Friction

Research Article

Understanding the microforming related tribological science of engineered magnesium alloys at high temperatures

Venkatesh Behera, Sushanta Kumar Panigrahi✉

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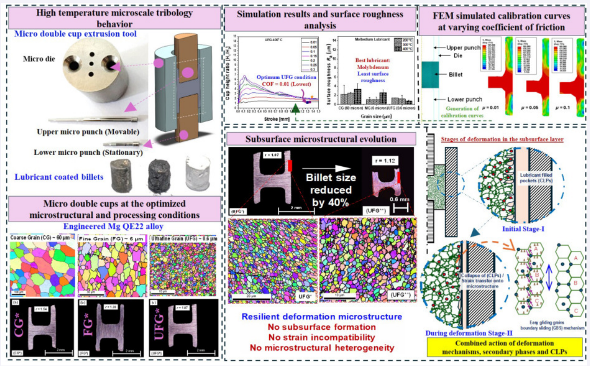
ABSTRACT: Microforming is a promising approach to micromanufacturing miniaturized components. The material flow and tribological aspects of microforming are affected by the size effect. The size effect phenomenon is influenced by parameters such as the initial microstructure, deformation temperature, lubricant type, and billet geometry. The scope of this study is to establish tribology-based scientific knowledge by considering all the mentioned parameters. As a case study to mimic the tribological interaction during microforming, a micro double cup extrusion (MDCE) test is performed on engineered Magnesium QE22 materials. The experiments were performed on various grain sizes, lubricants, and temperatures. A comprehensive investigation of all the conditions indicated that the ultrafine-grained (UFG) microstructure is the best-suited initial microstructural condition for maintaining excellent surface morphology, surface roughness, and microstructural homogeneity. The coarse grain (CG) microstructure exhibited substandard surface properties and microstructural heterogeneity. Electron backscatter diffraction (EBSD) microstructural analysis revealed tribological interactions with the activated micromechanisms under coarse-grained (CG), fine-grained (FG), and ultrafine-grained (UFG) conditions. Under CG conditions, the activation of twins induced dynamic recrystallization resulting in a greater cup height ratio and coefficient of friction (COF). This shows the incompetence of the CG microstructure in accommodating friction-induced shear. On the other hand, the UFG microstructure demonstrated a resilient microstructure that easily accommodated the induced frictional shear by activating the grain boundary sliding (GBS) mechanism. The activation of the GBS mechanism resulted in complete anhelation of the frictional subsurface layer, thereby eliminating the tribological size effect that remained unaffected even when the billets were downsized.



KEYWORDS: : microforming; hot tribology; size effect

1 Introduction

Microforming technology is a micromanufacturing method that can economically produce miniaturized microparts from sheet or bulk materials. The industrial practicality of microforming technology is still a challenge because of the unraveled scientific knowledge. Over the past decade, researchers have tried to gain insights into microscale deformation issues, such as the "size effect", by experimentally scaling down macro metal forming technologies [1, 2]. With this approach, two primary size effect-related problems: (i) material flow and (ii) friction, have been identified. Microforming at elevated temperatures and microstructural engineering/control is an established methodology for managing material flow problems and improving microscale plasticity. Eichenhueller et al. [3] conducted studies on high-temperature microforming of a CuZn15 alloy. The results indicated that high temperature enabled homogeneous material behavior and reduced process variability. Kim and Sa [4] developed micro gears using equal channel angular pressing (ECAP)-processed magnesium alloy. They selected a hot microextrusion route that resulted in high strength and homogeneous material properties of the developed microcomponents. A unique superplasticity-based methodology is incorporated to manufacture micropins and microcups using rare earth-containing magnesium alloys. Researchers have clearly emphasized the importance of microstructural engineering in obtaining high strength and homogeneous mechanical properties [5]. These recent publications on high-temperature/superplastic microforming have confirmed the credibility of high-temperature deformation and microstructural in enhancing material flow by activating allied/second-order slip systems.

During high-temperature forming applications, serious issues

Department of Mechanical Engineering, Indian Institute of Technology Madras, Chennai 600036, India.

✉ Corresponding author. E-mail: skpanigrahi@iitm.ac.in

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such as high friction, wear and surface-initiated fatigue result in billet material adhesion to the die and punch surfaces, peeling of the work material, and improper surface properties [6]. To overcome such problems at the microscale, the information needed to choose a suitable lubricant is currently unavailable. On the other hand, the frictional size effect during high-temperature microforming has never been investigated. Therefore, strategies must be established to address the frictional size effect at high temperatures. Works in the literature on the room-temperature frictional size effect suggest the existence of a region of extensive shear deformation that develops on the product surface purely because of friction interactions between the microtool and the workpiece material. The material experiences a shear deformation gradient rising gradually from the center region to the surface of the workpiece [7]. Hence, understanding the tribological characteristics is crucial for manufacturing near-net-shaped micro products with superior dimensional accuracy, mechanical properties, and surface finish.

Many experimental and analytical methods have been developed under ambient conditions to mimic the frictional interactions between the tool and working material during metal forming at room temperature. The primary objective of these tests is also to determine the suitability of a particular lubricant and to estimate the coefficient of friction (COF)/friction factor accordingly [8]. Some of the widely recognized friction tests include ring compression tests [9, 10], spike forging tests [11], double cup extrusion tests (DCEs) [12, 13], and T-shaped compression tests [14]. Among them, the DCE is widely used because of its greater concurrence with the existing metal-forming process. As observed in real metal forming operations, the DCE test involves extensive material deformation and high pressure between the material and tools. Engel and Eckstein [15] were the first to investigate the tribological size effect via DCE tests. They discussed a mechanical rheological model related to open lubrication pockets (OLPs) and closed lubrication pockets (CLPs) and their effects on tribology during miniaturization. Chan et al. [16] used DCE experimentation to study interfacial friction by varying the dimensions and grain size of a pure copper material. They reported an increased cup height ratio due to inhomogeneous deformation in large/coarse-grained workpieces. The inverse effect is observed in workpieces with finer grain sizes. They also correlated the tribological size effect with the surface layer model and noted the role of OLPs and CLPs during microscale deformation. In a research publication, Ben et al. [14] studied the influence of ultrasonic vibration during DCEs at varying length scales and reported a reduction in frictional force. The overall friction significantly decreases when ultrasonic vibrations are used while the sample size is reduced. The above-cited literature corroborates the application of the DEC test with miniaturization, particularly under ambient-temperature metal-forming conditions. Furthermore, none of the literature highlights the importance of microstructure, lubrication, and specimen dimension in tribology.

In addition to the resolution of size effect-related issues, high-temperature microforming provides the following benefits: better formability, ease of manufacturing complex geometries, and the ability to deform materials with complex crystallography, such as magnesium and titanium alloys. Given the advantages of high-temperature microforming, the size intrinsic friction evolution is a topic that must be understood via experimental research. Therefore, implementing a DCE study at high temperatures necessitates understanding the parameters affecting the friction/COF.

The literature highlights the importance of microstructural engineering, lubrication, and processing parameters and their impact on microforming behavior at room temperature. However, to date, tribology studies on high-temperature microforming has never explored for any materials. This research aims to gain scientific insight into friction and its association with microstructure, lubrication, and specimen miniaturization during high-temperature microforming. A complex microforming material, a rare earth containing magnesium alloy, is chosen as the subject material. The rationale for selecting Mg-rare-earth (RE) alloys is the inability of Mg alloys to withstand wear, especially above 150 °C [17]. To overcome these limitations, Mg-RE alloys containing rare earth elements (Gd, Y, etc.) are the preferred choice for tribological applications because of their high thermal stability and ability to facilitate a protective oxide layer on the Mg alloy surface, leading to enhanced wear resistance [18, 19]. Because of the mentioned advantages of Mg-RE alloys, a Nd-containing rare earth alloy, Mg QE22, was selected for the present study. The Mg-QE22 alloy material is microstructurally engineered to generate stable initial microstructures with varying grain sizes [20, 21]. Three high-temperature lubricants, namely, molybdenum (M), graphite (G), and boron nitride (BN), were selected for the microscale double cup extrusion (MDCE) experiments. A thorough analysis and detailed investigation, such as electron backscatter diffraction (EBSD), surface profilometry, and scanning electron microscopy (SEM), will be carried out on the developed double cups to elucidate the underlying micromechanism. Such pioneering research and observations on tribological effects in high-temperature microforming could benefit the industrial implementation of the process.

