EPiC Series in Technology
Volume 4, 2026, Pages 22–29
Proceedings of The 11th International Conference on Tribology in Manufacturing Processes & Advanced Surface Engineering

Effect of Die Surface Conditions on Microstructural Changes and Force Behavior in Micro-Extrusion of Pure Copper

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Abstract

In recent years, copper-based conductive and heat dissipation components have required fine structures for miniaturization and enhanced functionality. Micro-forming is an excellent processing method characterized by high productivity and suitability for mass production. Since micro-forming is highly influenced by tribological conditions, proper setting of die surface conditions is essential. In micro-extrusion, surface coatings such as diamond-like carbon (DLC) have been explored to optimize tribological conditions. However, the effects of die surface properties on the internal deformation state (evolution of the work-hardened microstructure and the resulting plastic-flow) during forming have not been quantitatively characterized. In this study, micro-extrusion tests of pure copper were conducted using two types of dies: one with a DLC coating and one without any coating. Changes in the internal deformation with stroke progression were analyzed using kernel average misorientation (KAM) maps and geometrically necessary (GN) dislocation density. Measurement of GN dislocation density enabled quantitative evaluation of the internal deformation state within the material. Consequently, changes in GN dislocation density with stroke progression closely corresponded to the force behavior, suggesting that die surface conditions significantly affect not only the frictional state at the material surface but also the internal deformation.

1 Introduction

In recent years, copper-based conductive and heat dissipation components have increasingly demanded fine microstructures to achieve miniaturization and enhanced functionality. Micro-forming, characterized by high productivity and suitability for mass production, has been recognized as a promising manufacturing method for these components [1-3]. Compared to macro-scale forming, micro-forming is more sensitive to size effects, and thus tribological conditions between the material and die play a significant role in controlling forming force and product shape [4-7]. Accordingly, appropriate control of die surface conditions tailored to the intended forming process is essential.

In micro-extrusion, surface coatings on dies have been explored to reduce forming forces and extend die life. Funazuka et al. reported that diamond-like carbon (DLC) coatings effectively reduce friction between the die and aluminum alloy samples during micro-extrusion, demonstrating notable lubrication effects [8]. Even for copper materials, DLC coatings have been reported to provide effective lubrication in micro-extrusion, thereby reducing the extrusion force [5,9]. However, the forming force is influenced not only by friction at the sample-die interface but also by the internal deformation state within the sample. In this paper, the term internal deformation state refers to the evolution of work-hardened microstructure and the resulting plastic flow. Understanding how die surface conditions affect such the internal deformation state is necessary for optimizing processing conditions aimed at reducing the extrusion force and extending die life. Nevertheless, the influence of die surface properties on the internal deformation within the sample during micro-extrusion of copper and copper alloys remains have not been quantitatively characterized.

This study aims to elucidate the effect of die surface conditions on the internal deformation state within the sample during cold micro-extrusion of pure copper billets. By analyzing the internal deformation state under different die surface finishes, the relationship between die surface characteristics and the internal deformation state within the sample is discussed.

2 Experimental procedure

2.1 Micro-extrusion test

The extrusion test conditions were set to be equivalent to those used in the authors' previous studies [10]. The test material was pure copper, which was processed and heat-treated to obtain the average grain size to 127.3 μm and then machined to cut out a cylindrical billet with a diameter of 1.7 mm and a height of 6.0 mm. For the micro-extrusion process, forward and backward extrusion was used. Fig. 1 shows a schematic diagram of the die and punch. The die has a split structure to remove

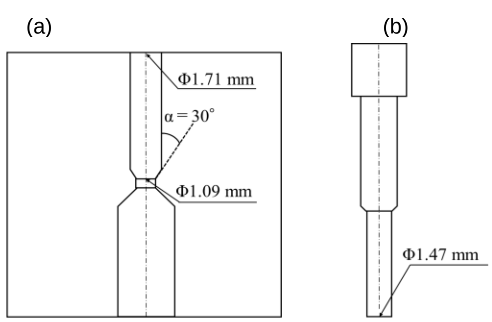

Fig. 1 Dimensions of the (a) die and (b) punch [10].

