f/h_{\text{max}} < 0.875 \) [27]. For all the indentation tests performed in this study the ratio \( h_f/h_{\text{max}} \) falls within the range \( 0.875 < h_f/h_{\text{max}} < 1 \) thus metal pile-up, rather than sink-in, occurs.

The parameter \( C \) in Equation 4.1.4 was then determined experimentally by measuring, with scanning electron microscopy, the actual contact area \( A_{\text{actual}} \) of an indentation of known depth and comparing this value with the ideal projected area \( A_{\text{ideal}} \) of an indentation of the same depth, calculated from Equation 4.1.3. The parameter \( C \) is then [32, 33]:

\[ c = \frac{A_{\text{actual}}}{A_{\text{ideal}}} \]  

For a pyramidal indentation where metal pile-up occurs \( c \) is typically between 1–1.2 and should be independent of indentation depth [8, 24, 32, and 34]. In our studies \( c = 1.2 \).
4.1.3. Results

Figure 4.1.3 shows typical plots of the indentation force \( F \) versus indentation depth \( h \) for tests performed on each of the 6061 thermal conditions at each of the indentation loading rates. During pyramidal indentation \( \dot{\varepsilon}_{\text{ind}} \) is a linear function of \( \dot{h}/h \). Therefore an “apparent” average indentation strain rate can be expressed as:

\[
\dot{\varepsilon}_{\text{ind}} = \frac{1}{h} \left( \frac{dh}{dt} \right) 
\]

The dependence of \( \sigma_{\text{ind}} \) and \( \dot{\varepsilon}_{\text{ind}} \) upon \( h \) are shown in Figs. 4.1.4 and 4.1.5. Both \( \sigma_{\text{ind}} \) and \( \dot{\varepsilon}_{\text{ind}} \) increase significantly, by several orders of magnitude in the case of \( \dot{\varepsilon}_{\text{ind}} \), when \( h \) is less than about 4 \( \mu \text{m} \). In the case of the indentation stress, \( \sigma_{\text{ind}} \) increased to up to 6.5 GPa for the smallest indentation depth considered (\( h = 500 \text{ nm} \)).

Figure 4.1.1: Schematic representation of a three-sided Berkovich pyramidal indenter with its tip blunted to a radius of \( R = 500 \text{ nm} \). The indentation contact area \( A(h) \) resulting from this indenter is given by Eq. (4.1.3).
Figure 4.1.2: SEM images of Berkovich indenter which was used in the present study. The radius of the indenter tip is 500 nm (Top view and side view).
Increasing loading rate

Load Rate = 10 mN/sec
Load Rate = 500 mN/sec
Load Rate = 1000 mN/sec
Load Rate = 2000 mN/sec
Figure 4.1.3: Indentation force versus indentation depth curves for the 6061 aluminium alloy test material, indented at four loading rates, in the (a) 6061-O, (b) 6061-T₄, and (c) 6061-T₆ thermal conditions.

While this magnitude of $\sigma_{\text{ind}}$ is large, and clearly reflects the commonly observed indentation size effect, its magnitude is considerably less than Hertzian elastic contact stress $P_{\text{Hert}}$, which is given, for a rigid spherical indenter, of radius $R$, indenting a flat surface, as [35, 36]:

$$P_{\text{Hert}} = \frac{3F}{2\pi a^2} \quad 4.1.7$$

where the contact radius $a$ is

$$a = \left( \frac{3FR}{4E_r} \right)^{1/3} \quad 4.1.8$$
and $E_r$ is the reduced elastic modulus. The maximum Hertzian elastic contact stress, which occurs for $F$ corresponding to the indentation force at the smallest indentation depth of this study ($h = 500$ nm) and $R = 500$ nm, is $P = 59$ GPa.
Figure 4.1.4: Average indentation stress $\sigma_{\text{ind}}$ (Eq. (4.1.4)) versus indentation depth for the 6061 aluminum alloy test material, indented at four loading rates, in the (a) 6061-O, (b) 6061-T4, and (c) 6061-T6 thermal conditions.
Load Rate = 10 mN/sec
Load Rate = 100 mN/sec
Load Rate = 500 mN/sec
Load Rate = 1000 mN/sec
Load Rate = 2000 mN/sec
Figure 4.1.5: Apparent average indentation strain rate $\dot{\varepsilon}_{\text{ind}}$ (Eq. (4.1.6)) versus indentation depth $h$ for the 6061 aluminum alloy test material, indented at four loading rates, in the (a) 6061–O, (b) 6061–T4, and (c) 6061–T6 thermal conditions.

4.1.4. Discussion

4.1.4.1. The strain rate sensitivity of indentation stress

The 6061 aluminum alloy test material shows a nonlinear logarithmic dependence of $\sigma_{\text{ind}}$ upon $\dot{\varepsilon}_{\text{ind}}$ (Fig. 4.1.6). The data in this figure were obtained from indentation depths ranging from 0.5 to 8.0 $\mu$m. Since indentation tests were performed at a range of loading rates we can extract, from Fig. 4.1.6, data that show the dependence of $\sigma_{\text{ind}}$ upon $\dot{\varepsilon}_{\text{ind}}$ at specific levels of $h$. 
Figure 4.1.6: Logarithmic plots of $\sigma_{\text{ind}}$ versus $\dot{\varepsilon}_{\text{ind}}$ at a loading rate of 10 mN/sec for the 6061–O (annealed), 6061–T4, and 6061–T6 thermal conditions.

Figure 4.1.7 shows logarithmic plots of $\sigma_{\text{ind}}$ versus $\dot{\varepsilon}_{\text{ind}}$ for indentations of nine depths from $h = 0.5$ to 8.0 $\mu$m. The strain rate sensitivity increases with decreasing $h$. This can be illustrated by performing linear regression analysis of the data in Fig. 4.1.7 and plotting the measured slope, the strain rate sensitivity $m$, as a function of indentation depth (Fig. 4.1.8). The parameter $m$ displays a clear dependence upon both $h$ and the heat treatment condition of the 6061 aluminium alloy.
Figure 4.1.7: logarithmic plots of $\sigma_{\text{ind}}$ versus $\dot{\varepsilon}_{\text{ind}}$ at different indentation depths for a: 6061-O, b: 6061-T4, and c: 6061-T6 thermal conditions.

When the indentations are deep ($h > 4.0 \, \mu m$), the value of $m$ is less than 0.02, and is in good agreement with values of $m$ obtained from constant uniaxial strain rate, and strain rate change, tests performed on 6061 aluminium alloy tensile samples [37]. The magnitude and the test-to-test scatter of $m$ increases significantly with decreasing indentation depth.
4.1.4.2. Depth-dependent mechanism of indentation deformation

The indentation depth dependence of $m$ (Fig. 4.1.8) suggests that, for the 6061 aluminium alloy samples tested in our study, there exists an indentation depth dependence of the underlying deformation mechanism. This dependence may arise from either increased difficulty to glide a dislocation through the indentation plastic zone or the increased difficulty to nucleate a dislocation in the region of a shallow, compared to a deep, indentation. Either mechanism will cause a change in the strain rate sensitivity of $\sigma_{\text{ind}}$.

By assuming a time-dependent deformation during indentation as an obstacle-limited dislocation glide process where $\dot{\varepsilon}_{\text{ind}}$ can be expressed as [38, 39]:

$$\dot{\varepsilon}_{\text{ind}} = \dot{\varepsilon}_0 \left( \frac{\sigma_{\text{ind}}}{E} \right)^2 e^{\frac{-\Delta G_{\text{thermal}}(\sigma_{\text{ind}})}{kT}}$$  \hspace{1cm} (4.1.9)

In this equation $\Delta G_{\text{thermal}}(\sigma_{\text{ind}})$ is the thermal energy that must be supplied in order for; i) a dislocation to either overcome an obstacle that exists within the microstructure (i.e. a
dislocation glide limited mechanism) or ii) a new dislocation to be nucleated within the indented material (i.e. a dislocation nucleation limited mechanism). \( \Delta G_{\text{Thermal}}(\sigma_{\text{ind}}) \) is clearly stress dependent and will decrease when \( \sigma_{\text{ind}} \) is large. The term \( \left( \frac{\sigma_{\text{ind}}}{E} \right)^2 \) is proportional to the dislocation density, \( \dot{\varepsilon}_0 = 10^{11}\text{sec}^{-1} \) is a material constant [40], \( k \) is Boltzmann constant, and \( T \) is temperature in Kelvin.

Figure 4.1.9 shows the calculated \( \Delta G_{\text{Thermal}}(\sigma_{\text{ind}}) \) versus \( \dot{\varepsilon}_{\text{ind}} \) for indentations made at depths \( h \) from 0.5 to 8.0 \( \mu \text{m} \). The fact that the dependence of \( \Delta G_{\text{Thermal}}(\sigma_{\text{ind}}) \) upon \( \dot{\varepsilon}_{\text{ind}} \) is dependent upon both \( h \) and heat treatment condition indicates that the strength of the obstacles changes with both these parameters. Since \( \Delta G_{\text{Thermal}}(\sigma_{\text{ind}}) \) decreases with increasing \( \sigma_{\text{ind}} \) and \( \dot{\varepsilon}_{\text{ind}} \) one can extrapolate the data trends in Fig. 4.1.9 and define \( \Delta G_{\text{Thermal}}(\sigma_{\text{ind}}) \) occurring at a very low strain rate, say \( \dot{\varepsilon}_{\text{ind}} = 0.001\text{sec}^{-1} \), as the apparent activation energy \( \Delta G_0 \) of the obstacles that are limiting the dislocation nucleation/glide process. Figure 4.1.10 shows the resulting \( \Delta G_0 \) versus \( h \) for the three 6061 aluminum alloy tested at different heat treatment conditions. One can clearly see that the activation strength \( \Delta G_0 \) of the deformation rate controlling obstacles, whether they are physical obstacles impeding dislocation glide or represent nucleation stress to create a dislocation in the otherwise perfect material beneath the indenter, increases when \( h \) is small, less than about 4 \( \mu \text{m} \), and increases, for any value of \( h \), when the 6061 aluminum alloy is in the tempered, \( T_4 \) and \( T_6 \), conditions. Since the difference in these two thermal conditions relative to the annealed condition is the presence of coherent “particles or solute clusters”, one can deduce that \( \Delta G_0 \) must be dependent, at least in part, upon physical obstacles that pre-exist in the microstructure.
4.1.5. Conclusions

This study of the effect of indentation strain rate, $\dot{\varepsilon}_{\text{ind}}$, on the indentation depth dependence of the average indentation stress $\sigma_{\text{ind}}$ of the 6061 aluminum alloy has indicated that while $\dot{\varepsilon}_{\text{ind}}$ decreases by several orders of magnitude as indentation depth increases during a typical nano/micro-scale indentation test, its affect on $\sigma_{\text{ind}}$ does not completely account for the commonly observed depth dependence of $\sigma_{\text{ind}}$. This is confirmed by our observation that the strain rate sensitivity $m$, determined from indentations of a constant depth but differing values of $\dot{\varepsilon}_{\text{ind}}$, increases with decreasing indentation depth.