2 Experimental

2.1 High-temperature MDCE setup

MDCE at high temperatures is carried out by integrating an induction heating system into the microforming press tool, as shown in Fig. 1(a). To maintain dimensional tolerance during the test, a cooling arrangement is integrated to dissipate the heat generated by the tooling system during the test. The MDCE tooling system consists of an upper punch (movable) and a lower punch (stationary), along with a cylindrical die cavity (Fig. 1(b)). The cylindrical sample is placed between the lower punch and the upper punch before the test begins. The upper punch advances downward during the test, forcing the sample to flow in the clearance between the die and the punch. Since the lower punch and die cavity remain stationary during the extrusion process, the top cup is formed via backward extrusion, whereas the lower cup is formed via forward extrusion. Friction plays a crucial role in determining the height of the cup. Generally, the material flow around the lower cup is more restrictive, resulting in a lower cup height than the upper cup height. The ratio of the upper and lower cup heights of the extruded specimen is defined as the cup height ratio (r), which is shown in Eq. (1):

$$r = h_{\text{upper}}/h_{\text{lower}} \tag{1}$$

where h_{upper} is the height of the upper cup, and h_{lower} is the height of the lower cup.

The cup height ratio is sensitive to friction. In an ideal case, when there is no friction, the heights of the upper and lower cups are equal, and the cup height ratio tends to unity. As the friction increases, the height of the lower cup starts to decrease because of restricted material flow.

2.2 Material selection and tooling

A Mg QE22 alloy containing neodymium (Nd), a rare earth element, was chosen to study the influence of microstructural grain size. This alloy is selected because of its high thermal stability due to the grain boundary pinning effect of the (Nd) RE elements and decent high-temperature formability [20, 22]. Three grain size ranges were synthesized via thermomechanical processing and heat treatment, as mentioned in Ref. [21]. The ultrafine-grained (UFG) grain microstructure was developed via the friction stir processing (FSP) technique, wherein a rotating tool is normally forced and simultaneously transversed onto the Mg QE22 alloy material while imposing extensive shear. The tool rotation speed and number of FSP passes were optimized and studied in Ref. [21]. As a result of extensive shear, the Mg alloy material dynamically recrystallizes, resulting in equiaxed grains with a UFG microstructure. The fine-grain (FG) microstructure was obtained by heat treating the FSP-processed Mg QE22 UFG material at 450 °C for 1 h. The coarse grain (CG) material is the as-received Mg QE22 alloy. The inverse pole figure (IPF) map of the synthesized initial microstructure is shown in Fig. 2. The microstructure appears equiaxed under all three initial conditions, with average grain sizes of CG (60 μm), FG (6 μm), and UFG (0.6 μm).

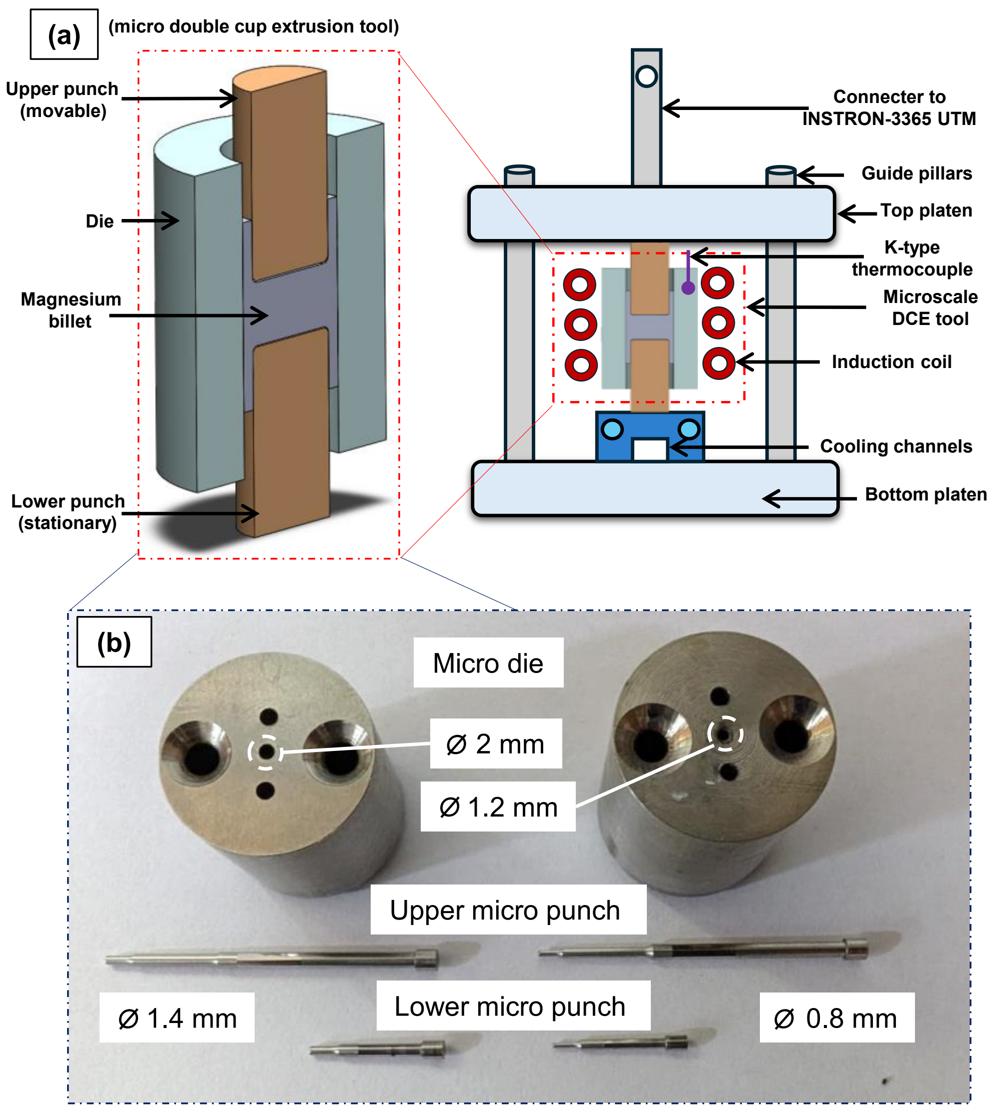

Fig. 1 MDEC press tool:schematic of (a) press tool setup and (b) micro tools (die and punches).

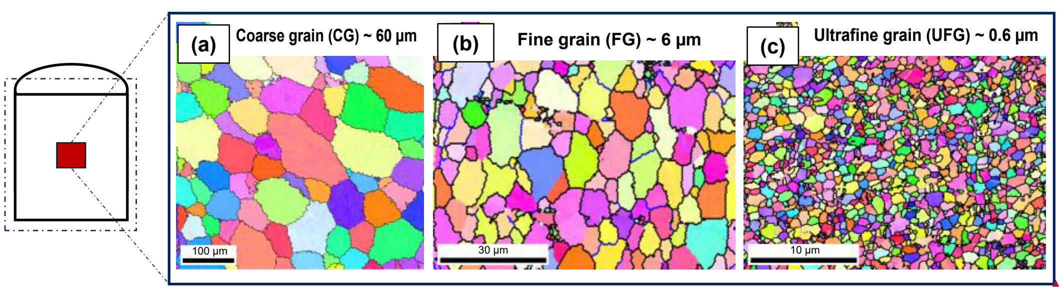

Fig. 2 IPF maps of the initial microstructures synthesized for the MDCE studies: (a) coarse-grained (CG), (b) fine-grained (FG), and (c) ultrafine-grained (UFG).

Cylindrical billets were extracted under all the microstructural conditions for MDCE studies as per the dimensions mentioned in Table 1. Magnesium alloys, hexagonal closed-packed (HCP) crystal structure materials, have limited room-temperature formability because of insufficient slip systems. Therefore, high temperatures above 225 °C are recommended to activate the second-order slip system to accommodate the forming-related deformation strain. Therefore, the safe hot workability domain of Mg and its alloys is generally above 200 °C [23, 24]. Therefore, temperatures of 200, 300, and 400 °C were used for the MDCE experiments. Under all the conditions, the upper punch will be moved to a distance, as mentioned in Table 1, from the billet top at a constant crosshead velocity of 0.01 s^{-1}.