the billet after extrusion and consists of three sections: a container section, a tapered section, and a bearing section. The inside diameter of the container is 1.71 mm, the inside diameter of the bearing is 1.09 mm, and the angle (die semi-angle) α between the die face of the tapered section and the extrusion axis direction is 30°. In the experiment, two types of die were used: one with a polished surface (mirror surface die) and one coated with diamond-like carbon (DLC die). The punch tip diameter was 1.47 mm, and the cross-sectional area reduction rate was set to 59.4% for forward extrusion and 73.9% for backward extrusion. For both dies, a mirror-finished punch was used. Micro-extrusion was performed at room temperature under the following conditions: ram stroke velocity 0.10 mm/s, maximum ram stroke 2.00 mm, hold time 1.0 s, and lubricant kinematic viscosity 429 mm^2/s. To observe microstructure changes with the progression of stroke, interrupted tests were conducted by stopping the ram at predefined stroke positions less than 2.00 mm.

2.2 Microstructural observation

After the micro-extrusion tests, the samples were sectioned parallel to the extrusion direction. The cross-sections were observed using optical microscopy for shape observation and electron backscatter diffraction (EBSD) for crystallographic orientation analysis. Kernel average misorientation (KAM) maps were obtained using OIM Analysis version 8.0, developed by TSL Solutions. To further assess the effect of die surface conditions on the internal deformation state within the sample, an analysis focusing on dislocation density was conducted. There are two types of dislocations: geometrically necessary (GN) dislocations and statistically stored (SS) dislocations. GN dislocations represent additional dislocation storage required to accommodate lattice curvature arising from heterogeneous plastic deformation [11,12]. The GN dislocation density is known to correlate with KAM values, which quantify orientation differences between adjacent pixels obtained from crystallographic orientation analysis [13-15]. In this study, GN dislocation density was calculated from KAM maps derived from crystallographic orientation analysis to quantitatively evaluate changes in the internal deformation state with the progression of stroke. Using this method, the effects of differences in die surface conditions on the changes in the internal deformation state within the sample were investigated. To derive the GN dislocation density, it is necessary to obtain the variation of the average KAM value with respect to step size ($\frac{d}{ds}\theta_{ave}$) from EBSD observations. However, acquiring multiple EBSD maps with varying step sizes for each sample and field of view would be impractically time-consuming. Therefore, the analytical method proposed by Daigo et al. [16], which calculates $\frac{d}{ds}\theta_{ave}$ from average KAM values obtained by varying the kernel radius from 1 to 5, was employed in this study.

3 Results

Fig. 2 shows the product shapes extruded using (a) the mirror surface die and (b) the DLC die with a stroke of 2.00 mm. This figure includes the forward extrusion length (l_f) and backward extrusion length (l_b). Compared to the mirror surface die, the DLC die exhibits a shorter l_b and a longer l_f. Comparing the maximum forces in the stroke-force curves (Fig. 3), the DLC coating reduced the maximum force by 0.21 kN (8.6%). The maximum extrusion force is determined by the forces required to extrude the sample forward and backward, and the ratio of l_b to l_f is governed by the balance between these forces. These results suggest that DLC coating on the die reduces the force in micro-extrusion of pure copper, with a greater force reduction effect in the forward extrusion region than in the backward extrusion region.

Focusing on the force behavior, as indicated by the arrows in the fig. 3, both dies exhibit three sequential behaviors from the start of the test: (1) a linear increase, (2) a peak, and (3) a gradual decrease. The initial linear increase in force during the early stage of the extrusion test corresponds to the increase in the deformed volume at the forward and backward ends of the sample. The gradual decrease in force observed in the later stage corresponds to the reduction in friction force due to the decrease in contact area between the sample and the container section of the die. In this stage showing a gradual decrease in force, the gradient of the force is approximately constant, indicating that deformation has already stabilized. However, the peak of the force–stroke curve cannot be explained by such simple reasons. In our previous study [10], we pointed out the relationship between the peak of the force–stroke curve of the force and the dead metal-like zone (DMLZ)that forms near the contact surface with the tapered section of the die immediately after the start of the test. The DMLZ acts as a resistance to subsequent plastic flow and is thought to alter the effective extrusion ratio, resulting in peak of the force. However, quantitative evaluation of changes in the internal deformation state with stroke progression has been insufficient, and the effects of differences in die surface conditions have not been accurately evaluated.