Figure 4.1.9: Plots of the thermal activation energy $\Delta G_{\text{thermal}}(\sigma_{\text{ind}})$ (Eq. (4.1.9)) versus $\dot{\varepsilon}_{\text{ind}}$ at different indentation depths for the 6061–O, 6061–T4, and 6061–T6 thermal conditions.
Figure 4.1.10: Variation of the apparent activation strength $\Delta G_0$ of the obstacles that limit dislocation glide versus indentation depth $h$. $\Delta G_0$ was taken as the value of $\Delta G_{\text{Thermal}}(\sigma_{\text{ind}})$ at a very low indentation strain rate $\dot{\varepsilon}_{\text{ind}} = 0.001$ sec$^{-1}$. $\Delta G_0$ increases with decreasing $h$ for the three material conditions tested which suggests that the nature of the operative dislocation/obstacle interaction is indentation depth dependent.

The depth dependence of the strain rate sensitivity of $\sigma_{\text{ind}}$ is analyzed by assuming that the deformation during indentation occurred, for all depths from 500 nm to 8 $\mu$m, by a thermally-activated dislocation-nucleation/glide limited process. The apparent activation energy of the obstacles that either interfere with the glide of existing dislocations or represent sites for the nucleation of new dislocations was calculated and found to increase with decreasing indentation depth.

While the data, and the analysis, presented in this research cannot identify definitively which type of feature, obstacles that limit the glide of pre-existing dislocations or obstacles that represent dislocation nucleation sources, are responsible for the observed increase in the strain rate sensitivity of $\sigma_{\text{ind}}$ when the indentation depth is small, our
observation that the apparent activation strength of these features also increases with tempering of the 6061 aluminium alloy suggests that pre-existing obstacles to dislocation glide have an important influence on the strain rate sensitivity of $\sigma$ of the 6061 aluminum alloy over all the indentation depths tested.

### 4.1.6. References


4.2. Assessment of the depth dependence of the indentation stress during constant strain rate nanoindentation of 70/30 brass

The results of constant strain rate nanoindentation tests performed on annealed and 80% cold-worked 70/30 brass are presented in this section which address the effect of indentation strain rate and indentation depth upon the indentation stress and the kinetics of the deformation process. The analysis of the parameters that influence dislocation thermal activation could shed light on rate controlling mechanisms in the plastic deformation (i.e. SMFF) of studied materials (i.e. 70/30 brass) in the present research. At any given indentation depth from 0.2 to 2.0 μm the strain rate sensitivity $m$, and the apparent activation energy $\Delta G_0$ of the deformation process show a clear dependence upon indentation depth and prior cold-work. The data from all indentation tests, regardless of indentation depth, fall on the same linear trend of apparent activation work $\Delta W'$ versus $\Delta G_0$ however the annealed material displayed a distinctly different linear trend than the 80% cold-worked material. This suggests that the operative dislocation-obstacle mechanism that occurs during the indentation is essentially independent of indentation depth but depends upon the prior dislocation density of the indented material.

4.2.1. Introduction

Pyramidal indentation testing is typically performed under conditions of constant indentation loading rate, $\dot{P}$, or constant indentation displacement rate, $\dot{h}$ [38]. In either case, the average indentation strain rate, $\dot{\varepsilon}^{\text{ind}} = \dot{h}/h$, decreases with increasing indentation depth $h$. In order to understand the dependence of the underlying mechanisms of indentation deformation one must perform indentation tests under conditions of constant $\dot{\varepsilon}^{\text{ind}}$. This is particularly of interest over the indentation depth range from

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\[ h = 0.1 - 5.0 \mu m \] where the average indentation stress \( \sigma_{ind} \) is observed to be strongly indentation depth dependent.

Previously the results from indentation tests performed on the 6061 aluminum alloy were reported and observed that the strength \( \Delta G_0 \) of the dislocation-obstacle interactions that limit the rate of deformation was significantly increased when the indentation depth was less than about 4 \( \mu m \) [11]. Since these tests were performed under constant \( \dot{P} \) conditions they must be corroborated with indentation tests performed under constant \( \dot{\varepsilon}_{ind} \) conditions to confirm our reported findings regarding the depth dependence of the operative mechanism of plastic deformation during indentation.

Lucas and Oliver [41] were the first to propose that constant \( \dot{\varepsilon}_{ind} \) indentation tests could be performed when the indentations are made under conditions of constant \( \dot{P}/P \). Since then several investigator have extended their work to show that even in the cases where the indentation depth was small, indentation performed under constant \( \dot{P}/P \) still resulted in constant \( \dot{\varepsilon}_{ind} \) [42-44].

The results from nanoindentation tests are reported that were performed under conditions of constant \( \dot{P}/P \) to investigate the effect of \( \dot{\varepsilon}_{ind} \) upon the indentation depth dependence of \( \sigma_{ind} \) for annealed and cold-worked 70/30 brass. These data are then used to calculate the indentation depth dependence of the apparent activation energy \( \Delta G_0 \) and the activation volume \( V^* \) of the underlying obstacle-limited dislocation glide mechanism that operates during the indentation process.

### 4.2.2. Experimental procedure

The 70/30 brass alloy used in his study was initially annealed at 500\(^\circ\)C for 3–4 hours. The material displayed a large number of annealing twins within equiaxed grains of about 100 \( \mu m \) diameter (Fig. 4.2.1). Several samples of the annealed brass were then deformed to 80% thickness reduction by plane-strain cold rolling.

Nanoindentation tests were performed on small cubical samples cut from the annealed and the cold–worked materials. In preparation for testing the samples were, at first,
mechanically ground and polished to a finish of 0.05 μm alumina powder. The samples were then gently polished using colloidal silica which slightly etches the specimens [45]. Finally, to remove any deformation introduced earlier during mechanical polishing, the samples were chemically polished/etched. Chemical polishing/etching is, in fact, an alternative to electro-polishing, to reach a strain-free surface, which does not require as much setup time and experimentation for proper operating conditions [45, 46]. In the chemical polishing, the sample surface is immersed in or swabbed with (for several seconds or minutes) a chemical solution that dissolves the surface of material at a uniform rate. This technique removes surface layers of materials without inducing mechanical deformation such that the polished surface is free of deformation and smeared layers that may occur after mechanical polishing.

Figure 4.2.1: Optical micrographs of the annealed 70/30 brass material. The microstructure contains large equiaxed grains along with annealing twins (AT).
The samples were immersed for 2 minute in a chemical solution of 25 ml phosphoric acid, 25 mL glacial acetic acid, 25 mL nitric acid, 0.5 mL hydrochloric acid at 64°C [45]. The chemical polishing solution was removed by rinsing the specimen in methanol and blowing it dry in air.

Constant $\dot{P}/P$ indentation tests were performed at 25°C on the annealed and the cold-worked brass samples with a nanoindentation tester made by Micro-Materials Ltd (Wrexham, UK). By maintaining constant $\dot{P}/P$ during the indentation process ensured that the average indentation strain rate $\dot{e}_{ind} = \dot{h}/h$ was maintained. The indentation tests were performed at constant strain rates of $\dot{e}_{ind} = 0.005, 0.05, 0.5,$ and $1.0 \text{ sec}^{-1}$ to indentation depths of $h = 0.2, 0.4, 0.8,$ and $2.0 \mu\text{m}$. Triplicate indentation tests were performed at each condition of $h$ and $\dot{e}_{ind}$ for the annealed and the cold-worked brass materials. A total of 96 indentation tests were therefore performed in this study.

A three–sided pyramidal diamond indenter (Berkovich) was used to perform the nanoindentation tests. This indenter had a tip radius of $R \approx 100\text{nm}$ as measured by scanning electron microscopy. The minimum indentation depth for self-similar deformation in this experimental study was estimated to be $h_{\text{crit.}} \approx 6\text{nm}$ by using the previously reported equation $h_{\text{crit.}} \approx R \left(1 - \sin 70.3^\circ\right) = 0.06R$ [47-50].

The projected indentation contact area was calculated as [22-24, 26, 51]:

$$A(h) = 24.56(h + 0.06R)^2$$  \hspace{1cm} 4.2.1

and the average indentation stress was calculated as:

$$\sigma_{ind} = \frac{P}{cA(h)} = \frac{P}{24.56c(h + 0.06R)^2}$$  \hspace{1cm} 4.2.2

where the constant $C$ represents the effect of metal pile–up or sink–in around the indentation. In our studies, values of $c$ previously reported for annealed and cold-worked copper [32] were used; namely, $c=0.9$ (annealed brass) and $c=1.2$ (cold-worked brass).
4.2.3. Results

4.2.3.1. Effect of $\dot{P}/P$ on the indentation $P$–$h$ curves

Figure 4.2.2 shows curves of indentation force $P$ versus depth $h$ from tests performed under various levels of $\dot{P}/P$ on the annealed and the cold-worked 70/30 brass. Although these figures do not show data from all the indentation tests performed in this study, the shape of the curves shown is typical of the other indentation tests performed. The magnitude of $P_{\text{max}}$, is approximately three-times larger for the 80% cold-worked brass than for the annealed brass at indentation depth of 2.0 $\mu$m. This indicates the increased flow stress of the cold-worked material. At any given indentation depth, $P$ increases with increasing $\dot{\varepsilon}_{\text{ind}}$ (that is increasing $\dot{P}/P$). This indicates the strain rate sensitivity of the flow stress of the 70/30 brass material.
Figure 4.2.2: Indentation load $P$ versus indentation displacement $h$ curves from tests performed at various levels of $\dot{P}/P$ from 0.005 to 1.0 sec$^{-1}$ on 70/30 brass for shallow indentations ($h=0.2$ µm) and deep indentations ($h=2.0$ µm) in the: a) annealed, and b) 80% cold-worked conditions.

Figure 4.2.3 shows a plot of $\dot{\varepsilon}_{\text{ind}} = \dot{h}/h$ versus $h$ for one test performed at each level of $\dot{P}/P$ on the cold-worked brass sample. Over the indentation depths of interest in this study, $h = 0.2$ to 2.0 µm, maintaining constant $\dot{P}/P$ resulted in constant $\dot{\varepsilon}_{\text{ind}}$ with $\dot{\varepsilon}_{\text{ind}} \approx 0.5\dot{P}/P$. 
4.2.3. Apparent average indentation strain rate

Figure 4.2.3: Apparent average indentation strain rate $\dot{\varepsilon}_{\text{ind}} = \dot{h}/h$ versus indentation depth $h$ for 80% cold-worked 70/30 brass indented at different values of $\dot{P}/P$. The average indentation strain rate is fairly constant, and approximately equal $0.5 \dot{P}/P$, across the range of indentation depth from $h = 0.2$ to $2.0 \mu$m.