2.3 Selection of high-temperature lubricants

Graphite (G), Molybdenum (M), and Boron Nitride (BN) spray lubricant cans are used in the current study (Fig. 3(a)). The detailed technical data for the lubricants are presented in Table 2. These lubricants were selected on the basis of their high-temperature stability, flash point, good bonding up to 450 °C, and primary application. The exact concentration of the spray is under a trademark; however, the aerosol combination consists of acetone, ethanol, propane, butane, etc., along with a lubricant compound (graphite, boron nitride, and molybdenum). Before lubricant application, the microlillet was polished and ultrasonically cleaned. The spray lubricants were applied from a distance of 8 cm at a spraying angle of 60° with a generous quantity of lubricant (3–5 sprays per sample) to ensure maximum coverage. A curing time of 1 h was maintained before the billets were used for MDCE experimentation. Photographs of the billets after the application of various lubricants are shown in Fig. 3(b).

2.4 Postprocessing and characterization

Initially, the double cups developed are sliced axially using a diamond wafer blade with a Bain cutting machine to measure the cup heights, h_{upper} and h_{lower}. The MDCE cups were examined via SEM to inspect the surface morphology. To understand the microscopic mechanism at the subsurface, EBSD analysis near the surface was performed. The final polishing for the EBSD analysis was performed using a 0.2 μm silica suspension. The surface roughness was measured via a profilometer (Jr25, NANOVIA) with a maximum vertical resolution of 1 nm. Postprocessing software is used to eliminate errors and outliers from the raw data.

Table 1 Dimensions of the micro tools used for the MDCE study

Billet diameter (mm)	Die diameter (mm)	Punch diameter (upper/lower)	Upper punch displacement (mm)
2	2	1.4	1.5
1.2	1.2	0.8	0.9

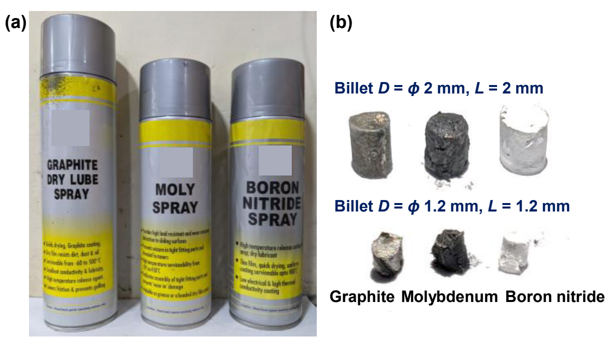

Fig. 3 Photographs of the lubricant spraycan and the coated microbillets before MDCE.

3 Finite element method (FEM) based simulations to study the microscale double cup extrusion process

Abaqus 2D FEM simulation studies were conducted to analyze the material flow and obtain the calibration curves by varying the friction coefficient. The simulation for the current work is carried out via a two-dimensional axisymmetric model due to process symmetry. Upsetting experiments were performed on the Mg QE22 microbillets via exact deformation variables such as sample dimension, grain size, and temperature. These stress–strain data are provided as inputs for simulation studies. The geometry and dimensions of the exact tool (punches and dies) used in the experiments were used to create the 2D model. During the simulation, the punches (both upper and lower) and the die were considered rigid. The simulation studies were performed via the coupled temperature displacement module available in Abaqus software, along with arbitrary Lagrangian Eulerian (ALE) adaptive meshing in Fig. 4.

The Abaqus simulation results at different friction coefficients for the fine-grained microstructure at 400 °C. Similarly, the results for all the other microstructural and temperature variations were obtained. Under all other conditions (Fig. 4), the cup height ratio increased significantly with increasing friction coefficient. The outputs of the simulation studies are plotted as calibration curves. These calibration curves are used in the subsequent sections to determine the COF for the various lubricants at high temperatures during microforming.

4 Results and discussion

4.1 Surface morphology of the samples after microscale double cup extrusion (MDCE)

MDCE experiments at high temperatures are carried out on Mg QE22 alloy billets to understand friction and its effect on tribology. The experiments are performed on all microstructures and processing variables, which are (i) microstructures: UFG (0.6 μm), FG (6 μm), and CG (60 μm) (Fig. 2); (ii) lubricants: graphite, M, and BN (Fig. 3); and (iii) temperatures: 200, 300, and 400 °C. After the experiments, the micro double cups were subjected to SEM under all the conditions to study their surface morphology. The SEM images (Fig. 5) illustrate the surface morphology comparison of Mg QE22 microcups made under all the microstructural conditions and processing variables. The role

Table 2 Technical data sheet of the selected high-temperature spray lubricants

Name of lubricant	Appearance	Serviceable temperature	Particle size (μm)	Flash point	Primary use
Graphite dry lube spray	Black	−20–450 °C	250–385	Highly flammable vapor (flash point below 23 °C)	High-temperature lubricants for forming and manufacturing-related studies
Moly spray	Gray	−50– 450 °C	180–225	−18 °C	
Boron nitride spray	Milky white	800 °C air;1,950 °C inert gas	600–800	−104 °C	

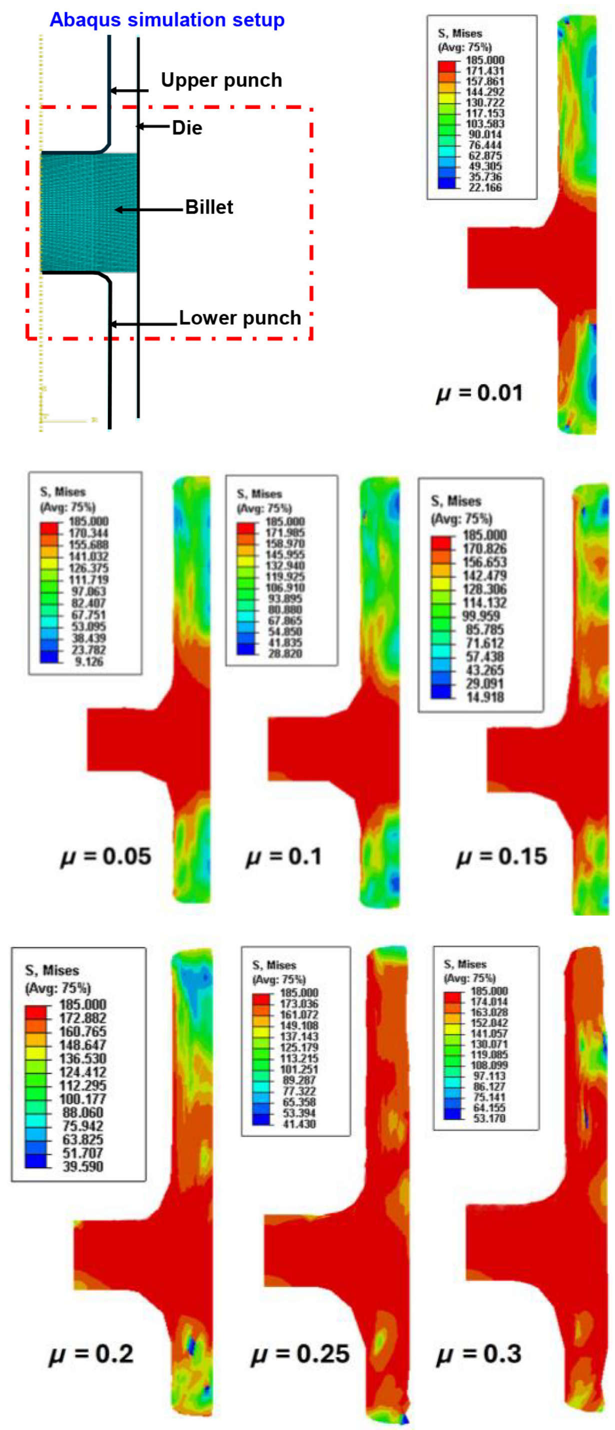

Fig. 4 Abaqus FEM modeling of the MDCE process along with the results.

of initial grain sizes (UFG, FG, and CG) during the MDCE process on surface morphology can be established from the results. Among all the grain sizes, the microcups developed with the UFG microstructure presented the best surface morphology (Fig. 5(a)). However, at low temperatures (200 and 300 °C), signs of severe deformation, cracks, and material peeling are apparent. These surface defects may arise from the greater strain hardening ability of the UFG microstructures. In the UFG microcups, no signs of defects, such as microcracks, material peeling, or delamination, were observed (Fig. 5(a)). The surface quality was relatively smooth at 400 °C under all the UFG microstructural conditions. An optimum surface morphology was observed by using both graphite (G) and M lubricants at 400 °C. The double cups developed in the FG microstructure exhibit cracks at lower temperatures (200 °C), as shown in Fig. 5(b). Magnesium and its alloys generally cannot accommodate large plastic strains at lower temperatures. The slip activity is enhanced by the activation of second-order slip systems that are activated above 225 °C. Therefore, defects such as surface cracks and deep grooves can be more prominent at lower temperatures (200 °C). Under FG conditions (Fig. 5(b)), the best surface morphology was observed at 300 °C when the molybdenum lubricant was used. Temperatures above 400 °C resulted in severe material peeling and detachment. Among all the microstructural conditions, the surface morphology of the CG microstructure is inferior, as shown in Fig. 5(c). Material peeling, adhesion, and plastic deformation indicate the inability of the microstructure to accommodate the imposed large shear gradient in the CG microextrusion condition. In the subsequent sections, a detailed microstructural investigation will be carried out to understand the effects of processing variables (microstructure, lubricants, and temperature) on the microscale deformation mechanism.

