Contact potential difference measurement of adhesion process during micro/meso-scale injection upsetting

Takehiko Makino*, Toshinari Michimoto, Shinpei Moriyama, Tohru Kikuchi

Department of Mechanical Engineering, Nagoya Institute of Technology, Gokiso-cho, Showa-ku, Nagoya, Aichi 466-8555, Japan

Abstract

In micro/meso (between macro and micro)-scale metal forming, the contribution of friction between the material and tool on the material deformation is higher than that in macro-scale metal forming. The friction during forming strongly depends on the adhesion process. The process was studied by measuring the contact potential difference distribution of the tool surface after an injection upsetting stroke. The effect of the annealing temperature of materials and the forming speed on the amount of adhesion was quantified by the measurements.

1. Introduction

Small parts with high strength and reliability are required for creating personal devices. Such parts are expected to be increasingly produced by metal forming, which improves their mechanical properties. In micro/meso (between macro and micro)-scale metal forming, the deformation anisotropy caused by crystal plasticity and processing history is not negligible. The friction between material and tool in this scale is essentially nonuniform and nonconstant during forming. In addition, the contribution of friction on the material deformation is higher than...
that in macro-scale metal forming. The change in friction character during forming strongly depends on the adhesion process. This process is difficult to be directly observed by experiments. Therefore, an atomistic-calculation analysis of the interaction between heterogeneous atoms, performed by first-principles electronic-state calculations, has been utilized for the study of interfacial phenomena, such as adhesion during forming (Makino et al., 2011). Furthermore, surface conditions at the earlier stages of the adhesion process are required to be quantitatively measured. One of the surface properties, the work function, i.e., the minimum energy needed to remove an electron from a surface, is well known to be sensitive to atomic-scale changes at the surface. The work function has also been estimated with high accuracy by first-principles calculations. Contact potential difference (CPD) is the difference in the work function between a specimen and reference (probe) surface and can be measured using a noncontact vibrating probe in air (Kelvin–Zisman method). Zharin et al. (1998) showed that the phenomena related to rubbing surfaces can be studied by in situ CPD measurements. A macro-scale injection upsetting process was used by Nishimura et al. (1996) to estimate the adhered volume using optical images and surface roughness data. However, whether CPD measurement can be adapted to quantify a small change in the frictional adhesion formed in a micro/meso-scale injection upsetting process is yet undetermined.

The objective of this study is to quantify the adhesion process during micro/meso-scale injection upsetting by CPD measurements. In order to observe the process, an apparatus and relevant procedures for measuring CPD were developed.

2. Procedures

2.1. CPD measurement apparatus

The CPD measurement apparatus developed for this study (Fig. 1) comprises (A) a Kelvin probe with a numeric conversion part, (B) a position-controlled X-Y stage, and (C) a data recording system. These three components, each including a microcontroller, were electrically independent to reduce noise propagation and were connected by an infrared data communication system.

(A) The Kelvin probe comprises a copper cylinder (C1100) with a 1.0 mm diameter, an electric nonconduction plate, and a piezoelectric element. The probe is attached to one side of the plate, and the other side is fixed to the piezoelectric element. The probe is oscillated by a sine wave with 20 V in amplitude and 114 Hz in frequency that was applied to the piezoelectric element. This frequency was chosen so that the amplitude of the probe stays constant and the dispersion of the measured CPD values with time at a point of the specimen becomes minimum. After adjusting the gap between the probe and specimen surface, the alternating current caused by the difference between the work functions of the probe and specimen is detected as an alternating voltage through a high ohmic resistor, operational amplifiers, and filters. The electrical circuit used in this study was based on that by Klein et al. (2003). The amplitude of the alternating voltage with the variation of external voltage applied to the probe is stored and compared by the microcontroller. The external voltage that gives the weakest amplitude is identified as CPD. The numeric conversion part continuously transmits the measured CPD data to (C) using the infrared data communication system.
(B) The X-Y stage is positioned by two micrometer calipers that are rotated by two motors, which are operated by another microcontroller; each full revolution shifts the stage by 0.5 mm. This microcontroller receives the signal from (C), moves the stage, and sends a confirmation signal back to (C). When the revolution of the motor in (B) stops, the electrical current to the motor also stops to eliminate noise.

(C) The data recording part collects CPD data from (A), appends time and date information, and sends the resulting data to a PC via a cable, thrice for each point. After the completion of the measurement, the average of the last two values is used as the raw data because the first one is often measured during the movement of the X-Y stage. After sending the third data value to the PC, part (C) sends a signal to (B) to move the stage by one step.