4.2.3.2. Indentation depth dependence of $\sigma_{\text{ind}}$

Figure 4.2.4 shows the average indentation stress $\sigma_{\text{ind}}$ (Eq. 4.1.4) versus indentation depth $h$ for both the annealed and the cold-worked brass samples. $\sigma_{\text{ind}}$ decreases with increasing $h$ and, thus, displays the well-known indentation depth dependence of the indentation stress. At any value of $h$, the annealed brass shows considerably lower $\sigma_{\text{ind}}$ than the cold-worked brass. The indentation depth at which $\sigma_{\text{ind}}$ becomes essentially independent of $h$ is also larger in the cold-worked sample. At any indentation depth, $\sigma_{\text{ind}}$ increases with increasing average indentation strain rate $\dot{\varepsilon}_{\text{ind}}$ however the degree of this strain rate sensitivity appears to also be dependent upon $h$. 
Assessing the effect of prior cold-work and strain rate on the indentation depth dependence of the operative deformation mechanisms which ultimately determine $\sigma_{ind}$ are of particular interest. The trends of $\sigma_{ind}$ versus $h$ shown in Fig. 4.2.4 may be influenced by the presence of thin work-hardened surface layers resulting from the sample preparation steps (section 4.2.2). Since the same preparation steps were performed on both materials, their effect on $\sigma_{ind}$ should be the same for both the annealed and the cold-worked sample. Thus in Fig. 4.2.5 a plot of $\sigma_{ind,cold-worked}/\sigma_{ind,annealed}$ versus indentation depth is shown. This normalized plot removes the effect of sample preparation induced cold-work and indicates that, since $\sigma_{ind,cold-worked}/\sigma_{ind,annealed}$ decreases when $h$ is less than about 0.4 $\mu$m, $\sigma_{ind}$ of the annealed sample shows a different indentation depth dependence than that of the cold-worked brass material and this difference is dependent upon the average indentation strain rate. These dependences will be considered in detail in the subsequent sections.

4.2.3.3. Strain rate sensitivity of $\sigma_{ind}$

It is common to express the strain rate sensitivity of the flow stress of ductile metals in terms of a power-law function. When applied to an indentation test, performed with a geometrically self-similar indentation such as the Berkovich pyramid used in this study, this results in the following equation

$$\sigma_{ind} = D \dot{\varepsilon}_{ind}^m$$  \hspace{1cm} (4.2.3)

where $D$ is a temperature dependent constant and $m$ is the strain rate sensitivity parameter. In this study the data obtained from the indentation tests performed at different levels of $\dot{P}/P$ were grouped into nine specific depths from $h = 0.2$ to $2.0$ $\mu$m and, for each depth, the $m$ parameter were evaluated by performing a linear regression on logarithmic plots of $\sigma_{ind}$ versus $\dot{\varepsilon}_{ind}$. 

Figure 4.2.4: Average indentation stress $\sigma_{\text{ind}}$ versus indentation depth $h$ at different strain rates for the a) annealed, and b) 80% cold-worked 70/30 brass alloy. Error bars indicating the magnitude of the scatter in $\sigma_{\text{ind}}$ across triplicate tests performed under the same conditions is shown for the shallow and the deep indentations.
Figure 4.2.5: Variation in the normalized indentation stress, $\frac{\sigma_{\text{ind\_cold\_worked}}}{\sigma_{\text{ind\_annealed}}}$, versus indentation depth, $h$, for tests performed at four levels of indentation strain rate $\dot{\varepsilon}_{\text{ind}}$. The error bars on the plots indicate the scatter in $\frac{\sigma_{\text{ind\_cold\_worked}}}{\sigma_{\text{ind\_annealed}}}$ arising from the multiple indentation tests performed under each condition.

Figure 4.2.6 shows a plot of the resulting strain rate sensitivity parameter $m$ versus indentation depth $h$ for both the annealed and the cold-worked brass. The parameter $m$ displays a dependence upon both $h$ and cold-work. When the indentations are deep ($h \geq 1.0 \mu m$), $m$ is between 0.005 and 0.001 and is, therefore, similar in magnitude to previously reported values obtained, by uniaxial tensile testing, for coarse-grained fcc metals [52-55]. The observed depth dependence of $m$ is similar to what was reported from tests performed under constant $\dot{P}$ conditions for 6061 aluminium alloy material [56].
4.2.4. Discussion

4.2.4.1. Depth dependency of indentation deformation

The results of our indentation tests performed at four levels of constant indentation strain rate indicate that both the strain rate sensitivity and the indentation depth dependence of the average indentation stress $\sigma_{\text{ind}}$ is dependent upon prior cold-work (Figs. 4.2.4 to 4.2.6). This agrees with the previous work of Abu Al-Rub and Voyiadjis [57] that showed that the indentation size effect is expected to be influenced by prior dislocations and the additional work hardening that occurs during indentation.

![Graph showing the variation of indentation strain rate sensitivity parameter, $m$ (Eq. 4.2.3), versus indentation depth, $h$, in the annealed and the 80% cold-worked 70/30 brass samples. The strain rate sensitivity of $\sigma_{\text{ind}}$ clearly increases with decreasing $h$. The error bars indicate the range of scatter in the measured $m$ for triplicate indentation tests performed at each condition.](image)

Our observation that the strain rate sensitivity parameter $m$ is higher for the cold-worked compared to the annealed brass samples indicates that cold-working affects the
underlying process of obstacle-limited thermally-activated dislocation glide. This observation has also been made by others who investigated the effect of cold-work on the strain rate sensitivity of several metal systems including Cu, Al, Nb, and Ta [58-60].

When analysing the dependence of the strain rate sensitivity upon both $h$ and prior cold-work one must consider the process of plastic strain via a mechanism of time-dependent obstacle-limited dislocation glide. The following Arrhenius-type equation describes the equivalent indentation shear strain rate $\dot{\gamma}_{\text{ind}} = \sqrt{3}\dot{\varepsilon}_{\text{ind}}$ resulting from indentation, performed at an equivalent indentation shear stress $\tau_{\text{ind}} = \sigma_{\text{ind}}/3\sqrt{3}$ and temperature $T$ as [40]:

$$\dot{\gamma}_{\text{ind}} = \dot{\gamma}_p \left(\frac{\tau_{\text{ind}}}{\mu}\right)^2 \exp\left[-\frac{\Delta G_{\text{Thermal}}(\tau_{\text{ind}})}{kT}\right]$$ \hspace{1cm} 4.2.4

where $\dot{\gamma}_p$ is a constant (approximately equal to $10^{11}$ sec$^{-1}$ [40]), $\mu$ is the elastic shear modulus at room temperature (taken to be 37 GPa for the 70/30 brass [61]), $k$ is Boltzmann’s constant, and $\Delta G_{\text{Thermal}}$ is the thermal energy necessary for the dislocation, subjected to $\tau_{\text{ind}}$, to overcome the obstacle that limits its glide. $\Delta G_{\text{Thermal}}$ can be written as:

$$\Delta G_{\text{Thermal}} = \Delta G_0 - V^*\sigma_{\text{ind}}$$ \hspace{1cm} 4.2.5

where $\Delta G_0$ is the apparent activation energy of the obstacles that limit the dislocation motion, and $V^*$ is the apparent activation volume.

Equations 4.2.4 and 4.2.5 were used to analyse the $\sigma_{\text{ind}}$ versus $h$ data obtained from our experiments and, in so doing, calculated values of $\Delta G_0$ and $V^*$ for indentation tests performed, on the annealed and the 80% cold-worked brass samples, at nine indentation depths between $h = 0.2$ and 2.0 $\mu$m. Figure 4.2.7 shows a plot of the calculated apparent activation volume $V^*$, normalized with respect to the Burgers vector cubed ($b_{\text{brass}} = 0.26$ nm [61]), versus indentation depth for the annealed and 80% cold-worked brass at different indentation strain rates. At the early stages of indentation ($h < 1.0$ $\mu$m), $V^*$ increases with increasing indentation depth but becomes independent of depth when $h$
was large. The dependence of $V^*$ upon indentation depth is in agreement with the findings from displacement rate change indentation tests performed on pure polycrystalline Cu [62] and with findings from indentation tests performed on fcc and bcc metals [55]. Figure 4.2.7 indicates that, for a given value of $h$, the annealed brass shows higher $V^*$ than the cold-worked brass. Also, $V^*$ decreases, for both materials, with increasing $\dot{\varepsilon}_{ind}$.

Figure 4.2.8 shows the $\Delta G_{\text{Thermal}}$, calculated from the indentation data using Eq. 4.2.4, versus $\dot{\varepsilon}_{ind}$ for indentations made, at depths from 0.2 to 2.0 $\mu$m for both the annealed and the cold-worked brass samples. $\Delta G_{\text{Thermal}}$ is dependent upon the indentation depth and the degree of cold-work of the indented material. The data trends in Fig. 4.2.8 can be extrapolated to very low strain rates, say $\dot{\varepsilon}_{ind} = 0.0005$ sec$^{-1}$ which approximates $\sigma_{ind} = 0$, to estimate the apparent average activation energy $\Delta G_0$ of the obstacles.

Figure 4.2.9 shows a plot of the resulting $\Delta G_0$ versus $h$ for the annealed and the 80% cold-worked brass. The activation strength $\Delta G_0$ of the deformation rate controlling obstacles depends upon both indentation depth and prior plastic deformation. This is in good agreement with our previously published work on the depth dependence of the indentation deformation of Al alloys [56].
Figure 4.2.7: Normalized activation volume, $V^*/b^3$, versus indentation depth for the a) annealed, and b) 80% cold-worked 70/30 brass. $V^*/b^3$ first increases with increasing $h$ but tends to become constant when $h$ is large. The error bars indicate the range of scatter in $V^*/b^3$ for triplicate indentation tests performed at conditions of the highest and the lowest indentation strain rate $\dot{\varepsilon}_{ind}$.
The data presented above indicate that when indentation is performed with a geometrically self-similar indenter under conditions of constant indentation strain rate the resulting values of \( m \), \( \Delta G_0 \), and \( V^* \) are dependent upon both indentation depth and the level of prior plastic deformation of the 70/30 brass material.

To assess these dependencies further we depict the data from our indentation tests on Haasen plots of inverse activation area, \( 1/\Delta a \), versus applied equivalent indentation shear stress, \( \tau_{ind} \). Assessing how the shape of these curves is affected by indentation depth and degree of cold work is of particular interest. Previous analyses have shown that linear dependence of \( 1/\Delta a \) with \( \tau \) result from plastic deformation that occurs by a mechanism of dislocation glide that is limited by dislocation-dislocation interactions [63, 64]. The slope of this linear trend is inversely proportional to the mechanical activation work \( \Delta W \) of the obstacles that limit the rate of dislocation glide [65].