4.2 Surface roughness analysis of the developed double microcups

One of the key objectives of any microforming process is to develop micro products with a good surface finish. Therefore, selecting the right lubricant is essential during any microscale deformation process, both at room and high temperatures. The role of high-temperature lubricants (molybdenum, graphite, and boron nitride) and their influence on surface quality during the MDCE process are studied by evaluating the surface roughness via an optical profilometer (Jr25, NANOVIA, USA) (Fig. 6(a)). The profilometer studies indicate an increase in the surface roughness value under CG and FG grain size conditions with increasing temperature (Figs. 6(b)–6(d)). The profilometry results correlate well with the surface morphology analysis, wherein surface delamination, material peeling, and cracks were observed at lower temperatures, whereas at high temperatures, issues such as material detachment and adhesion contributed to an increase in surface roughness in the case of CG and FG materials. However, in the case of UFG, the surface roughness value decreases with increasing temperature. A similar trend in surface morphology was found, where two optimized surfaces were found at 400 °C under UFG conditions. However, this observation in the UFG microstructure is unconventional and indicates the potential for grain size dependence on surface roughness. The correlation between the grain size and the surface roughness will be studied in the subsequent sections via microstructural analysis.

Among the selected high-temperature lubricants, molybdenum lubricants resulted in the lowest surface roughness values across varying grain sizes and temperatures. The order of high-temperature lubricants favorable for microforming applications to obtain the best surface roughness is molybdenum > graphite > boron nitride. Similar lubricant performance has been reported in [25]. Strip drawing test studies on warm and hot forming of aluminum alloys have shown that graphite lubricant is better than boron nitride.

4.3 The effect of processing variables on the friction coefficient

MDCE is performed to study the influence of process variables (microstructure and temperature) and lubrication on tribology during microscale deformation. The obtained microcups were sliced to measure the upper and lower cup heights. The cup height

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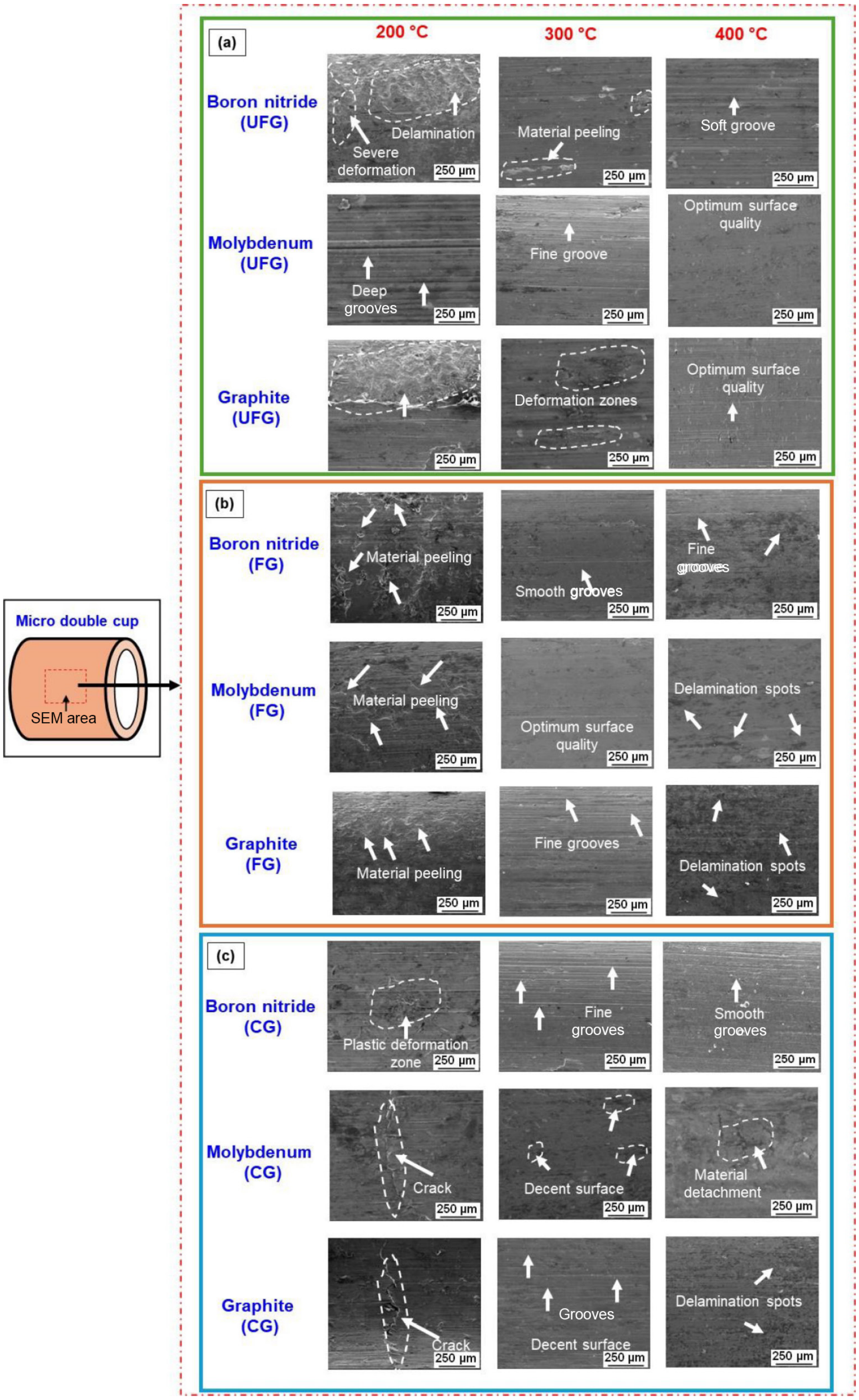

Fig. 5 Results comparing the surface morphology of the micro double cups developed by using three distinct high-temperature lubricants (G, M, and BN) along with variable grain sizes: (a) UFG, (b) FG, and (c) CG microstructures.

ratios are calculated via Eq. (1). The cup height ratios were plotted on the calibration curves obtained via FEM analysis at different friction coefficients. Friction coefficient values were found for the micro double cups under all microstructural and processing conditions, as shown in Fig. 7.

The correlation between the experimental and simulation results led to the following conclusions: (i) The optimum cup height ratios among all the temperatures for the CG, FG, and UFG microstructures are 1.50, 1.38, and 1.07, respectively. (ii) The UFG microstructure at 400 °C resulted in the lowest cup height

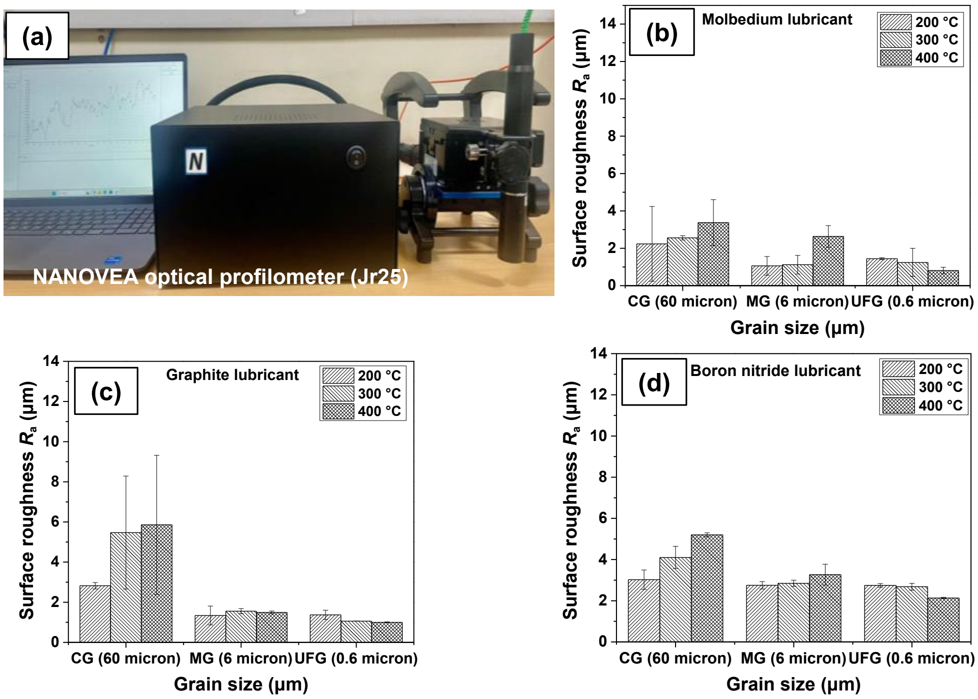

Fig. 6 Surface roughness (R_a) study: (a) optical profilometer (Jr25 NANOVEA USA), (b) R_a results for the molybdenum lubricant, (c) R_a results for the graphite lubricant, and (d) R_a results for the BN lubricant at various grain sizes and temperatures.

