Possibility of Applying Superplastic Forging to the Microforming of SUS304

Masahito KATOH and Yasunori TORISAKA

National Institute of Advanced Industrial Science and Technology, 1-2-1, Namiki, Tsukuba 305-8564 Japan.

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The microforming of SUS304 stainless steel with fine grain structures is carried out utilizing superplastic phenomenon. The configurations of superplastic-formed structures are studied, and possibility of superplastic microforming with SUS304 stainless steel is discussed. The microforming utilizing superplastic phenomenon successfully achieved the formation of pyramid-shaped projection with 50 μm base and 10 μm height on SUS304 specimen with ultra-fine grain structure averaging to 250 nm. It is believed that the surface roughness of the micron-sized structure, obtained in this experiment, may have reached below the Rayleigh limit for light with wavelength of at least 2 μm where the surface becomes smooth enough for infra-red (IR) device applications.

KEY WORDS: stainless steel; grain refinement; super plasticity; microforming.

1. Introduction

Various processes for the fabrication of microcomponents for microdevices have been proposed and studied in past.¹⁾ One such process known as “silicon process” is used to fabricate minute structures on the surface of silicon plates by the use of photolithography and etching. The product fabricated using the process showed high precision and functionality. However, silicon microcomponents produced with this process exhibit brittleness and poor corrosion resistance, especially in alkaline aqueous solution. These problems have now been addressed by a technology known as microforming.

Microforming is a promising technology that can produce plastic and metallic microcomponents. Microforming of plastic, glass, and metallic-glass have been studied in past.¹⁾ However, these plastic and metallic-glass components are not suitable for high-temperature applications. Compared to plastic and metallic-glass, polycrystalline metals have been found to be better suited for high-temperature applications. Metals with fine grain structures of less than 10 μm in size are recognized for their superplasticity that give the material good transfer property in working with die-structures. Therefore superplastic forming has become an attractive technique in the microforming process for microcomponents.²⁾ Yoshimura *et al.* have reported on superplastic micro-formation of Ti–4.5Al–3V–2Mo–Fe alloy with 2–3 μm in grain size using silicon die; and moreover, with this material they have also fabricated microforming die for plastic optical wave guide.³⁾

The dimension of superplastically formed microcomponent and its surface accuracy are dependent on the grain size of the work-piece where the grain size is to be further

reduced to fabricate smaller size and higher accuracy microcomponents. The authors successfully reduced the grain size of SUS304 austenitic stainless steel down to sub-micron realm by their thermomechanical treatment. And consequently, the refined SUS304 stainless steel thus obtained showed superplasticity at appropriate temperature and strain rate.^{4–6)} The thermomechanical treatment involves cold-working for strain-induced martensitic transformation, and annealing for recrystallization with a reverse transformation of martensitic phase to austenitic phase.

In this study microforming of SUS304 stainless steel with fine grain structures is carried out by utilizing the superplastic phenomenon. The configurations of the superplastic-formed structures are observed, and the possibility of the superplastic microforming with SUS304 stainless steel is discussed.

2. Experimental Procedure

A SUS304 bar (in shape of 40 mm square) of commercial purity was solution-treated for 5 min at 1 373 K. Chemical compositions of as-received SUS304 bar used are shown in **Table 1**. The solution-treated bar was cut into 50 mm long billets that were then used for thermomechanical treatment.

A thermomechanical treatment diagram for grain refinement of the billet is shown in **Fig. 1**. The thermomechanical

Table 1. Chemical composition of conventional SUS304 specimen (mass%).

C	Si	Mn	P	S	Ni	Cr	Fe
0.050	0.30	1.74	0.036	0.005	8.20	18.80	bal.

Fig. 1. Thermo-mechanical treatment for grain refinement.

treatment consisted of three steps. In the first step the billet was multi-directionally upset at 273 K to introduce strain-induced martensitic phase and was annealed at 973 K for 3 600 s for recrystallization with reverse transformation of martensitic phase to austenitic phase. It was then cooled immediately in water. Next, the obtained billet was multi-directionally upset at 273 K and was annealed at 973 K for 1 800 s and then was water-cooled again. For the third step the billet was machined into rectangular shaped bar of 15 mm width and 60 mm length and 10 mm thickness, and cold-rolled at less than 5% reduction ratio to achieve a thickness of 1 mm. Here in order to control the working heat that obstructed martensitic transformation the rolled billet was cooled in iced water for every rolling pass. The rolled billet was annealed at 973 K for 420 s. The annealed plate was then cooled in water. All annealing operations were carried out at conditions that complete austenitic transformation, which were established by previous works.⁷⁾ The microstructure of the annealed plate was investigated by transmission electron microscopy (TEM).

The working amount given by multi-directional upsetting is evaluated with integrated reduction ratio R , which is calculated by the following Eq. (1).

$$R(\%) = \frac{(1 - (A_1 \times A_2 \times \dots \times A_n) / (B_1 \times B_2 \times \dots \times B_n)) \times 100}{\dots} \quad (1)$$

Where, A_n : billet height after each upsetting,

B_n : billet height before each upsetting.

Here, integrated reduction ratio R is equivalent to the total reduction ratio in rolling process.

In all cold working processes, total reduction ratio and integrated reduction ratio, R , were 90% and austenitic phase in the billet was almost transformed to martensitic phase by cold working.⁷⁾

The obtained annealed plates were machined into specimens. Also, the solution-treated specimens with 1 mm thickness were prepared by cold rolling and by solution treatment at 1 373 K for 5 min. Both of the specimens were used for high-temperature tensile test and microforming.