Figure 4.2.10 shows plots of \( b^2/\Delta a \) versus \( \tau_{ind} \) for the indentation tests performed on the annealed and the 80% cold-worked 70/30 brass at different indentation depths from 0.2 to 1.8 \( \mu \)m. The data show essentially linear trends with the slope increasing with indentation depth.
Figure 4.2.8: Plots of the thermal activation energy, $\Delta G_{\text{Thermal}}$, versus indentation strain rate at different indentation depths for the (a) annealed and (b) 80% cold-worked 70/30 brass material. The error bar in each plot indicates the typical range of scatter of $\Delta G_{\text{Thermal}}$ for triplicate indentation tests performed under identical test conditions.
Figure 4.2.9: Variation of the apparent activation strength $\Delta G_0$ of obstacles that limit the dislocation glide during indentation versus indentation depth $h$ for both the annealed and the 80% cold-worked 70/30 brass materials. The data of all three tests at each strain rate have been shown.

The activation strength $\Delta G_0$ of the obstacles to dislocation glide can be expressed as the sum of the thermal activation energy $\Delta G_{\text{Thermal}}$ (Eqs. 4.2.4, 4.2.5) and the mechanical activation work, $\Delta W = V^* \sigma_{\text{ind}}$. Therefore, $\Delta G_0$ (Fig. 4.2.9) is linearly related to $\Delta W$. Figure 4.2.11 shows a plot of $\Delta G_0$ versus the apparent activation work $\Delta W'$, calculated as the reciprocal of the slope of the curves in Fig. 4.2.10, for the annealed and the 80% cold-worked 70/30 brass material. This plot indeed shows linear relationship between $\Delta G_0$ and $\Delta W'$. While the linear trends include data from all indentations, regardless of depth, it is interesting to note that the data from the cold-worked brass follow a distinctly different linear trend than those from the annealed brass sample. At any value of $\Delta W'$, $\Delta G_0$ is larger for the cold-worked than for the annealed material. Since $\Delta W = \tau_{\text{ind}} b \Delta \alpha$, and $\tau_{\text{ind}}$ is larger for the cold-worked material, this would indicate that the apparent activation area $\Delta \alpha$ is much smaller for the cold-worked material than for the annealed material.
Figure 4.2.10: $b^2/\Delta a$ versus $\tau_{\text{ind}}$ for the indentation tests performed on the (a) annealed, and (b) 80% cold-worked brass at different indentation depths from 0.2 $\mu$m to 1.8 $\mu$m.
Since the data from both the shallow and the deep indentations fall on the same linear trend in Fig. 4.2.11, it can be concluded that the actual activation length characteristic of the obstacles to dislocation glide in either the annealed or the cold-worked brass material must be considerably less than the 0.2 μm depth of the shallowest indentations tested. This is reasonable since all our data indicated that the rate of dislocation glide is controlled by dislocation-dislocation type interactions.

![Graph](image)

**Figure 4.2.11:** Linear relationship between $\Delta G_0$ and $\Delta W'$ for the annealed and the 80% cold-worked 70/30 brass material. The data of all three tests at each strain rate have been shown.

4.2.4.3. Role of twins in the indentation deformation process

Figure 4.2.12 shows optical micrographs of the 80% cold-worked brass material. Comparing this figure with that of the annealed material (Fig. 4.2.1) indicates that the 80% cold-work has resulted in extensive dislocation slip and mechanical twinning. In fcc metals with low stacking fault energy ($\text{SFE}_{70/30\text{brass}} = 14 \text{ mJm}^{-2}$ [66, 67]), mechanical twining is a dominant deformation mode compared with dislocation slip such that the percentage of twinned material is expected to increase with plastic deformation [67, 68].
Since twinning is, essentially a variety of dislocation glide, involving the motion of partial instead of complete dislocations, the description of the operative deformation mechanism in terms of Eqs. 4.2.4 and 4.2.5 is valid and the parameter of $\Delta G_0$, $\Delta a$, and $\Delta W'$ shown in Figs. 4.2.9–4.2.11, reflect the motion and interaction of a dislocation population consisting of widely spaced partial dislocations.

![Micrograph of cold-worked 70/30 brass material](image1)

**Figure 4.2.12:** Optical micrographs of the cold-worked 70/30 brass material at two magnifications. The microstructure contains elongated grains/twins and mechanical twin (MT) clusters in a few grains.
4.2.5. Conclusions

The results from constant strain rate nanoindentation tests performed on annealed and 80% cold-worked 70/30 brass are reported to investigate the effect of $\dot{\varepsilon}_{ind}$ upon $\sigma_{ind}$ and thereby deduce the effect of indentation depth $h$, over the range from 0.2 to 2.0 $\mu$m, on the operative mode of plastic deformation.

At any given indentation depth, $\sigma_{ind}$ increases with increasing indentation strain rate and both the strain rate sensitivity $m$, and the apparent activation energy $\Delta G_0$ of the deformation process show clear dependence upon indentation depth and prior cold-work. The $\Delta G_0$ of the obstacles that limit dislocation glide during indentation decreased from 0.63 to 0.57 eV and from 0.58 to 0.48 eV with increasing indentation depth for the cold-worked and the annealed brass.

The data from our tests were then analysed by creating Haasen plots of $b^2/\Delta a$ versus $\tau_{ind}$. The data fell on linear trend of decreasing slope with decreasing indentation depth. The reciprocal of the slope is proportional to the activation work $\Delta W$ of the deformation process. The data from all indentation tests, regardless of indentation depth, fall on the same linear trend of $\Delta W'$ versus $\Delta G_0$, however, the annealed material displayed a distinctly different linear trend than the 80% cold-worked material. The fact that such linear trends exist suggest that the basic operative dislocation-obstacle mechanism during the indentation is essentially independent of indentation depth and the fact that $\sigma_{ind}$ is clearly depth dependent only results from an increase in the local dislocation density around shallow, compared to deep, indentations. This is in agreement with the theories proposed by others (Nix and Gao [2]). Our observation that the annealed 70/30 brass displays a different linear relationship between $\Delta W$ and $\Delta G_0$ than the cold-worked brass suggests that, despite the fact that $\tau_{ind}$ is larger, the activation area of the obstacles in the cold-worked material is very small, and the activation length of the obstacles is considerably less than the depth, $h = 0.2$ $\mu$m, of the smallest indentations tested in this study.
Our optical micrographs clearly show the existence of mechanical twinning in the microstructure of the 80% cold-worked 70/30 brass material. The values of $\Delta G_0$, $\Delta a$, and $\Delta W'$ determined in this study therefore are representative of an obstacle-limited dislocation glide process involving partial dislocations.

4.2.6. References


4.3. Microindentation-based assessment of the dependence of the geometrically necessary dislocation upon depth and strain rate

In this section an assessment is presented of the indentation depth and strain rate dependency of “geometrically necessary” dislocations. Pyramidal microindentation tests were performed at various loading rates from 1 to 1000 mN/sec on annealed samples of pure copper, 70/30 brass, and 5052 aluminum alloy to study the effect of indentation strain rate on the indentation depth dependence, from \( h = 0.5 \) to 9 \( \mu \text{m} \), of the average indentation stress. The average indentation stress displayed a clear strain rate sensitivity which was dependent upon indentation depth. The model of Nix and Gao was applied to calculate the density of statistically stored dislocations (SSDs) and geometrically necessary dislocations (GNDs) as a function of indentation depth. The GND density displayed the characteristic decrease with increasing \( h \). The average indentation stress, \( \sigma_{\text{ind}} \) was observed to decrease with increasing \( h \) and, for any given \( h \) increase with increasing loading rate.

This observed dependence of \( \sigma_{\text{ind}} \) upon \( \dot{\varepsilon}_{\text{ind}} \) was analyzed for the data obtained from small indentation depths, \( h=500-800 \text{ nm} \), to assess the operative mechanism of time-dependent deformation associated with the GNDs. It was observed that for the high stacking fault energy (SFE) 5052 aluminum alloy and pure copper, the thermal activation energy \( \Delta G_{\text{thermal}} \) of the deformation rate followed essentially the same dependence upon \( \sigma_{\text{ind}} \) regardless of \( \dot{\varepsilon}_{\text{ind}} \), however in the 70/30 brass test material, which possess a lower value of SFE, the \( \Delta G_{\text{thermal}} \) showed a dependence upon \( \sigma_{\text{ind}} \) that was highly strain rate dependent. In the case of the high SFE material, the apparent activation volume, \( V^* \) of the deformation process was found to decrease with increasing \( \rho_{\text{GNDs}} \) in a way indicative of deformation occurring by a process that is listed by dislocation/dislocation

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interactions. Our data indicate that in fcc materials of low SFE the deformation during
nanoindentation occur by a more complex mechanism than simple time dependent
dislocation glide limited by dislocation-obstacle interaction. The operative deformation
mechanism most likely involves micro-twinning.

4.3.1. Introduction
Micro/nano-indentation testing techniques offers the ability to study rate-sensitive local
deformation mechanisms of microscopic volumes of material. It is well-known, that the
micro indentation hardness of materials displays strong indentation depth dependence
[61, 69-72]. The physical interpretation of this indentation depth effect has been
attributed to the evolution of the so-called “geometrically necessary” dislocations
(GNDs) which are introduced in the material in order to provide the necessary lattice
rotation to accommodate the shape of the indenter [51, 69, 73-75]. This gives rise to an
extra dislocation density in the indented material compared to the same material that is
uniformly strained.

Plastic deformation that is applied uniformly to a “large” volume of material is
accommodated by the motion of “statistically stored” dislocations (SSDs) upon preferred
crystallographic slip systems.

It is possible that GNDs and SSDs have fundamental differences in their crystallographic
orientation and hence their mobility. GNDs may be more sessile than SSDs and this may
affect the work hardening and the strain rate sensitivity of micro-indented material [69].
The smaller the indentation depth, the larger the density of GNDs relative to SSDs.

Nix and Gao [2] expressed the density of GNDs and SSDs as the functions of the
indentation depth \( h_c \), and the large–depth indentation stress, \( \sigma_0 \) as:

\[
\rho_{\text{GND}} = f\left( \frac{1}{h} \right) = \frac{3\tan^2 \theta}{2bh_c}
\]

\[
\rho_{\text{SSD}} = f(\varepsilon_p) = \frac{1}{27} \left( \frac{\sigma_0}{\alpha \mu b} \right)^2
\]
where $\theta$ is the angle of the flank of a pyramidal indenter relative to the indented surface (for a Berkovich indenter $\theta = 27.9^\circ$), $\mu$ is the elastic shear modulus, and $\varepsilon_p$ is the equivalent plastic strain.

Voyiadjis and Faghihi [4] applied these equations to express the indentation stress $\sigma_{ind}$ at a small indentation depth in terms of $\rho_{GND}/\rho_{SSD}$ and $\sigma_0$ as:

$$\left(\frac{\sigma_{ind}}{\sigma_0}\right)^2 = 1 + \frac{\rho_{GND}}{\rho_{SSD}}$$

4.3.3

Since GNDs are “forced” to nucleate in the very small region directly below the indentation, they may be oriented along non-easy slip crystal systems. Thus, their Burgers vector and mobility may be fundamentally different than SSDs. These differences may affect the way plastic deformation can occur.