ratio (1.07). (iii) The cup height ratio decreased with increasing temperature for all grain sizes; for example, in the UFG microstructure, it decreased from 4.14 at 200 °C to 1.07 at 400 °C. (iv) Among the three high-temperature lubricants, the molybdenum lubricant was found to be the best (based on the COF), irrespective of the temperature and microstructure variations, as marked with a blue square in Fig. 7. The COF corresponding to the cup height ratio is determined via experimental and FEM analyses. Microcons with the lowest friction coefficient are selected from all three microstructures (UFG, FG, and CG) (Fig. 8), and such conditions are listed in Table 3. These cases were further subjected to EBSD analysis to understand the underlying micromechanism and its influence on tribology.

4.4 Understanding the role of microstructure and its effect on the tribological subsurface

During the microextrusion process, material flows in the cavity due to the interaction between the microextrusion die, punch, and billet material. Owing to this friction interaction, a shear gradient is generally observed near the subsurface (as shown in the red-highlighted part in Fig. 9(a)) while moving toward the center. This shear gradient influences the subsurface microstructure during the MDCE process. The surface finish and defect propensity of the microcups to be developed depend on the ability of the microstructure to withstand deformation. This means that at high-temperature microscale deformation, the microstructure and lubrication play crucial roles in the tribological behavior. Therefore, a stable microstructure that can easily accommodate the imposed shear deformation would benefit the tribological aspect of microforming.

To understand the micromechanisms and their relationships with tribology, the EBSD microstructure is captured near the subsurface of all three microstructures (Fig. 9(a)). The IPF map of the coarse-grained condition (CG*) after deformation is shown in Fig. 9(b). The grains near the subsurface are fully recrystallized with a basal texture. Large twin lamellae (marked in yellow) within the coarse grains just after the substructure zone indicate the twin-induced dynamic recrystallization mechanism (TDRX). TDRX has formed via three sets of nucleation phenomena (Fig. 9(b)): (i) mutual intersection of contraction twins $\{10\bar{1}1\}$ (shown using red arrows) leads to high stress concentration zones that trigger TDRX formation; (ii) segmentation of original grains by primary twins facilitates the formation of extended twin boundaries, and these extended boundaries are energetically rich sites ideal for new TDRX grains; and (iii) subdivision of twin laminar boundaries (shown using white arrows) in the process of accommodating frictional shear strain leads to the transformation of low-angle grain boundaries (LAGB) to high-angle grain boundaries (HAGB), thereby resulting in the formation of new recrystallized grains [26]. Complementing these observations, a high fraction of low-angle grain boundaries (48%) was found during the analysis. The schematic in Fig. 10(a), marked with a gray arrow, shows the twinning-based nucleation mechanisms. The refined grains observed in the substructure region are due to the complete transformation of low-angle boundaries within the twin lamellae into high-angle boundaries in the process of accommodating the severe friction-induced strains [27]. Additionally, the occurrence of significant twinning indicates sufficient plastic deformation behavior, which led to delamination and cracks during strain accommodation in the Mg QE22 CG material. Therefore, a condition such as CG* with microstructural heterogeneity is not ideal for microforming applications.

The subsurface under fine-grain conditions (FG*) consists of fine necklace structures around the grains. These necklace structures indicate a discontinuous dynamic recrystallization process. The grains next to the substructure layer show slight grain growth compared with the initial microstructure. The IPF map in Fig. 9(c) indicates a high fraction of high-angle grain boundaries (79%). This shows a greater recrystallization propensity under fine-grain conditions (FG*) than under coarse-grain conditions (CG*). The discontinuous recrystallization mechanism can be attributed

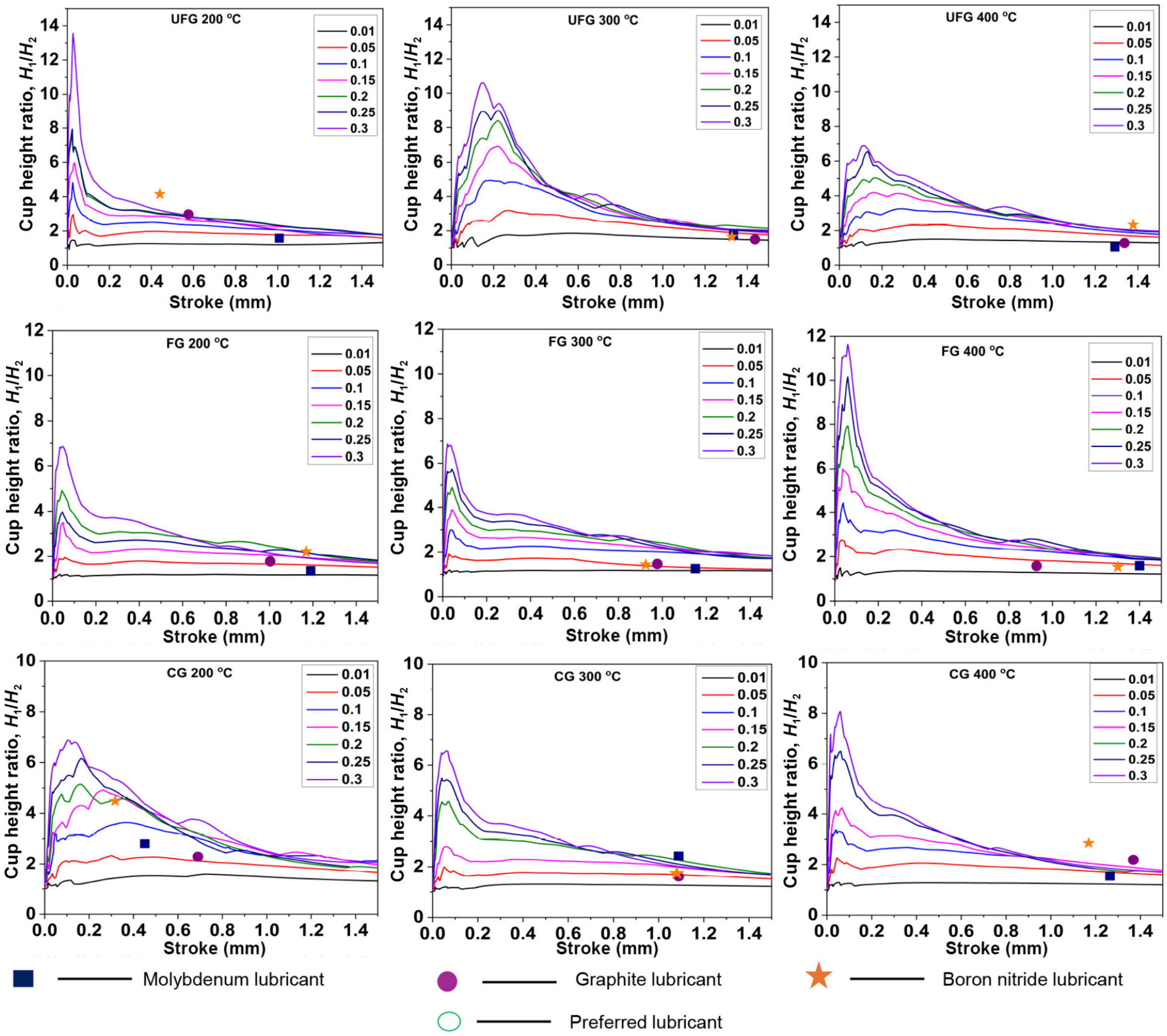

Fig. 7 Comparison of the experimental cup height ratios and the calibration curves obtained via Abaqus simulations with various process variables.

to the presence of thermally stable $Mg_{12}Nd$ precipitates along the grain boundaries, as shown in Fig. 10(b) using blue circles. The transfer of shear deformation strain due to friction between the die surface and the billet leads to the generation of a strain field/dislocation around the grain boundaries. These dislocation-rich zones along the grain boundaries are also termed particle deformation zones (PDZs), and such zones are preferential sites for the nucleation of new recrystallized grains. Therefore, the strain induced by the tribological interaction is utilized to facilitate recrystallization. However, substantial grain size heterogeneity is observed for FG*.