The probe scans a 4 mm × 4 mm area of the tool surface after forming at the 0.5 mm interval. The total number of measuring points is \(8 \times 8 = 64\) for each area. The length of a side of the measuring area was determined because the diameter of the contact area at the 3 mm stroke was close to 3 mm. The dispersion of CPD values with time at a point of the specimen and each CPD value at 64 points were constantly evaluated to confirm reproducibility, as shown in Fig. 2(a). The reliability of CPD’s spatial resolution was confirmed by measuring artificially adhered aluminum on a die steel surface within the 4 mm × 4 mm area, as shown in Fig. 2(b). The high CPD areas well agreed with the adhered areas.

Fig. 2. (a) Dispersion of measured CPD values at 64 points (before forming) and (b) spatial distribution of measured raw CPD.

2.2. Data conversion

The raw value measured by the Kelvin probe is essentially an averaged value in the area directly under the probe cylinder. In this study, the raw value is analyzed by converting it to the ratio of the adhered area. The ratio indicates the size of the area occupied by the fully covered adhesion. The adhered area is assumed to show the CPD value of the bulk material’s surface. As shown in Fig. 3, the ratio of the adhered area is calculated as \((\text{raw value} - \text{background value}) / \text{bulk material’s value} - \text{background value}\). The background value is an average of the three values measured at one side of the measured area, which is outside the contact area. The bulk material’s value was measured in advance on the surface of a polished aluminum plate as 1461 mV. The adhered area’s ratio is plotted in the 0–0.2 range. The brighter contrast indicates the region with the higher amount of adhesion.

Fig. 3. Conversion of raw CPD data to ratio of adhered area.

2.3. Injection upsetting apparatus

For the measurements, a flat surface is preferable for the specimen (tool). Injection upsetting (Fig. 4(a)) is a process in which a cylindrical material is upset by being pushed through the channel inside the container against the flat tool and injected into the gap between these tools. On the flat tool surface, the material simultaneously
deforms and slides. In this study, the channel diameter is 1.0 mm, and the gap is 0.5 mm (Makino et al., 2010). The injection upsetting apparatus with the screw mechanism is shown in Fig. 4(b). The screw’s rotation makes a punch move forward through a die to press the material; the die is nonrotatable. The screw is connected to gears and rotated by a stepping motor at various speeds until the predetermined stroke is reached. Furthermore, this forming apparatus can be used as an interface direct-observation apparatus (Makino et al., 2013) by replacing the flat tool with a quartz glass plate (Fig. 4(c)).

2.4. Experimental conditions

The specimen was a die steel (SKD11) with a Rockwell C hardness of 58. The surface roughness of the specimen plate was 0.01 \( \mu \text{mRa} \). The material was commercial pure Al (A1070) with 1.0 mm in diameter and 5.0 mm in length. Both the end faces of the material were polished using an emery paper and a jig to maintain the perpendicularity. The material was annealed to examine the effect of the contact pressure at the specimen (tool) surface on the adhesion process. The materials were annealed in air by contact with a ceramic hot plate. Tensile tests for micro/meso-scale specimens (Makino et al., 2012) have shown that the yield points are 250 MPa for an as-received (as-drawn) material, 160 MPa for a material annealed at 250 °C, and 70 MPa for that annealed at 350 °C. In addition, to examine the effect of the forming speed on the quantity of adhesion, the forming speed was set to be 6 and 12 mm/min.

Two experiments were performed for each condition. The identical specimen surfaces were polished with a buff and 1 \( \mu \text{m} \) diamond spray after each measurement. After the polishing operation, an ultrasonic cleaning process using acetone was conducted. Before inserting a material in the container channel, a drop of lubricant oil with a kinematic viscosity of 460 mm\(^2\)/s was placed on the channel entrance to be applied over the material and the punch. In order to securely attach the end of the material to the specimen surface through the screw and the die, the torque needed to rotate the screw was tested by a torque wrench. After the forming process, any lubricant oil remaining on the specimen surface was removed using a cotton wool and acetone.

3. Results

3.1. Optical micrographs of specimen surface after injection upsetting

Fig. 5 shows the optical micrographs of the specimens after the strokes of the injection upsetting process. The micrographs displayed in adjacent columns are the results of the same condition. Before forming, only the contrast from carbide particles is distinguishable. After the process, it is evident that straight and sharp lines radiate from the center of the contact area of the material on the specimen surface. There is no relationship between the position of the carbide particles and that of the lines. The area in which the density of the lines is high is defined by a diameter of approximately 0.7–1.2 mm. The length of each line in the micrograph at the punch stroke of 3 mm is longer than that of 1 mm. As a result, the density of lines at the stroke of 3 mm is higher than that of 1 mm. However, there is no significant difference between the sizes of the areas in which the lines are observed in the micrographs for all the strokes and annealing temperatures. No other shapes except lines and carbide particles can
be observed in the micrographs. It was confirmed that the lines were sharply transcribed to the surface of a
deformed end face of the material after the 3 mm stroke.