In this study ambient temperature nano-/micro-scale indentation tests were performed on three common fcc materials at various levels of indentation loading rate to assess the dependence of the GNDs upon indentation depth and loading (strain) rate. From these data, one can characterize the thermal activation energy $\Delta G_{thermal}(\sigma_{ind})$ of GNDs as a function of indentation stress and compare it to equivalent values that correspond to the motion of SSDs. It can then be compared the data obtained from the three types of fcc metals to assess the effect of stacking fault energy on the $\Delta G_{thermal}(\sigma_{ind})$ of GNDs.

4.3.2. Experimental procedure

The three fcc alloys studied are 5052 aluminum, pure copper, and 70/30 brass in the annealed conditions. These alloys possess different stacking fault energies, strain hardening rates, and tendency for twining. Table 4.3.1 shows the characteristics of these alloys. Their microstructures contain large equi-axed. Annealing twins are clearly visible in 70/30 brass microstructure.

Room-temperature micro-indentation tests were performed, with a diamond Berkovich indenter, using a NanoTest indentation testing platform made by Micro Materials Ltd. (Wrexham, UK). The instrument was positioned on an anti-vibration base and was enclosed in a temperature-controlled cabinet.
Indentation tests were performed to a maximum indentation load of 2000 mN at four loading rates: 1, 10, 100, and 1000 mN/sec. Between three to five microindentation tests were performed on each material, at each of the four indentation load rates. Forty eight microindentation tests were performed. During each test the indentation depth, \( h \), was corrected for both thermal drift and elastic compliance of the indenter.

**Table 4.3.1: The characteristics of the three fcc metals studied in this research**

<table>
<thead>
<tr>
<th>Test material</th>
<th>Composition</th>
<th>( SFE ) (mJ/m(^2))</th>
<th>Grain size (( \mu )m)</th>
<th>( b ) (nm) [96]</th>
<th>( \mu ) (GPa) [96]</th>
</tr>
</thead>
<tbody>
<tr>
<td>70/30 brass</td>
<td>70% Cu, 30% Zn</td>
<td>7(-)14 [76-79]</td>
<td>75 [86]</td>
<td>0.256</td>
<td>37</td>
</tr>
<tr>
<td>Pure copper</td>
<td>99.9% Cu</td>
<td>55(-)78 [80-83]</td>
<td>25 [86]</td>
<td>0.256</td>
<td>42.1</td>
</tr>
<tr>
<td>5052 aluminum</td>
<td>2.2% Mg, 0.40% Fe, 0.25% Si, 0.17% Cr, 0.1% Mn, 0.1% Cu, 0.1% Zn</td>
<td>145(-)200 [84, 85]</td>
<td>52 [87]</td>
<td>0.286</td>
<td>25.4</td>
</tr>
</tbody>
</table>

**4.3.3. Results**

**4.3.3.1. Indentation load–depth (\( P\)-\( h \)) plots**

Figure 4.3.1 shows typical plots of the indentation force, \( P \), versus the indentation depth, \( h \), for one test performed on each of the metal alloys at each of the indentation loading rates.

An interesting phenomenon which is observed in the \( P\)-\( h \) curves is the steps in the \( P\)-\( h \) curve of the 5052 aluminum alloy. This behavior is analogous to serrated flow observed in tensile tests performed on the same alloy and results from solute-dislocation interactions. As the loading rate is increased the nature of serrated flow changes from ideal stair–step–like at the lowest rates (i.e. 1 mN/sec, Fig. 4.3.1a), to quite smooth parabolic curves at the highest rates (i.e. 1000 mN/sec, Fig. 4.3.1d).

The low loading rate indentation tests, performed on the 5052 aluminum alloy, clearly show the well known Portevin–LeChatelier (PLC) effect, resulting from the interaction between Mg solutes and mobile dislocations [88]. The PLC instabilities arise only if the waiting times of dislocations at the obstacles are in the order of the diffusion times of
solutes towards dislocations. This is why the PLC effect is observed in the $P-h$ curves from the low but not the high loading rates.

4.3.3.2. Indentation stress, $\sigma_{ind}$, versus indentation depth, $h$

The indentation contact area $A$ is related to the indentation contact depth $h_c$ as:

$$A = \pi (\tan^2 \alpha)(h_c + \Delta h)$$

where $\alpha$ is the half angle of the Berkovich indenter ($\alpha = 70.3^\circ$), $h_c$ is contact depth, and $\Delta h = R(1 - \sin \alpha) = 0.06R$ is the minimum indentation depth for self-similar deformation [91-94]. In practice, the Berkovich tip is not a perfect pyramidal shape; in our Berkovich indenter, we measured the indenter tip radius $R = 500$ nm. In the present research, however, the smallest studied depth was 500 nm which is therefore much bigger than $\Delta h$ so that all the indentation tests satisfy the self–similar requirement [94]. $\sigma_{ind}$ then can be written as:

$$\sigma_{ind} = \frac{P}{cA} = \frac{P}{24.5c(h_c + 0.06R)}$$

In this equation, the constant $c$ represents the effect of metal pile-up or sink-in around the indentation chosen to be 0.9 for the pure copper and the 70/30 brass materials [95], and 1.2 for 5052 aluminum alloy [97].

The dependence of $\sigma_{ind}$ upon $h$ for the lowest and the highest load rates is shown for each of the three metals tested in Fig. 4.3.2. $\sigma_{ind}$ increases significantly when $h$ is less than about 4 $\mu$m.
a

\begin{align*}
P (\text{mN}) & \quad h (\text{\mu m}) \\
\text{Brass} & \\
\text{Cu} & \\
\text{Al 5052} &
\end{align*}

b

\begin{align*}
P (\text{mN}) & \quad h (\text{\mu m}) \\
\text{Brass} & \\
\text{Cu} & \\
\text{Al 5052} &
\end{align*}
Figure 4.3.1: Indentation load versus indentation depth for all tested materials, (a): 1 mN/sec, (b): 10 mN/sec, (c): 100 mN/sec, and (d): 1000 mN/sec.
Figure 4.3.2: Indentation stress versus indentation depth for all tested materials at (a): the lowest load rate; 1 mN/sec, and (b): the highest load rate; 1000 mN/sec.
It was observed that for all the materials tested $\sigma_{\text{ind}}$ increases, for any given indentation depth, when the loading (strain) rate increases. This is shown for the 70/30 material in Fig. 4.3.3.

![Figure 4.3.3: Indentation stress versus indentation depth at different loading rate in the 70/30 brass material. For a given indentation depth, the higher the loading rate, the larger the indentation stress.](image)

4.3.3.3. Indentation strain rate, $\dot{\varepsilon}_{\text{ind}}$, versus indentation depth, $h$

The average plastic strain rate during indentation, $\dot{\varepsilon}_{\text{ind}}$, is correlated to the indentation displacement, $h$, and the indentation displacement rate $\dot{h}$ as:

$$\dot{\varepsilon}_{\text{ind}} = k \frac{\dot{h}}{h}$$  \hspace{1cm} 4.3.6

where $k$ is a geometrical constant, taken to be equal to 1 [51].

Figure 4.3.4 shows the variation in the average indentation strain rate, $\dot{\varepsilon}_{\text{ind}}$, versus indentation depth, $h$, for pure copper indented at different loading rates. In this study, we
considered only $\dot{\varepsilon}_{\text{ind}}$ data arising from indentations made to depths greater than 500 nm. This was done to avoid the effects of surface oxide layers and the finite indenter tip radius on the calculated $\sigma_{\text{ind}}$ and $\dot{\varepsilon}_{\text{ind}}$.

Figure 4.3.4: Apparent average indentation strain rate, $\dot{\varepsilon}_{\text{ind}}$ versus indentation depth for the pure copper indented at four loading rates. $\dot{\varepsilon}_{\text{ind}}$ decreases with increasing $h$ under constant loading rate condition. The shape of the curves is the same for the 5052 aluminum alloy and the 70/30 brass.

4.3.3.4. Indentation stress, $\sigma_{\text{ind}}$, versus indentation strain rate, $\dot{\varepsilon}_{\text{ind}}$

Figure 4.3.5 shows a logarithmic plot of $\sigma_{\text{ind}}$ versus $\dot{\varepsilon}_{\text{ind}}$ at one specific loading rate, 10 mN/sec, and indicates that the test materials all show a nonlinear logarithmic dependence of $\sigma_{\text{ind}}$ upon $\dot{\varepsilon}_{\text{ind}}$. Although this figure does not show data from indentation tests performed at the other loading rates, the shape of the curves shown is typical of the other indentation tests performed. When $\dot{\varepsilon}_{\text{ind}}$ is small the indentation stress is essentially
independent of $\dot{\varepsilon}_{\text{ind}}$ but becomes increasingly more strain rate dependent when $\dot{\varepsilon}_{\text{ind}}$ is increased beyond 0.01 sec$^{-1}$. The shape of the curves in Figure 4.3.5 is characteristic of a deformation process that is occurring by the mechanism of obstacle-limited dislocation glide. When $\dot{\varepsilon}_{\text{ind}}$ is less than about 0.01 sec$^{-1}$, $\sigma_{\text{ind}}$ is not limited by the frequency of thermally assisted “jumps” of the dislocations over obstacles and thus displays “athermal” behaviour with little dependence upon $\dot{\varepsilon}_{\text{ind}}$. The sharp increase in the $\sigma_{\text{ind}}$, when $\dot{\varepsilon}_{\text{ind}}$ is greater than about 0.01 sec$^{-1}$, reflects that the dislocation motion past obstacles is forced to occur at a rate that requires a combination from both thermal atomic vibration and applied stress with the amount of the necessary applied stress increasing with increasing $\dot{\varepsilon}_{\text{ind}}$.

Figure 4.3.5: Logarithmic plot of $\sigma_{\text{ind}}$ versus $\dot{\varepsilon}_{\text{ind}}$ at loading rate of 10 mN/sec for 70/30 brass, pure copper and 5052 aluminum alloy.
4.3.4. Discussion

In this study, constant load rate indentation has been performed to determine the effect of strain rate on the indentation depth dependence of the indentation stress. Our primary interest is to characterize the obstacles strength that limit the time-dependent glide of GNDs. It can be done by carefully assessing the indentation data obtained from our most shallow indents, where the primary dislocation distribution consists of GNDs as follows:

Using Eq. 4.3.6, one can calculate the \( \rho_{GND} \) as:

\[
\rho_{GND} = \left[ \left( \frac{\sigma_{ind}}{\sigma_0} \right)^2 - 1 \right] \rho_{SSD}
\]  

where \( \rho_{SSD} \) is calculated with Eq. 4.3.2 by setting \( \sigma_0 \) to be equal to \( \sigma_{ind} \) from the largest indentation depths (h= 9000 nm).

These equations were used to calculate \( \rho_{GND} \) from the \( \sigma_{ind} \) versus \( h \) data from the indentation tests performed at the lowest indentation loading rate. It is then assumed that because of the geometrical similarity of the indentations, the dislocation density in the indentation plastic zone for all the indentations made to a given depth in a given material is the same regardless of loading rate.