The microstructural evolution under ultrafine grain microstructure (UFG*) conditions after the MDCE process. Figure 9(d) was significantly different from that under CG* and FG* conditions. The ultrafine grain microstructure is resilient to imposed shear frictional deformation. The grain structure near the subsurface layer appears slightly elongated. However, no sign of dynamic recrystallization/change in texture or twinning exists. It is well known that such microstructurally engineered ultrafine grain microstructures can trigger grain boundary sliding (GBS) mechanisms, which have also been studied extensively [5]. The $Mg_{12}Nd$ precipitates present in the Mg QE22 alloy play a pivotal role in the activation of the GBS mechanism by pinning the grain boundaries and restricting grain growth, even when the temperature is high (400 °C) (as shown in the schematic in Fig. 10(c)). During GBS, the strain imposed by friction between the micro die and billet is fully accommodated by the sliding of adjacent grains, and the dislocation activity is restricted along the grain boundaries. The GBS mechanism eliminated subsurface formation under UFG* conditions, as observed in the IPF map. This is the reason for the exceptional surface finish and the lowest friction coefficient under the condition UFG*. Therefore, microstructural engineering is crucial and has a determining role in the tribology, friction coefficient, surface finish, and overall material properties.

4.5 Tribological size effect its response to high temperatures and varying microstructures

The size effect is an anomaly observed during microforming under ambient conditions that causes an increase in friction when the specimen geometry decreases. To investigate the influence of size on high-temperature microforming, the dimensions of the original Mg billets are downsized/reduced by 40%. These billets are then subjected to the MDCE process using the selected molybdenum lubricant under optimum performance conditions (FG* and UFG*). These downsized billet conditions are referred to hereafter as FG** and UFG**. The results in Figs. 11(a) and 11(b) illustrate the double cups that show little deviation in the

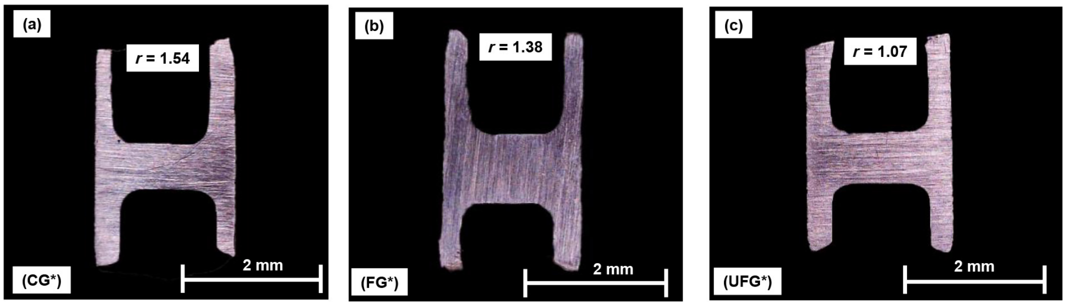

Fig. 8 Cross-sectional view of the micro double cups selected for microstructural analysis: (a) CG (300 °C), (b) FG (300 °C), and (c) UFG (400 °C).

Table 3 Selected cases marked with *, showing the lowest cup height ratio/friction coefficient

Condition	Microstructure (μm)	Temperature (°C)	Cup height ratio (*r*)	Friction coefficient (*μ*)	Lubricant
CG	60	200	2.80	0.10	Moly
CG	60	200	2.29	0.05	Graphite
CG	60	200	4.47	0.20	BN
CG*	60	300	1.54	0.05	Moly
CG	60	300	1.74	0.05	Graphite
CG	60	300	2.24	> 0.30	BN
CG	60	400	1.79	0.10	Moly
CG	60	400	2.18	> 0.30	Graphite
CG	60	400	2.84	> 0.30	BN
FG	6	200	1.41	0.05	Moly
FG	6	200	1.77	0.05	Graphite
FG	6	200	2.20	0.20	BN
FG*	6	300	1.38	0.05	Moly
FG	6	300	1.46	0.05	Graphite
FG	6	300	1.43	0.05	BN
FG	6	400	1.40	0.05	Moly
FG	6	400	1.57	0.05	Graphite
FG	6	400	1.52	0.05	BN
UFG	0.6	200	1.57	0.05	Moly
UFG	0.6	200	2.96	0.30	Graphite
UFG	0.6	200	4.14	> 0.30	BN
UFG	0.6	300	1.33	0.01	Moly
UFG	0.6	300	1.49	0.05	Graphite
UFG	0.6	300	1.64	0.05	BN
UFG*	0.6	400	1.07	0.01	Moly
UFG	0.6	400	1.27	0.01	Graphite
UFG	0.6	400	2.33	> 0.30	BN

cup height ratio (*r*) (approximately 5%) under the conditions (FG** and UFG**) compared with the 2 mm diameter billet studies (FG* and UFG*). The EBSD IPF maps captured near the subsurface layer (marked in red) of the downsized microcups (FG** and UFG**) are shown in Fig. 11(a). The results indicate an indistinguishable deformation mechanism compared with that of larger billets (FG* and UFG*). In the FG** material, discontinuous dynamic recrystallization is observed in the subsurface region. The fraction of high angle grain boundaries (HAGB) is 79%, with an average grain size of 10±2.0 μm. In the UFG** material, the grains appear free from any deformation strain. The average grain size is approximately 1.23±3.0 μm, indicating slight grain growth along with the activation of the GBS mechanism, as in UFG*. The results observed in the downsized conditions, FG** and UFG**, at high temperatures are inconsistent with the size effect behavior observed under ambient conditions [28, 29].

On the basis of the literature on micrforming [30], the size effect at room temperature microforming is associated with the model of OLPs and CLPs. The applied lubricant is trapped inside the CLPs and creates a hydrostatic cushion that counteracts friction [30–32]. With decreasing specimen dimension, the fraction of CLPs decreases, resulting in an increase in friction [33]. However, in the present work, MDCE studies were carried out at high temperatures, where the size effect phenomenon related to CLPs and OLPs in microforming is unexplored.

The phenomenon related to the size effect in high-temperature microforming can be divided into two stages. Stage I signifies the role of CLPs, and Stage II relates to the activities of allied micromechanisms in frictional strain accommodation. During the MDCE process, the starting stage (stage I) of deformation (Fig. 12) illustrates the lubricant filling action of surface asperities (also known as CLPs). The number of CLPs formed depends on the initial grain size of the billet. Therefore, lower and higher CLPs

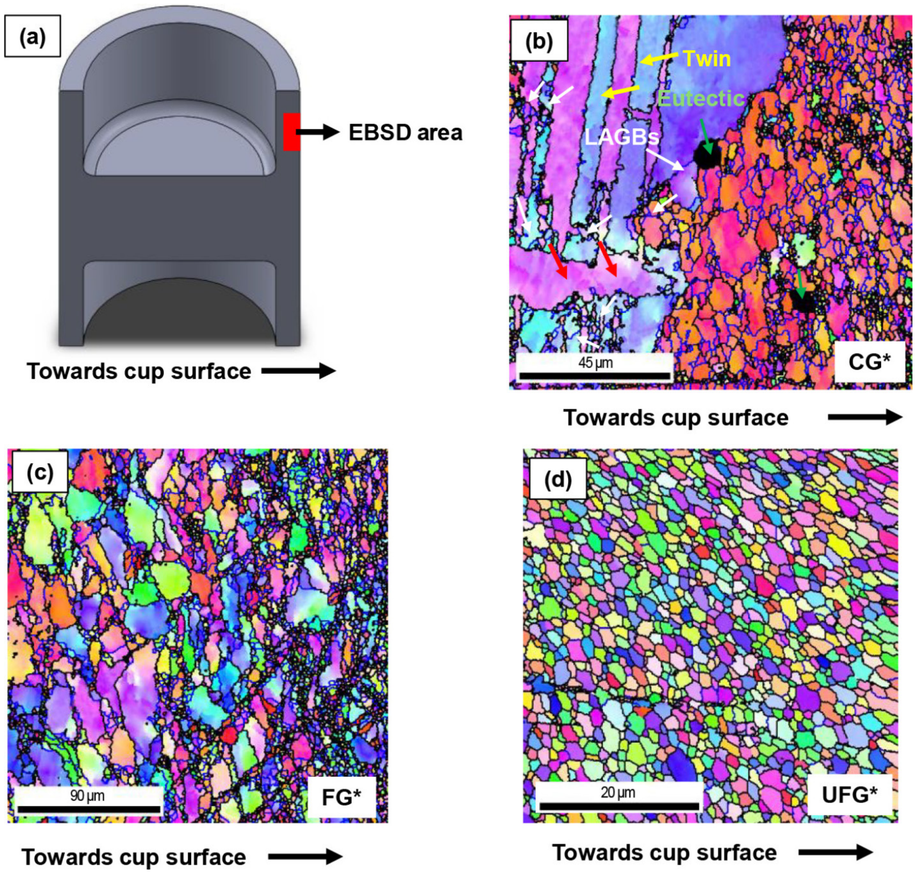

Fig. 9 Microstructural analysis of the subsurface on the microcups: (a) schematic showing the EBSD scan area; (b) IPF map (CG*) GS: 60 μm, T = 300 °C; (c) IPF map (FG*) GS: 6 μm, T = 300 °C; (d) IPF map (UFG*) GS: 0.6 μm, T = 400 °C.