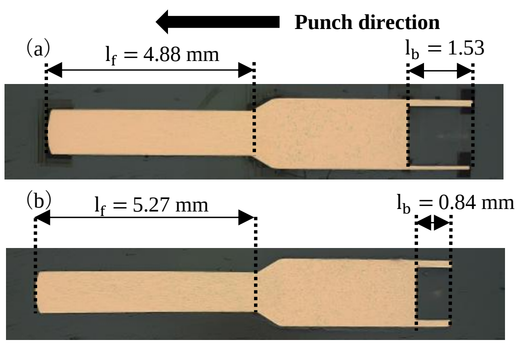

Fig. 2 Product shapes extruded using (a) the mirror surface die and (b) the DLC die.

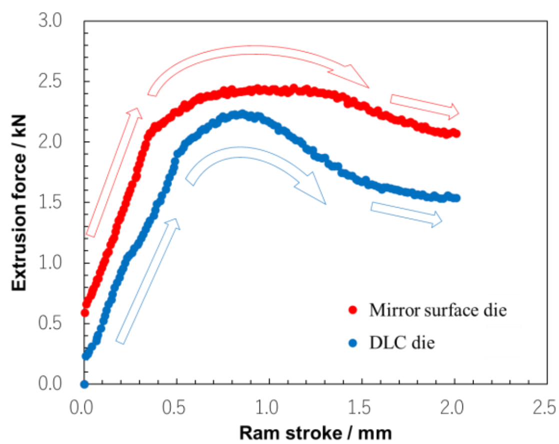

Fig. 3 Extrusion force - Ram stroke curve of the mirror surface die and the DLC die.

Fig. 4 shows the KAM maps of the tapered section for both the mirror surface die and the DLC die. In both dies, a heterogeneous distribution of KAM values is observed immediately after the start of the test (M1, D1), with deformation particularly concentrated near the contact surface with the die. Around the maximum force of the force-stroke curve for each die (M2, D2), the green-colored region expands inward toward the billet radius, indicating that large strains are introduced over a wide area in the tapered section. This behavior is attributed to the formation of the dead metal-like zone (DMLZ) immediately after the start of the micro-extrusion and to the subsequent transition of localized deformation inward from this zone toward the billet radius. After the peak of the force–stroke curve (M3, D3), a larger blue region with low KAM values is observed near the container section. This behavior is consistent with the author’s previous report [10], which showed that deformation stabilizes as the pronounced work-hardened deformation structure is pushed forward from the tapered section. Thus, the changes in the KAM maps with stroke progression reflect changes in the internal deformation state within the sample.

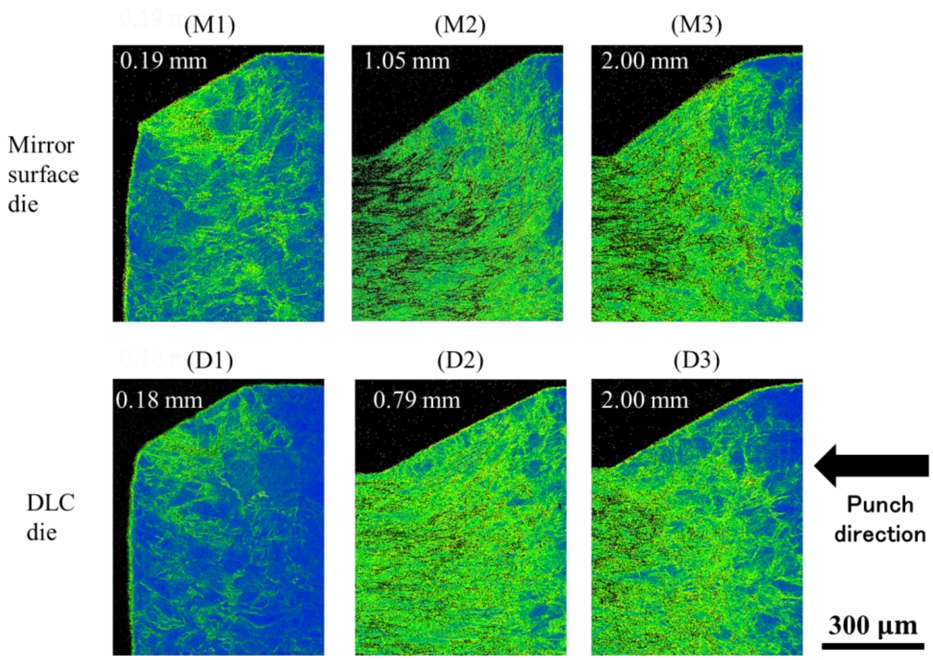

Fig. 4 KAM maps of the tapered section at ram strokes of (a) 0.18 mm, (b) 0.52 mm, and (c) 2.00 mm for the DLC die

4 Discussion

As shown in Section 3, KAM maps suggest changes in the internal deformation state as stroke progresses. However, evaluation of KAM maps remains qualitative. For quantitative evaluation, it is necessary to derive a metric that corresponds to changes in the internal deformation state.