To determine high-temperature tensile properties of the obtained specimens, high-temperature tensile test was made with servo hydraulic testing machine in vacuum at 973 K and at strain rate of $5.6 \times 10^{-5} - 1.0 \times 10^{-3} \text{ s}^{-1}$. The prepared specimens were JIS 7 type as shown in Fig. 2. The tensile

Fig. 2. Dimension of the tensile test specimen.

Fig. 3. SEM image of the Vickers indentation as the die for microforming.

 Fig. 4. Mounting of the sample with die. Dimension of samples were $\phi 4 \text{ mm} \times 1 \text{ mm}$.

specimen was set into a testing machine and was heated up to the testing temperature by high-frequency induction heating.

Specimens for microforming were made in shape of discs with 4 mm diameter and 1 mm thickness. The specimens were grinded by emery paper and polished to a mirror finish on one side.

An Inconel die with 13 mm diameter and 7 mm thickness was used in the microforming experiment. One flat surface of the die was grinded by emery paper and then polished to mirror finish by buffing. An indentation to serve as microforming die was produced with Vickers hardness tester onto the mirrored surface of the Inconel die. The produced indentation was concave pyramid with square base with $50 \mu\text{m}$ side and $10 \mu\text{m}$ depth. SEM image of the indentation is shown in Fig. 3. The Vickers hardness, HV , of the die was approximately 410.

Fig. 5. TEM micrograph of sample after the thermo-mechanical treatment as Fig. 1.

Fig. 6. Optical micrograph of solution treated specimen.

The Inconel die and the specimen were set into a die-set as shown in Fig. 4. In the die-set, the die was placed on a lower holder and was secured by an upper holder. The specimen was put onto the die and was pressed using a punch. Both mirrored surfaces of the die and the specimen were in contact with each other. A small amount of high-vacuum silicone grease (Dow Corning Co. Ltd.) was thinly coated on the mirrored surface of the specimen to serve as lubricant. The die-set was then subjected to a servo hydraulic testing machine used in high-temperature tensile test. The specimen was pressed at 2 000 N in vacuum and the die-set was heated up to 973 K by high-frequency heating. The applied pressure and temperature were maintained at these values for 30 min to complete the microforming. Then the specimen was removed from the die, and the worked surface was observed by scanning electron microscope (SEM).

3. Experimental Results and Discussion

A TEM micrograph of the annealed specimen is shown in Fig. 5, and an optical micrograph of the solution-treated specimen is shown in Fig. 6. The grain structure of the annealed specimen is ultra-fine with the mean grain size of 250 nm. The solution-treated specimen has coarse grain structure with approximately 20–50 μm in grain size.

2 μ m

Fig. 9. Expanded SEM image of area B in Fig. 8(a).

rough surfaces shown by areas A and B respectively. The area A was qualitatively analyzed by energy-dispersed spectroscopy where it was found that the surface of the pyramid-shaped projection was composed of oxygen and silicon elements. It appears that these elements detected had originated from the silicone grease used as lubricant in microforming. A highly-magnified image of the area B is shown in Fig. 9. In the area B, the surface is observed to consist of particles with approximately 200–300 nm diameters, and those particle sizes are almost same as the grain size of the annealed specimen as shown in Fig. 5. It is believed that crystal grains in annealed specimen moved independently by grain boundary slippage in superplastic deformation, and the surface structure as shown in area B was formed with the grains' movement.

As mentioned earlier the solution-treated specimen could not be embossed in this experiment, whereas the annealed specimen was completely done into the pyramid shape. The force of 2000 N was applied to the 4-mm diameter specimen when microforming and the average pressure was approximately 159 MPa. This pressure is lower than the tensile strength of the solution-treated material, but is higher than the 0.2% proof stress. Therefore the solution-treated specimen has a likelihood of undergoing further deformation if the applied forming pressure increases. However, it was realized that the used Inconel die was slightly damaged after the microforming of the solution-treated specimen. So it is believed that the applied forming pressure could not be further increased in this experiment.

The above results show that we could achieve the microforming of SUS304 stainless steel successfully by utilizing the superplastic phenomenon. In this experiment the pyramid-shaped structure with 50 μ m base and 10 μ m height was formed using the specimen with a mean grain size of 250 nm. It is believed that fabrication of micron-sized structures with superplastic microforming is doable at SUS304. The example of the structures are projections with size less than several tens of micrometer or gratings with intervals of less than several tens of micrometer. However the size limitation of the superplastic-formed structures should be addressed in future works. Furthermore, the micron-sized structure obtained in this experiment may have surface roughness within Rayleigh limit in relation to the wavelength of light with at least 2 μ m, and it appeared that the surface is smooth enough to be applicable for optical devices utilizing infrared light.

4. Conclusion

Microforming of SUS304 stainless steel was carried out. Microforming utilizing superplastic phenomenon successfully achieved forming pyramid-shaped projection with 50 μ m base and 10 μ m height on the SUS304 specimen with ultra-fine grain structure of 250 nm average. It is believed that superplastic microforming can be employed to form micron-sized structure of sub-0.1 μ m with the surface roughness of less than grain size.

The micron-sized structure obtained in this experiment may have surface roughness within Rayleigh limit in relation to the wavelength of light with at least 2 μ m, and it is believed that the surface is smooth enough to be applicable in optical devices utilizing infrared light.

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