are expected in the case of CG and UFG materials, respectively. It is apparent that the CG material is soft and that the UFG material is relatively hard because of the grain boundary strengthening effect. Owing to both the property difference and the presence of a greater number of CLPs in the UFG condition, the lubricant is trapped in a relatively greater fraction of asperities. These trapped lubricant pockets impose a hydrostatic pressure that counteracts/resists the friction-induced shear strain for a longer distance than the CG material does. Eventually, because of the high deformation temperature and severe interfacial friction, the peaks and valleys (asperities) plastically deform and flatten, as shown in Fig. 12. In the next stage of deformation, the frictional strain imposed is transferred into the microstructure directly and accommodated by their allied micro-mechanism activity (stage II). Depending on the operating micromechanism (TDRX, discontinuous dynamic recrystallization (DDRX), GBS), the extent of the subsurface deformation layer changes. Under optimized conditions (UFG*), the combined action of GBS mechanism activation and a large fraction of CLPs resulted in deformation homogeneity and complete anhelation of subsurface formation.

Downsizing the billets (UFG** and FG**) by 40% led to a reduction in the number of CLPs that affected only stage I. However, stage II remained unaffected, and sufficient grains in the deformation zone led to the activation of relevant micromechanisms in the UFG** and FG** conditions. This resulted in homogeneous deformation and fair accommodation of friction-induced shear strain.

Along with the initial microstructure, the second phase particles $Mg_{12}Nd$ and their presence along the grain boundaries assisted in bearing frictional strain. Under UFG* and UFG** conditions, the initial UFG microstructure is engineered by friction stir processing, which positions the $Mg_{12}Nd$ precipitates along the grain boundaries. These particles promoted GBS and restricted grain growth at high temperatures. Additionally, frictional shear strain at high temperatures resulted in the discretization of $Mg_{12}Nd$, which precipitated into a nanoscale regime. These nanosized hard $Mg_{12}Nd$ precipitates restrict further sliding, including abrasion wear [34], and help minimize friction strain.

These observations related to the size effect and the role of micro mechanisms are novel and indicate the role of optimizing microstructural engineering and process variables (lubrication and temperature) to benefit the tribological aspect of microforming.

4.6 Comparison of tribology during MDCE

Room-temperature MDCE has been well studied in the literature for various easy-to-deform materials, such as copper and aluminum alloys. For example, the MDCE at room temperature for pure copper [16] resulted in average cup height ratios of 3.67 and 3.82 for grain sizes of 20 and 150 μm, respectively. The cup height ratio for the CuZn15 material [31] with machine oil is approximately 1.5–2 for samples with similar dimensions and increases further with decreasing depth. At room temperature, the material plasticity is extensively affected by anisotropic grain properties, resulting in irrational local deformation when only a few grains flow into the microsized cavity. For improvement, ultrasonic vibrations were employed to mitigate tribological degradation during microscale deformation. A cup height ratio of 1.18 was achieved via ultrasonic vibrations on both the upper and the lower punches [35]. However, for practical microtooling applications, ultrasonic vibrations can affect the manufacturing

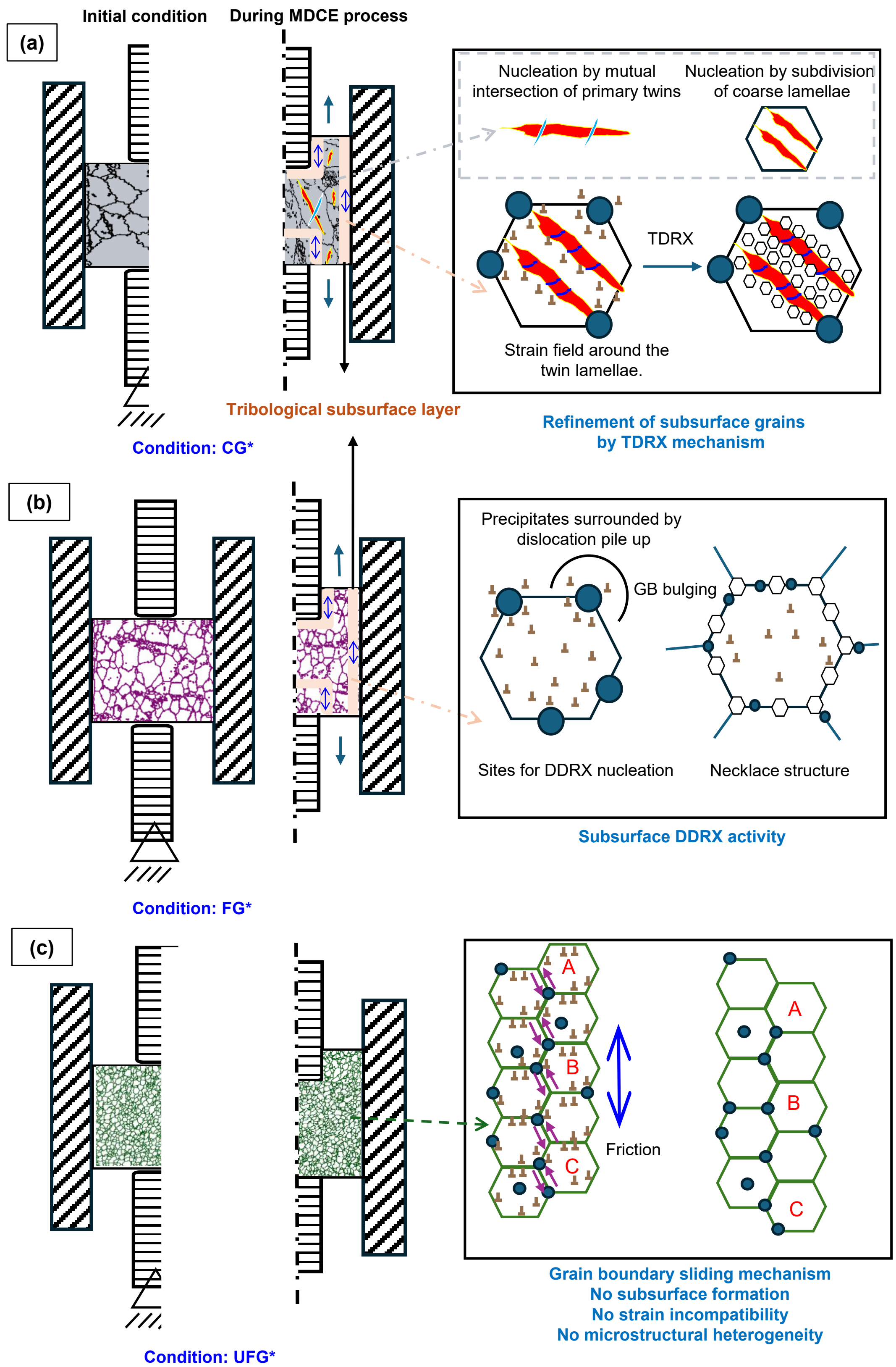

Fig. 10 Schematic illustrating the deformation mechanisms in the subsurface region: (a) coarse-grained (CG*) GS: 60 μm, T = 300 °C; (b) fine-grained (FG*) GS: 6 μm, T = 300 °C; (c) ultrafine-grained (UFG*) GS: 0.6 μm, T = 400 °C.

accuracy and repeatability, as microforming tooling systems are sensitive to vibrations.