Figure 4.3.6 shows the \( \rho_{GND} \) versus indentation depth for the tested materials at the lowest loading rate (i.e. 1 mN/sec). It is clear from this figure that for low indentation depths, \( \rho_{GNDs} \gg \rho_{SSDs} \) and for large indentation depths where \( \sigma_{ind} \approx \sigma_0 \), \( \rho_{SSDs} \gg \rho_{GNDs} \).
Figure 4.3.6: \( \rho_{\text{GND}} \) versus \( h \) for the lowest indentation load rate (1 mN/sec) for three fcc materials studied

It is considered that in the three materials studied, the plastic deformation during indentation occurs by a time–dependent obstacle limited dislocation glide process which can be described by the following Arrhenius-type expression [96]:

\[
\dot{\varepsilon}_{\text{ind}} = A \rho_m b \nu
\]  

4.3.8

where \( A \) is a geometrical constant, \( \rho_m \) is the density of mobile dislocations, \( b \) is Burgers vector, and \( \nu \) is the average dislocation velocity.

The velocity \( \nu \) is related to the thermal activation strength function \( \Delta G_{\text{thermal}}(\sigma_{\text{ind}}) \) of the primary obstacles to dislocation glide and can be given as [96]:

\[
\nu = \beta b v \exp \left[ -\frac{\Delta G_{\text{thermal}}(\sigma_{\text{ind}})}{kT} \right]
\]  

4.3.9

where \( \beta \) is a dimensionless constant, \( v \) is the atomic jump frequency, and \( k \) is the Boltzmann constant.
Combining Eqs. 4.3.8 and 4.3.9 gives the following expression for the indentation creep rate as a function of $\Delta G_{\text{thermal}}(\tau_{\text{ind}})$ as:

$$
\dot{\varepsilon}_{\text{ind}} = B\rho_m b^2 v \exp\left[-\frac{\Delta G_{\text{thermal}}(\sigma_{\text{ind}})}{kT}\right]
$$

4.3.10

In this analysis we assume that the total dislocation density in the indentation plastic zone is a function of indentation depth but not a function of loading rate (i.e. strain rate). It is also assumed that the mobility of the GNDs may indeed be quite different than that of the SSDs.

For the case of shallow indentations, $h=500-800$ nm, $\rho_{\text{GNDs}} \gg \rho_{\text{SSDs}}$. In this case, therefore, the $\rho_m \approx \rho_{\text{GNDs}}$ and Eq. 4.3.10 becomes:

$$
\dot{\varepsilon}_{\text{ind}} = B\rho_{\text{GNDs}} b^2 v \exp\left[-\frac{\Delta G_{\text{thermal,GNDs}}(\sigma_{\text{ind}})}{kT}\right]
$$

4.3.11

With this information we can calculate values for $\Delta G_{\text{thermal,GNDs}}$ that result in Eq. 4.3.11 producing the measured $\dot{\varepsilon}_{\text{ind}}$ for indentations made at the various loading rates.

Figure 4.3.7 shows $\Delta G_{\text{thermal,GNDs}}$ versus $\sigma_{\text{ind}}$ for the three fcc alloys tested. Comparing these plots one can see that the $\Delta G_{\text{thermal,GNDs}}$ versus $\sigma_{\text{ind}}$ data are more aligned along a uniform trend, insensitive to loading rate, for the 5052 aluminum and the pure copper compared to the 70/30 brass material. This indicates that the 5052 aluminum and the pure copper undergo the deformation by a single mechanism that displays a stress dependence characteristic of a dislocation glide process limited by discrete obstacles which can be well predicted by an equation of the form of Eq. 4.3.11.

The fact that the 70/30 brass $\Delta G_{\text{thermal,GNDs}}$ data do not fall on a single trend when plotted again $\sigma_{\text{ind}}$ indicates that its deformation occurs by a more complex process. It is suggested that this difference in deformation mechanism is related to the lower SFE of the 70/30 brass.
Figure 4.3.7: $\Delta G_{\text{Thermal,GNDs}}$ calculated from Eqs. 4.3.11 versus indentation stress, $\sigma_{\text{ind}}$, for the a) 5052 aluminum, b) pure copper, and c) 70/30 brass.

Figure 4.3.8 shows activation energy of the deformation rate controlling obstacles, $\Delta G_{0,\text{GNDs}}$ versus SFE values, reported in the literature for the 5052 aluminum, 70/30 brass and pure copper materials. $\Delta G_{0,\text{GNDs}}$ was determined by extrapolating the $\Delta G_{\text{Thermal,GNDs}}$ versus $\sigma_{\text{ind}}$ trends in Fig. 4.3.7 to $\sigma_{\text{ind}} \approx 0$. $\Delta G_{0,\text{GNDs}}$ has values between 0.5 and 0.65 eV which is very similar in magnitude to $\Delta G_{0,\text{SSDs}}$ measured for pure copper [98]. Thus our data indicate that in the case of pure copper and 5052 aluminum, the kinetics of time-dependent deformation of GNDs is very similar to the conventional SSDs. Figure 4.3.8 shows that $\Delta G_{0,\text{GNDs}}$ increases with increasing SFE and, when the SFE is less than about 50 mJ.m$^{-2}$, the scatter in our data become so large that we conclude that Eq. 4.3.11 no longer predicts the time-dependent indentation creep process. Since we know that the low SFE promotes considerable twinning deformation in 70/30 brass thus we suggest that micro-twinning controls the deformation during nano-indentation of the 70/30 brass at
depths in the range of 500-800 nm. The mechanism of deformation by geometrically necessary twins has been studied previously [99].

Figure 4.3.8: $\Delta G_{0,\text{GNDs}}$ versus SFE, reported in the literature, for the shallowest (h= 500 to 800 nm) at different loading rates.

The apparent activation volume, $V^*$, is a kinetic characteristic of the rate-controlled deformation processes. When the deformation occurs by dislocation glide limited by the interaction of the dislocations with discrete obstacles, we can express $V^*$ as a function of $\Delta G_{\text{Thermal}}$, $\Delta G_0$, and $\sigma_{\text{ind}}$.

$$\Delta G_0 = \Delta G_{\text{Thermal}} + \sigma_{\text{ind}} V^*$$

Equation 4.3.12 was used to evaluate $V^*$ for the indentation data from the 5052 aluminum and the pure copper samples tested over the depths range h=500-800 nm where TTT. Figure 4.3.9 shows $V^*$ versus $\sigma_{\text{ind}}$ for these samples. It is clear from these graphs that $V^*$
decreases with increasing stress suggesting that the inter-obstacle spacing of obstacles to the glide of GNDs is decreasing when the stress, and hence the \( \rho_{\text{GNDs}} \), is increased. This is further illustrated in Figure 4.3.10 where \( V^* \) (Eq. 4.3.12) is plotted versus \( \rho_{\text{GNDs}} \). This plot indicates that \( V^* \) decreases with increasing \( \rho_{\text{GNDs}} \) and the dependence upon \( \rho_{\text{GNDs}} \) is the same for the 5052 aluminum and the pure copper material. The fact that \( V^* \) of both materials follows the same functional dependence upon \( \rho_{\text{GNDs}} \) indicates that both materials undergo a similar mechanism of deformation; namely, time-dependent dislocation glide that is limited by dislocation-dislocation interactions.

![Figure 4.3.9](image)

Figure 4.3.9: Normalized activation volume, \( V^*/b^3 \), versus average indentation stress, \( \sigma_{\text{ind}} \) for tests performed between \( h=500-800 \) at the lowest loading rate (1 mN/sec).
4.3.5. Summary

In this study microindentation tests were performed at various loading rates to indicate the operative deformation mechanisms at shallow indentation of depth between 500 to 800 nm. In this depth range, geometrically necessary dislocations represent the large majority of the mobile dislocation density. Studies were performed on three fcc materials; 70/30 brass, pure copper and 5052 aluminum alloy with very different stacking fault energies.

The materials with relatively high SFE displayed a relationship between $\sigma_{ind}$ and $\dot{\varepsilon}_{ind}$ indicative of deformation occurring a mechanism of time-dependent obstacle limited glide where the estimated total activation energy $\Delta G_0$ of the rate limiting obstacles was very close (0.5-0.65 eV) to that measured from large specimen uniaxial testing techniques.

It can therefore be concluded that the mobility of GNDs is very similar to that of the conventional statistically stored dislocations (SSDs) for these high SFE fcc materials. Studies of the apparent activation volume $V^*$ of these materials indicated that $V^*$ decrease
with increasing $\rho_{\text{GND}}$ in a way consistent with a dislocation glide process that is limited by dislocation/dislocation interaction.

The low SFE 70/30 brass material did not necessarily displayed a $\sigma_{\text{ind}} - \dot{\varepsilon}_{\text{ind}}$ response characteristic of a time-dependent obstacle limited dislocation glide deformation process. This is attributed to deformation occurring by a more complex mechanism probably involving micro-twinning made possible by the material’s low SFE.

4.3.6. References


Chapter 5
Discussions

This chapter discusses observations and provide further interpretation to the results obtained from the analysis of data from SMFF experiments performed on the bcc (1020 steel) and the fcc (6061 and 5052 aluminum, pure copper, and 70/30 brass) work pieces during the course of this study. These interpretation are required at by considering the fundamental data that were obtained from the uniaxial tensile tests (described in this chapter) and from micro-indentation tests reported in chapter 4. The overall objective of this assessment is to ultimately lead us to predict:

- The effect of microstructure and associated deformation mechanisms (i.e. dislocation slip and deformation twinning) on the flow formability of a metal.
- The effect of microstructure and mechanical parameters (i.e. yield stress ($\sigma_y$), rate of work hardening ($\dot{\theta}$), stacking fault energy (SFE), and grain shape) on the local equivalent plastic strain within a SMFF’d parts.
- The effect of strain rate and size effect on the slip/twin deformation mechanisms of the metal alloys which were used in the SMFF experiments.

The results from SMFF operations on 1020 steel provided a basis for using the microindentation technique to predict the invoked equivalent plastic strain $\varepsilon_p$, in a SMFF’d work piece. The resulting plots of the von-Misses equivalent plastic strain, $\varepsilon_p$, versus distance, $X$, from the work piece/mandrel surface clearly showed that maximum $\varepsilon_p$ occurs near the mandrel/work piece interface directly ahead of the nose of the internal ribs. $\varepsilon_{p,\text{Max}}$ reaches a value of about 160%-170% in the SMFF’d 1020 steel. It was found that thickness reductions greater than 51% resulted in the 1020 steel work piece tearing on a circumferential plane directly ahead of the longitudinal ribs. However, $\varepsilon_p$ becomes smaller near the roller/work piece interface and drops to the minimum in the mid-thickness. The $\varepsilon_p$ imparted to the work piece during SMFF is significantly higher than that imparted during smooth-mandrel flow forming. This is an important findings since it
indicates that the failure of SMFF’d work piece occurs close to the mandrel side compared with work pieces made by smooth mandrel flow forming which fail at the roller side. \( \varepsilon_p \) reaches approximately 1.7 close to the work piece/mandrel interface directly in front of the nose of the internal ribs and this, therefore, represents the forming limit for 1020 steel when subjected to the flow forming parameters used in this study.