![Fig. 5. Optical micrographs of specimen surface after injection upsetting.](image)

### 3.2. Spatial distribution of the adhered area’s ratio

Fig. 6 illustrates the spatial distribution of the ratio of the adhered area, measured on tool surfaces shown in Fig.
5. First, the change in the distribution with respect to stroke is examined. At the 1 mm stroke (top row), there are
no obvious differences among the conditions. In a comparison between these data and the data obtained before
forming, the former clearly indicate that the gray points are dispersed. At the 2 mm stroke (middle row), there are
brighter areas with more definite shapes except for the case of annealed materials. At the 3 mm stroke (bottom
row), there are clearly bright regions in the center of the measuring area. The bright regions in this row are slightly
larger than those in the middle row.

![Fig. 6. Spatial distribution of ratio of adhered area.](image)

Second, the change in the distribution with the annealing temperature and the forming speed is considered.
From a comparison between the results of the as-received and the annealed materials at 350 °C, the amount of
adhesion of the latter is apparently smaller at the 2 mm stroke (left). In a comparison between the results of the as-
received with a forming speed of 6 mm/min and those of 12 mm/min, the amount of adhesion of the latter is larger
at the 3 mm stroke (right). For a simple comparison, the highest values of the adhered area’s ratio in each measured
area are plotted against the stroke, as shown in Fig. 7. The averaged values for each condition are connected by
straight lines. The ratio of adhered area decreases as the annealing temperature increases. The scatter in the highest
values at the forming speed of 12 mm/min is larger for longer strokes. The highest adhered area’s ratios clearly
demonstrate the effect of the annealing temperature and forming speed.
4. Discussion

The transcription of sharp lines on the deformed material from the tool surface demonstrates that the lines are not from the material’s adhesion but scratches on the tool surface. The softer (even if hardened) material’s shape is rarely transcribed to the same material’s surface. The scratches were probably produced by oxide at the material’s end face during an early deformation stage. Although the ratio of the adhered area decreases as the annealing temperature increases, the appearance of the lines does not change with the annealing temperature. The material’s adhesion amount can be lower for lower contact pressures, which occur at higher annealing temperatures. Therefore, it is concluded that CPD characterizes the amount of adhesion. The scatter of the data with a forming speed of 12 mm/min may reflect the instability of the interaction between the material’s flow and the lubricant’s permeation behavior at the interface in the micro/meso-scale. In addition, the interfacial behavior during forming will be investigated using direct-observation experiments.

5. Conclusion

The adhesion process during micro/meso-scale metal forming can be quantified by a contact potential difference measurement. The spatial distribution of the adhered area’s ratio can be effectively used for the comparison of the results at different conditions. The highest values of the adhered area’s ratio plotted against the stroke clearly distinguish the effect of the annealing temperature and forming speed.

Acknowledgements

This work was supported by JSPS KAKENHI Grant Number 24560130.

References

Klein, U., Vollmann, W., Abatti, P., 2003. Contact potential differences measurement: Short history and experimental setup for classroom demonstration. IEEE Transaction on Education 46 (3), 338-344.
Makino, T., Dohda, K., Ishitani, A., Zhang H., 2010. Anisotropy of plastic deformation in micro/meso-scale metal forming - development of testing method -. Transaction of NAMRI/SME 37, 333-340.
Makino, T., Dohda, K., 2011. Chapter 3 - Modeling and Analysis at Micro-scales. Micro-Manufacturing, edited by Muammer Koc and Tugrul Ozel, John Wiley & Sons Inc., 43-70.
Makino, T., Suzuki, M., Fung, H., Fukui, Y., 2012. Tensile test of cylindrical aluminum after micro/meso-scale severe deformation. The proceedings of the 63th Japanese Joint Conference for the Technology of Plasticity, Fukuoka, 221-222.
Makino, T., Kawai, K., Murase, M., 2013. Analysis of frictional interface in micro/meso-scale injection upsetting. The proceedings of the 64th Japanese Joint Conference for the Technology of Plasticity, Osaka, 1-2.
Nishimura, T., Sato, T., Tada, Y., 1996. The evaluation of anti-galling characteristics by observation of adhesion morphologies using injection upsetting. Journal of Materials Processing Technology, 62, 235-241.
Zharin, A., Rigney, D., 1998. Application of the contact potential difference technique for on-line rubbing surface monitoring (review). Tribology Letters 4, 205-213.