The microscale tribological interactions during miniaturized cold microforming operations are distinct from those during hot microforming. HCP materials, such as magnesium and titanium alloys, can only be deformed to manufacture microcomponents via high-temperature microforming processes [36]. Therefore, the present article, for the first time, studies microscale hot tribology in magnesium alloys via MDCE experiments. Using a systematic strategy of microstructural engineering, lubricant, and process

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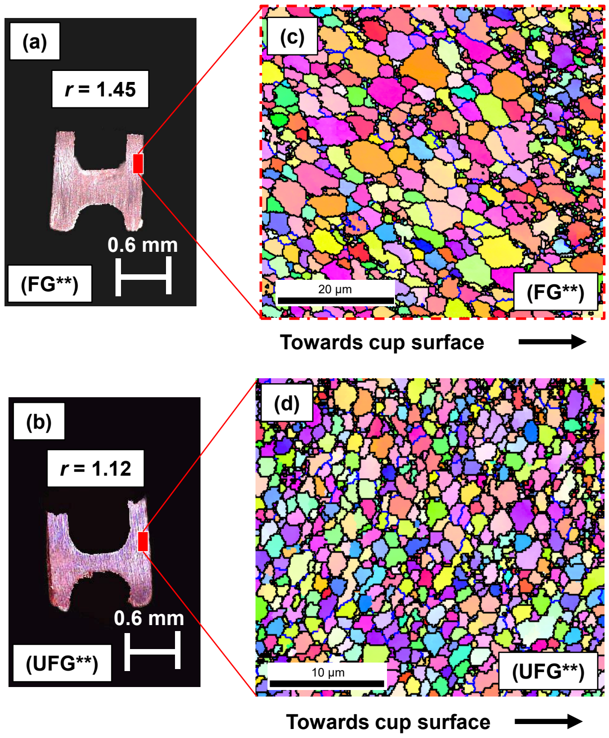

Fig. 11 Results showing the impact of miniaturization on the size effect: (a) IPF map under the condition (FG**) GS: 6 µm, T = 300 °C; (b) IPF map under the condition (UFG**) GS: 0.6 µm, T = 400 °C.

parameter optimization, a cup height ratio of 1.07 is achieved, representing the lowest value reported in the literature, by utilizing a UFG microstructure. This is the lowest cup height ratio obtained in comparison with that reported in the literature. Furthermore, the cup height ratio has almost no deviation when the samples are downsized.

5 Conclusions

The hot tribology at the microscale is studied through MDEC on a magnesium (QE22) alloy. The MDEC tests were performed at various microstructures (CG, FG, and UFG), temperatures (200, 300, and 400 °C), and lubrications (molybdenum, graphite, and boron nitride). For each microstructural condition, optimized parameters were identified on the basis of the lowest surface roughness, the lowest cup height ratio, and the defect-free surface morphology. These optimum conditions for UFG, FG, and CG materials are designated CG* (300 °C, 0.1 mm/s), FG* (300 °C, 0.1 mm/s), and UFG* (400 °C, 0.1 mm/s), respectively, which were further investigated. The key highlights of the present study include the following:

(1) Among the three lubricants, the microcomponents extruded with the molybdenum lubricant had the highest surface finish, irrespective of the initial microstructure and processing variables used. Therefore, molybdenum lubricant is recommended for microforming applications at high temperatures.

(2) The double cups obtained after the MDCE tests were analyzed to measure the cup height ratio (r). Friction calibration

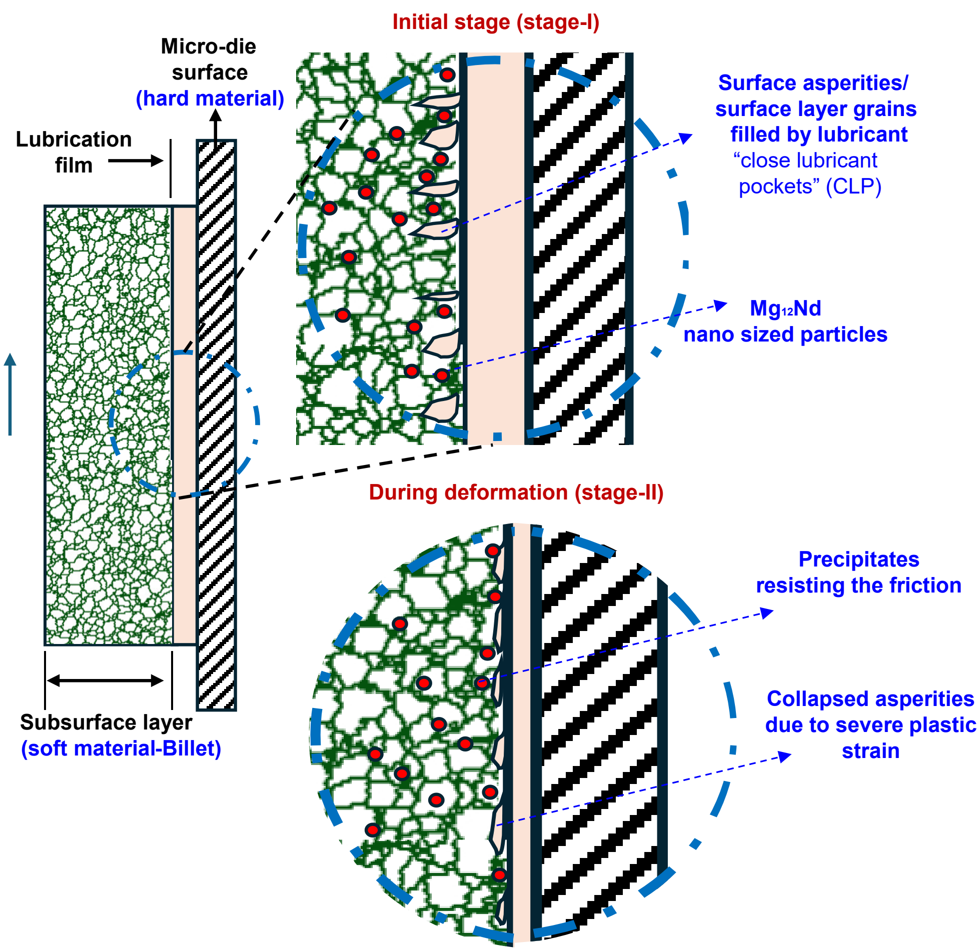

Fig. 12 Proposed mechanism for asperities and friction during high-temperature MDCE.

curves were developed via FEM simulations by varying the COF under all the mentioned conditions.

A comparison of the FEM calibration curves with the experimental results revealed that UFG* had the lowest COF and cup height ratio (*r*). The COF and *r* value for the UFG* condition are 0.01 and 1.07, respectively.

(3) Postmortem EBSD analysis near the subsurface indicated the activation of TDRX under CG*, DDRX under FG*, and GBS under UFG* conditions.

(4) Under UFG* conditions, the activation of the GBS mechanism led to anhelation of the subsurface layer. These findings indicate that the ultrafine-grain microstructure UFG* is resilient and can withstand frictional strain while maintaining grain homogeneity. This optimized microstructural condition (UFG*) can benefit the tribological aspects of high-temperature microforming.

(5) The size effect is dominant in microforming applications at room temperature. The high-temperature-based size effect phenomenon was studied by decreasing the initial billet geometries of the engineered materials. The FG and UFG materials were unaffected and showed no friction variations due to size reduction. This shows the potential of microstructural engineering in addressing the size effect phenomenon at high temperatures.

In the future, it would be interesting to study the microscopic surface roughness of micro double cups via sophisticated equipment such as atomic force microscopy (AFM) to understand the exact contributions of open and closed lubricant pockets and the role of surface asperities.

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Declaration of competing interest

The authors have no competing interests to declare that are relevant to the content of this article.

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Venkatesh Behera is a postdoctoral researcher at École Polytechnique Fédérale de Lausanne (EPFL), Switzerland. He got his master degree and Ph.D. degree in mechanical engineering at Indian Institute of technology, Madras, India. His research focuses on scale dependent tribology, lubricants for metal forming application, magnesium processing, superplasticity, manufacturing technology development, and structure property correlation.

Sushanta Kumar Panigrahi is a full-time professor and doctoral supervisor at Indian Institute of Technology, Madras, India. His research focuses on innovative material processing, advanced casting and sustainable manufacturing, high performance sheet development, solid-state processing and joining of similar and dissimilar materials, plasticity and deformation behavior of materials, and studying tribology and wear application of metallic materials.