The analysis of grain shape in the SMFF’d 1020 steel work piece showed that the ferrite grains are axially elongated (in the direction of rollers’ travel). The maximum stretch in the grains was observed in the region that endured the largest plastic strain \( \varepsilon_p \). The ferrite grains of the SMFF’d work piece experienced considerably less circumferential (hoop) strain. This was confirmed by metallographic analysis and uniaxial stress-strain responses of the SMFF’d work piece loaded in the circumferential and axial directions (Fig. 5.1). The tensile engineering stress-strain plots of the SMFF’d 1020 steel showed limited uniform elongation. This suggests that cross-slip or climb of dislocations was limited due to high dislocation density in the samples caused by severe plastic deformation during the SMFF process. Also, the yield stress (\( YS \)) and the ultimate tensile strength (\( UTS \)) is larger in the axial compared with the circumferential direction of SMFF’d work pieces. This is consistent with the observed direction of maximum elongated ferrite grains.
To study the deformation micro-mechanisms and the effect of strain hardening rates on the plastic strain distribution, SMFF operations were performed on two fcc aluminum alloys (6061 and 5052). Provided that the work piece material possesses a sufficiently high strain-hardening rate, large plastic strains \((i.e. \varepsilon = 1.7)\) can be imposed to the work piece without undergoing localized failure.

The strain hardening rate, \(\theta\), is calculated from the uniaxial tensile stress-strain curves. In the present study, normalized strain hardening rate \((\theta/G)\) was plotted against the strain, \(\varepsilon\) (Fig. 5.2).

The normalized strain hardening rate of the 5052 aluminum alloy is slightly larger than that of the 6061 aluminum alloy. The 5052 is an alloy in the Al–Mg alloy family with the Mg atoms dissolved in solid solution and, thus, it possesses a high hardening rate compared to other aluminum alloys. It is well established that the interaction between

Figure 5.1: The uniaxial tensile engineering \(\sigma - \varepsilon\) curves for as received and SMFF’d (in axial and circumferential directions) for 1020 steel.
dislocations and Mg solute atoms in the 5052 aluminum alloy leads to solid solution strengthening by reducing the dislocation mobility [1, 2].

Higher strain hardening rate causes the equivalent plastic strain, \( \varepsilon_p \), in the area directly ahead of the internal rib to be increased. The maximum \( \varepsilon_p \) is in the range of 1.2–1.4 for the 5052 aluminum alloy while it is in the range of 1–1.2 for the 6061 alloy. For a given SMFF condition, my tests indicate that both the maximum local equivalent plastic strain and the local plastic strain gradient increase with increasing strain hardening coefficient of the aluminum work piece material.

![Normalized work hardening rate (\( \frac{\theta}{G} \)) versus strain for 5052 and 6061 aluminum alloys.](image)

**Figure 5.2:** Normalized work hardening rate (\( \frac{\theta}{G} \)) versus strain for 5052 and 6061 aluminum alloys. The presence of Mg solute atoms may lead to the appearance of plastic instabilities resulting in the well-known serrated yielding or Portevin-Le Chatelier (PLC) effect which marks itself as steps in the \( \frac{\theta}{G} - \varepsilon \) curve at strains bigger than 2%.

The dimensions of micro-indentations, used to determine the local equivalent plastic strain \( \varepsilon_p \), are of depth and spacing of 7 \( \mu \)m and 100 \( \mu \)m, respectively. Since, the size of
the indentations is less than that of the grain size of the tested materials, the plastic
deformation during indentation occurs locally within only one or two grains. This clearly
indicates that variations in the measured the local equivalent plastic strain, $\varepsilon_p$, may be
due to grain–to–grain variation in the plastic flow. Also, within the small sub–micrometer
depth range, the microhardness values are strongly affected by the actual indentation
depth (see Sections 3.2 & 3.3).

Materials with different stacking fault energies (SFE), and therefore different *slip/twin*
ratios, respond differently to plastic deformation and therefore also to flow forming. To
study the effect of SFE, four fcc metals/alloys (pure copper, 70/30 brass, 5052 aluminum
and 6061 aluminum) were chosen to be flow formed (Table 3.3.1).

In the high SFE tested alloys, dislocation cross slip is the dominant mode of plastic
deformation. In the low-SFE tested materials (*i.e.* copper alloys) *twinning* is the main
deformation mechanism. A lower SFE usually leads to a higher strain hardening rate.

Slipping and twinning are, indeed, competitive phenomena. In the tested 70/30 brass,
this competition marks in uniaxial tensile engineering $\sigma-\varepsilon$ response as serration in the
flow curve (Fig. 5.3). The main reason of such serrated behaviour is that twin bands
effectively impede dislocations’ motion, causing substantial stress accumulations. As a
result, a serration comprising increase–and–release of stress appears in the stress–strain
curve of low SFE 70/30 brass material.
Most aspects of mechanical twinning seem to be beneficial for a high strain hardening capability. The suppression of cross slip due to the stacking fault would enhance the confinement of dislocation pile-ups to the easy glide slip planes and thus increases the strain hardening rate due to the long range back stresses from pile-ups. The long range back stress would encourage initiation of other bands in the same grain or adjust grains, which results in higher ductility.

Normalized strain hardening rate, $\theta/G$, versus strain for pure copper and 70/30 brass is shown in Fig. 5.4. This figure clearly indicates four distinct regimes (A-D) of strain hardening in the low stacking fault 70/30 brass. These regimes are similar to those reported previously from uniaxial tensile tests performed on low SFE materials [3-6]. Stage A is an initial sharp decrease in the work-hardening rate. This region is very similar in form to the strain hardening behaviour exhibited by the higher SFE polycrystalline fcc metals in the stage III work hardening region [7]. A constant work-hardening rate, Stage...
Stage $B$, region follows the Stage $A$. The onset of Stage $B$ is correlated with the initiation of deformation twinning. The normalized strain hardening rate, $\Delta \sigma / G$, is about 0.02 to 0.03 for the 70/30 brass during Stage $B$. This is in good agreement with El-Danaf et al. [3, 4] findings on the work hardening behaviour of 70/30 brass. Stage $C$ is characterized by a renewed decrease in the work-hardening rate. During this stage, the rate of primary twinning decreases. Finally, Stage $D$ indicates a region of constant work hardening rate, where extensive twin intersection, brought about by the activation of secondary twinning systems, occurs. The normalized strain hardening rate is about 0.01 in Stage $D$. Note that the strain hardening rate of the 70/30 brass during Stage $D$ is significantly larger than that exhibited by the high-SFE 6061 and 5052 aluminum alloys (Fig. 5.2).

In the SMFF’d 70/30 brass microstructure, more grain-to-grain variability in the dislocation slip step and mechanical twin densities were observed (Fig. 3.3.5). In the low SFE 70/30 brass, due to difficulty in cross slip, certain grain orientations are unable to undergo the required plastic deformation; therefore, the neighboring grains are required to deform more to accommodate the large displacements invoked during the SMFF process. This ultimately causes the high maximum local $\epsilon_p$ and high grain-to-grain variability in $\epsilon_p$. 
Figure 5.4: Normalized work hardening rate ($\theta/G$) versus strain for 70/30 brass and pure copper. Four distinct regions are seen in the data from the 70/30 brass material.
During a complicated forming process like SMFF, the applied strain rate, the existence of very high plastic strain gradients occurring over very small volume of work piece materials affect the amount that the material can be formed. These are determining parameters to assess the effect of microstructure and associated deformation mechanisms. The results of the present research show that within a distance of several micrometers from the work piece/mandrel interface, the equivalent plastic strain changes significantly. Furthermore, the strain rate applied during SMFF operation is also highly variable.

The fact that the SMFF’d samples displayed such high strain gradients over a distance of only several micrometers in the highest strained regions of the work piece and the fact that these high plastic strains were applied at very high rates makes the localized plastic deformation invoked during SMFF very similar to that invoked during the micro/nano-indentation tests presented in Chapter 4 of this thesis.

The results from the microindentation tests on the 6061 aluminum alloys, in three tempering conditions and over a range of indentation loading rates, showed that strain rate sensitivity of the indentation stress increases with decreasing indentation depth. The results from these tests were important as they clearly show that apparent activation energy of the obstacles to dislocation glide increases with decreasing indentation depth and are also dependent upon the heat treatment condition of the 6061 aluminum tested material. These findings are in agreement with the shape of $\varepsilon_p - X$ profiles (Fig. 3.2.6b–3.2.8b). By getting closer to the work piece/mandrel interface (i.e. $X \rightarrow 0$), strain rate sensitivity, $m$, increases.
Studies on the assessment of the depth dependence of the indentation stress during constant strain rate nanoindentation of 70/30 (annealed and 80% cold worked) brass have suggested that pre-existing plastic strain, significantly, affects the strain rate sensitivity and the operative dislocation-obstacle mechanisms. Such an investigation can lead one to answer whether the controlled introduction of twins in a polycrystalline fcc metal (i.e. 70/30 brass) lead to markedly enhanced rate-sensitivity of plastic flow? If so, what are the mechanistic contributions to such rate-sensitivity of deformation in twinned metals?

I has been observed that the strain rate sensitivity parameter, \( m \), is higher for the cold worked compared to the annealed brass samples which indicates that cold-working affects the underlying process of obstacle-limited thermally-activated dislocation glide process. The increased values of \( m \) and the strongly lowered values for activation volume \( V^* \) during nanoindentation testing of 80% cold worked 70/30 brass suggested another rate-controlling deformation mechanism may be occurring different from that known for fcc coarse grained materials without twins.

A key deformation mechanism for coarse grained fcc metals is the pinning of dislocations by small obstacles such as forest dislocations. For cold worked 70/30 brass, the main obstacle to dislocation motion is the high concentration of mechanical twins. That is, in addition to the classical deformation by dislocation slip limited by dislocation/dislocation interactions, the interactions between slipping dislocations and twin boundaries play an important role in the plastic deformation process. It has been suggested that the dislocation interaction with mechanical twin bands would take up most of the deformation and that the mechanical twin bands associated deformation mechanisms are likely to be much more rate-sensitive [8]. This is consistent with my experimentally observed trends of the effect of twin density on the strength and rate sensitivity of plastic flow, (i.e., the stress and the strain rate sensitivity of the cold worked 70/30 brass specimen with respect to the concentration of the mechanical twin bands). However, a relatively small \( m \) is observed in the annealed 70/30 brass where the concentration of mechanical twins is reduced.

The nanoindentation testing results from the 70/30 brass materials showed that the activation strength \( \Delta G_0 \) of the deformation rate controlling obstacles depends upon both
indentation depth and prior plastic deformation. $\Delta G_{\text{thermal}}$ and $\Delta G_0$ data obtained from the CSR nano-indentation tests performed, on 70/30 brass in the annealed and the cold worked conditions, in this study, were used to calculate the apparent activation volume, $V^*$, and apparent activation area, $\Delta a$, that characterize the deformation process at the various level of indentation depth from 0.2 to 2.0 $\mu$m. Linear tends of $b^2/\Delta a$ versus $\tau_{\text{ind}}$ indicated that the plastic deformation occurs by thermally activated time–dependent obstacle–limited dislocation glide process.