Fig. 5 shows the changes in GN dislocation density with stroke progression in the same field of view as Fig. 4, plotted on the force-stroke curves. Both the mirror surface die and the DLC die exhibit changes in GN dislocation density with stroke progression. GN dislocations arise from plastic strain gradients, and their variations reflect changes in the deformation state. The increase in GN dislocation density from the start of the test to the maximum force is considered to correspond to the transition of localized deformation inward toward the billet axis. The decrease in GN dislocation density after the maximum force corresponds to the phenomenon in which the pronounced work-hardened deformation structure is pushed forward from the tapered section. This suggests that GN dislocation density enables quantitative evaluation of the internal deformation state.

Comparing the changes in GN dislocation density between the mirror surface die and the DLC die, they are similar up to the maximum force but differ thereafter. DLC die which shows a more pronounced peak, exhibits a steeper decrease in GN dislocation density after the maximum force. In the DLC die, the work-hardened deformation structure formed up to the maximum force can be pushed forward within a shorter stroke than in the mirror surface die. As a result, deformation is considered to stabilize rapidly after the maximum force in the DLC die. These results suggest that differences in die surface conditions affect not only the frictional state at the specimen surface but also the internal deformation state within the specimen, thereby altering the force behavior.

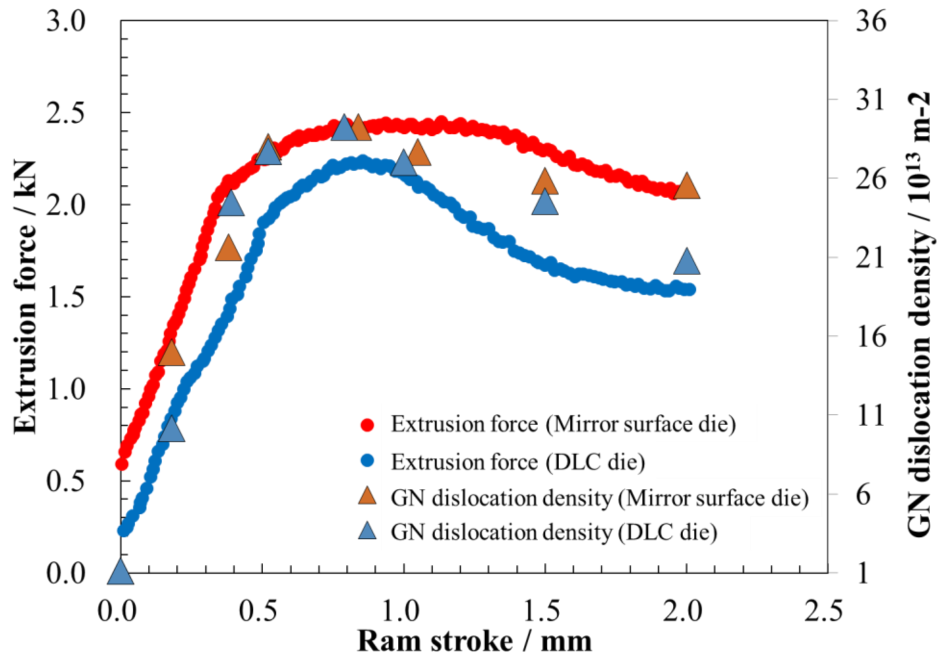

Fig. 5 Comparison between changes in geometrically necessary (GN) dislocation density with stroke progression and force behavior

Fig. 6 shows the results of GN dislocation density measurements in the tapered section around the maximum of the force-stroke curve for each die, with the region divided. The observation areas are shown in (a). As the sample passes through the tapered section, greater deformation occurs closer to the bearing section, resulting in smaller grain sizes. To capture the overall trend of the GN dislocation density, a wide field of view such as that shown in Fig. 5 can be used. However, grain size affects the analysis of GN dislocation. To obtain more accurate GN dislocation density measurements, the step size and field of view were adjusted according to the grain size in each observation area. Focusing on areas 1 to 5 near the contact surface with the die, GN dislocation density increases as the sample passes through the tapered section but shows little change in the bearing section. A similar trend is observed in areas 6 to 10 near the billet axis. However, in the bearing region, the average GN dislocation density is 22% lower in the region near the sample axis (areas 6 and 7) than in the region adjacent to the die–contact surface. (areas 1 and 2). This indicates that around the maximum force, heterogeneous deformation occurs in the radial direction, with sample following paths closer to the die contact surface experiencing greater deformation. These phenomena are common to both the mirror surface die and the DLC die, with no significant differences observed in the distribution of GN dislocation density. However, GN dislocation density is generally lower in the DLC die across most observation areas. Total dislocation density is the sum of GN dislocation density and SS dislocation density, and regions with high GN dislocation density tend to also exhibit high total dislocation density [16,17]. These results suggest that DLC coating on the die not only reduces forming force but also suppresses the increase in total dislocation density in the sample during processing. Appropriate control of die surface conditions may improve properties related to the total dislocation density of the processed product, such as fatigue resistance, stress relaxation behavior, and electrical characteristics.

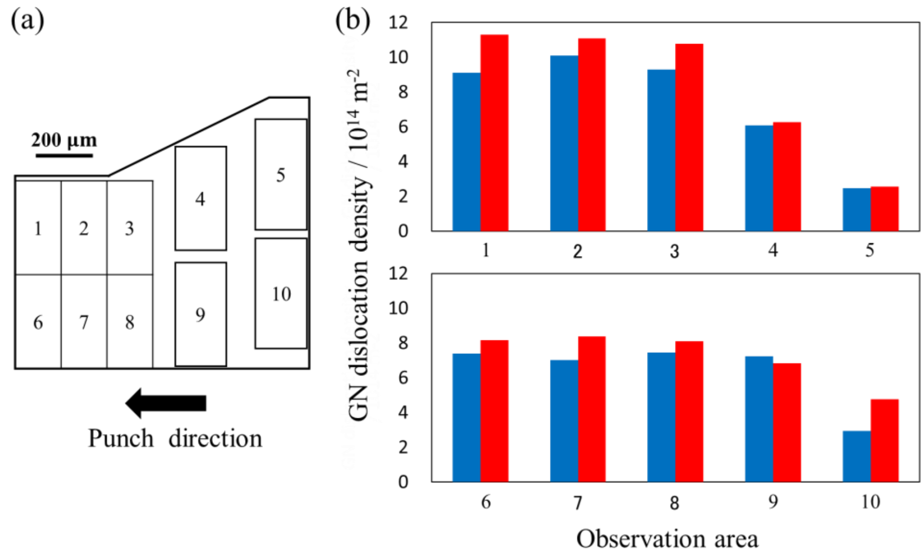

Fig. 6 (a) Schematic diagram of the observation areas, (b) GN dislocation density in each observation area

5 Conclusion

To investigate the effect of die surface conditions on microstructural changes and force behavior in micro-extrusion of pure copper, micro-extrusion tests were conducted using two types of dies with different surface finishes. Using KAM maps and GN dislocation density, the changes in the internal deformation state inside the sample with stroke progression and the influence of die surface conditions on these changes were analyzed, resulting in the following findings.

1. For both the mirror surface die and the DLC die, the GN dislocation density in the tapered section changed with stroke progression. This change reflects the formation of a work-hardened deformation structure in the tapered section and the pushing forward of this structure, suggesting that GN dislocation density enabled quantitative evaluation of the internal deformation state inside the material.

2. Changes in GN dislocation density with stroke progression corresponded well with force behavior. The DLC die, which exhibited a more pronounced force peak, showed a steeper decrease in GN dislocation density after the maximum force. In the DLC die, the work-hardened deformation structure formed up to the maximum force was pushed forward within a shorter stroke than in the mirror surface die, leading to rapid stabilization of deformation.

3. Around the maximum force in the force-stroke curve, no significant difference was observed in the distribution of GN dislocation density between the DLC-coated and mirror-finished dies. However, the absolute GN dislocation density values were lower in the DLC die than in the mirror surface die. DLC coating on the die suppressed the total dislocation density induced in the material during processing, which may improve fatigue properties, stress relaxation behavior, and electrical characteristics.

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