The slope of $b^2/\Delta a - \tau_{\text{ind}}$ plots changed with material conditions (annealed and cold worked 70/30 brass samples) and with indentation depths. This indicates that the strength (or the type) of obstacles that limit dislocation glide is different.

Studies on the assessment of the dependence of the geometrically necessary dislocation upon depth and strain rate on the polycrystalline fcc metals/alloys have suggested that GNDs density displayed the characteristic decrease with increasing $h$. Apparently, for the small indentation depths, GNDs are main contributing dislocations in order to accommodate for the lattice rotation imposed by indenter geometry. GNDs are, therefore, dependent upon $1/h$ and the indenter geometry. However, for large indentation depths (macroscopic regime), statistically stored dislocations (SSD) are responsible for plastic deformation. SSDs which are a function of plastic strain in the material are independent of $h$. Considering the changes in $\Delta G_{\text{thermal}}$ for the GNDs versus indentations stress $\sigma_{\text{ind}}$ and type of materials (SFE), it was found that in high SFE materials deformation occurs by a mechanism of time-dependent obstacle limited glide. However, in low SFE material (i.e. 70/30 brass), more complex mechanisms like micro-twinning and geometrically necessary twins (GNTs) may be contributing as well. Also, with increasing the SFE, $\Delta G_{\text{thermal,GNDs}}$ decreases. Dynamic recovery processes (i.e. dislocations’ cross slip and climb), as the main characteristics in high SFE materials are considered as the responsible factor in such behaviour.
The findings obtained from micro/nano-indentation tests presented in Chapter 4 explain/predict the plastic strain profile through a flow formed part, particularly in the high strain regions near the work piece/mandrel interface in the SMFF process. From examination of average strains and strain gradients as a function of indentation depth, it is clear that classical views of gradient plasticity models do not apply at very shallow depths.

Micro/nano indentation results in Chapter 4 show that $\sigma_{ind}$ (in all tested materials) is clearly depth dependent. This dependency results from an increase in the local dislocation density (GNDs) around shallow, compared to deep, indentations. Indentation results (Fig. 3.3.11) and metallographic pictures (Figs. 3.3.5 and 4.2.12) show that micro-twins, GNDs, and GNTs are the main contributing parameters responsible for higher local plastic strain, during SMFF, in low SFE brass compared with high SFE aluminum alloys.

Micro/nano-indentation testing is, therefore, a consistent and systematic technique to explain the micromechanical behaviour and mechanisms (i.e. in terms of size effect and GNDs contributions, dislocations’ motion and interaction, activation energy, activation volume, strength and type of dislocations limited obstacles) occurring during complex metal forming operations such as the SMFF process.
References


Chapter 6  
Concluding remarks and suggested future works

6.1. Conclusion

This chapter summarises the key conclusions that were arrived at as a result of my research. The main objective of my research was to assess the equivalent plastic strain in various common metals/alloys when subjected to an SMFF operation and then to analyze the effect of microstructure and mechanical parameters (i.e. $\sigma_y$, $\theta$, SFE) on the local equivalent plastic strain within the SMFF’d parts. The following conclusions can be drawn from the SMFF operations and micro/nano-indentation testing:

1. For the bcc and fcc materials, subjected to SMFF operations, the measured equivalent plastic strain, $\varepsilon_p$, was highest on the work piece/mandrel interface in the thin wall region directly in front of the internal ribs. However, this is not the case in smooth-mandrel flow forming process where the maximum equivalent plastic strain occurs at the work piece/roller interface. This difference reflects the increased (very severe) local plastic strain necessary for the work piece to fill the recessed splines of the mandrel.

2. Grains in the microstructure of the SMFF’d work pieces (i.e. ferrite grains in 1020 steel, $\alpha$-Al grains in 5052 and 6061 aluminum alloys, $\alpha$-brass grains in 70/30 grains and Cu grains in pure copper) was observed to be largely elongated in the regions that endured the highest plastic strain at the work piece/mandrel interface near the nose of the internal ribs and along the leading and trailing edges of the rib. This is consistent with maximum equivalent plastic strain being measured in the same region.

3. In the fcc metals/alloys subjected to SMFF operations, the increase in the yield stress in the axial direction after the SMFF operations (after an identical thickness reduction of 60%) is 80%, 74%, 69%, and 58% for the 70/30 brass, pure copper, 5052 aluminum, and 6061 aluminum, respectively. This is in an order that is consistent with the strain hardening rate $\theta$, and the strain hardening exponent, $n$, of the tested fcc materials. The maximum local equivalent plastic strain, the maximum plastic strain gradient, and the maximum grain-to-grain variability are higher in the 70/30 brass than in the pure copper
and the aluminum alloys. This indicates clearly the role of the strain hardening rate of fcc work pieces on the degree of grain-to-grain variability of the local plastic strain after an SMFF process.

4. By assuming a thermally activated dislocation obstacle-limited dislocation glide process, the apparent activation energy of the obstacles and the strain rate sensitivity of indentation stress was found to increase with decreasing indentation depth during constant loading rate microindentation testing on the aluminum and copper alloys (at depth range of 0.5 to 9 μm). This confirms fundamental changes in type and strength of the dislocation motion limited-obstacles of tested fcc metals/alloys with depth.

5. The results from constant strain rate (CSR) nanoindentation tests performed on the annealed and cold-worked 70/30 brass, at the depth range of 0.2 to 2 μm, showed that the thermal activation energy and the strain rate sensitivity are clearly dependent upon indentation depth and pre-exist dislocation density (i.e. cold work). The metallographic microstructures indicated that the cold-work operation on the 70/30 brass has resulted in the extensive dislocations slip and mechanical twinnings. It is, therefore, concluded that the calculated thermal activation energy and thermal activation strength of the obstacles in the cold worked brass reflect the motion and interaction of a dislocation population consisting of widely spaced partial dislocations (twins).

6. In high SFE materials (i.e. 5052 and 6061 aluminum alloys) deformation occurs by a mechanism of time-dependent obstacle limited glide. In low SFE material (i.e. 70/30 brass), more complex mechanisms like micro-twinning and geometrically necessary twins (GNTs) may be contributing as well.
6.2. Suggestion for future work

The findings of the investigations reported in this thesis open several research areas for future consideration. Some of these are described below.

1. FE simulation of cracking failures in spinning

Up to now, there is no research being conducted on the numerical simulation of cracking failures in the SMFF operations, although cracking failures significantly affect the production efficiency and product quality of such processes. It is believed that applying Finite Element (FE) analysis methods to predict first the local stress and plastic strain distributions within flow formed parts and then predicting the onset of cracking in the work piece and the tooling (forming rollers and mandrels) will be very beneficial to the metal forming industry. Of course the measured plastic strain profiles presented in this thesis, and in the reported research of Roy et al. [13], can be used as benchmarks against which to evaluate the FE predictions. Developing a suitable material damage model, i.e. defining damage initiation criteria and damage evolution mechanisms, may be one of the key aspects in the FE simulation of cracking failures in the SMFF process.

2. Experimental design and optimisation

To further analyse the effects of process parameters and their interactions on the quality of the SMFF’d parts, future experimental design of flow forming should attempt to take more input factors at various levels into account (i.e. tooling parameters, work piece parameters, machine parameters). This work has begun [Brandon Vriens, MESc Thesis, Western University, 2012] but my research has shown the effect of microstructural parameters on the underlying plastic strain distribution during SMFF and these microstructural parameters could be considered as variables in future optimization efforts.

3. Hot deformation (SMFF and CSR micro/nano-indentation)

It would be of great interest to perform SMFF at high temperatures in order to be able to perform the SMFF process on alloys that are too brittle to be deformed at ambient temperatures but are important materials for the aerospace and transportation industries (i.e. 7075 aluminum alloy, 6061-T6 aluminum alloy, wrought Mg alloys). New high-
temperature micro-indentation testing equipment could be applied to perform this investigation by following essentially the same experimental technique used in this thesis; namely, micro-indentation to measure local plastic strain distributions through actual SMFF parts followed by specialized micro-indentation tests to assess the fundamental mechanical properties, and their association with the microstructure, of the work piece material.
Curriculum Vitae
Meysam Haghshenas

Education

- Ph.D. (Mechanical and Materials Engineering); Western University (UWO), Canada (2009-2012).
  Dissertation: “Micro-mechanical assessment of the local plastic strain invoked during a splined mandrel flow forming operation”.
- M.E.Sc. (Metallurgical and Materials Engineering); University of Tehran, Iran.
- B.Sc. (Industrial Metallurgy); Iran University of Science and Technology, Iran.

Research Experience and Interests

- Structure–properties relationships in Materials; Materials characterization and mechanical testings.
- Ambient and high-temperature (Thermomechanical) processing of ferrous and non-ferrous alloys (Al alloys, AZ magnesium alloys).
- Nano/Micro-mechanical properties characterization of materials (nano/micro-indentation, and indentation creep)
- Fracture and stress analysis of welded structures.

Work and Teaching Experience

- R & D researcher at TransForm Automotive (GM/Ford part manufacturer), London, ON (2009-2011).
- Product Validation Expert at Vehicle Engineering Department, Automotive Industries Research & Innovation Centre of SAIPA, Iran (2008-2009).
Teaching and Research Assistant at Western University and University of Tehran as a Master and Ph.D. student (2003-2006, and 2009-2012).

Lecturer at Applied & Technical University, IWREC (2007-2009).

Honours and Achievements

- Nominated as top 5 “Driving Ideas to Market” at Ontario province by Ontario Centre of Excellence (OCE) -2009 Competition.
- Awarded UWO-Graduate Teaching Assistants’ Union (GTA) Academic Achievement Scholarship 2010-11.
- Awarded Western Graduate Research Scholarships (WGRS) 2009–2012.
- Awarded 2012/2013 Graduate Thesis Research Award.
- After Graduate Study: Diploma of International Welding Engineer (IWE); Iranian Welding Research & Engineering Centre (IWREC), Representative of IIW (International Institute of Welding).

University involvement & Professional membership

- The member of finance committee of UWO–GTA (2011-2012).
- Conference staff, COM 2011, Montreal, Canada.
- Conference staff, CMSC 2012, London, Canada.
- Canadian Institute of Mining, Metallurgy and Petroleum (CIM).
- Materials Research Society (MRS).

Computer and Software Experience

- Solid works, Microsoft Office, Abacus.
Publications

Peer-Reviewed Journal Papers


Conference Presentations

1- M. Haghshenas, R.J. Klassen, MRS 2013, San Francisco.
2- M. Haghshenas, L. Wang, R.J. Klassen 23rd CMSC, 2011, UBC.
5- M. Haghshenas, L. Wang, R.J. Klassen, MS&T 2011, Ohio.