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Grain size effect on mechanical behavior of thin pure titanium foils at elevated temperatures

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ABSTRACT

To contribute to process design of microforming at elevated temperatures, the effect of grain size on mechanical behavior of pure titanium foils was studied. Uniaxial tensile tests and microbending tests were performed at different temperatures of 298, 433, 573, and 723 K and at different strain rates of 10^{-3}, 10^{-2}, and 10^{-1} s^{-1}. Pure titanium (Ti) foils with various grain sizes of 2.7–42.4 µm were used. As results, flow stress of Ti foils with a larger grain size showed stronger dependency on the temperature and strain rate conditions. In particular, both the reduction rate of the yield stress and that of the ultimate tensile strength relative to that at 298 K increased with increasing grain size at higher strain rates of 10^{-2} and 10^{-1} s^{-1}. However, only at a higher temperature and at a lower strain rate of 10^{-3} s^{-1}, the flow stress increased for the foils with larger grain sizes. This is attributed to the dynamic strain aging, which is known as hardening behavior due to the diffusion of additional inclusion under higher temperatures. Furthermore, in microbending tests, the reduction rate of springback angle by increasing temperature also increased with increasing grain size. This is attributed to the increased reduction of strain gradient of the foils with a larger grain size. To clarify the mechanism of grain size effect on the mechanical behavior of pure Ti foils deformed at high strain rates, a composite constitutive model involving statistically stored dislocations (SSDs) and geometrically necessary dislocations (GNDs) was built with the capability of being employed at elevated temperatures. The effect of strain gradient on the material behavior was discussed in terms of its relationship with the density of GNDs.

1. Introduction

With the trend of miniaturization, microparts are now widely used in micro-electromechanical systems (MEMS) such as micro springs for electronics [1], microneedles for drug delivery [2], and bipolar plates for micro fuel cells [3]. Pure titanium (Ti) is often used for the manufacture of microparts in biomedical devices and implants because of its light weight, biocompatibility, and outstanding corrosion resistance [4]. Owing to its high productivity, and the near-net-shape and good properties of the formed products, microforming has been received considerable attention in the manufacture of microparts [5]. However, with increasing miniaturization, the material flow becomes inhomogeneous and the process becomes unpredictable [6] owing to the occurrence of size effects [7].

There are two main types of size effect existing in metallic materials: the specimen size effect and grain size effect [8]. To contribute to the design of microforming processes, several theoretical models have been developed. On one hand, to explain the phenomenon of “the smaller of the size, the weaker of the material” at microscale, such as thinner foils exhibiting lower flow stress [9], the surface layer model was proposed [1]. To simulate the forming behavior of microparts as well as the scattering of the process parameters, Geiβdörfer et al. [10] proposed a mesoscopic model. Lai et al. [11] and Peng et al. [12] simplified this model, and successfully predicted the flow stress of a material for different thicknesses. On the other hand, to explain the phenomenon of “the smaller of the size, the stronger of the material”, such as thinner foils exhibiting larger springback in microbending, strain gradient theory was proposed by Nix and Gao [13] and Fleck and Hutchinson [14], and simplified by Huang et al. [15]. By utilizing the model proposed by Huang et al. [15], Li et al. [16] successfully predicted the springback angles for pure aluminum foils with different thicknesses by both theoretical analysis and finite element (FE) analysis. Geometrically necessary dislocations (GNDs) are thought to be associated with strain gradients since they accumulate and accommodate the lattice curvature that arises whenever there is non-uniform plastic deformation [17]. To further enhance the understanding of the mechanical behavior of the materials in microforming, crystal plasticity (CP) model considering the orientation and distribution of the grain size was also used [18]. By the consider-

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ation of grain heterogeneity and using CP constitutive model, Yoshida [19] found that the surface roughness became larger with increasing ratio of grain size to thickness. The study of Özdemir [20] indicated that the scatter due to random grain orientations diminished as the ratio of grain size to thickness decreased.

In addition to the development of theoretical models to explain and simulate the size effects of materials, the approach of conducting a microforming process under heating [21] has also been suggested to minish the size effects since the material flow becomes more homogeneous at elevated temperatures [22]. Moreover, resistance heating (RH) is an effective approach to increasing the heating rate and to simplifying the experimental equipment [23, 24]. By using different RH-assisted microforming systems, microforming processes, such as micro deep drawing [25] and microbending [26], were conducted with pure Ti foils at elevated temperatures. As the temperature increased, the material formability was found to increase [25] and the springback angle was found to decrease [26]. To provide the engineering community with an evaluation of useful predictive models to assist the characterization of materials in RH-assisted microforming, Magargee et al. [27] studied the effect of an electric current on material deformation and used modified Hollomon and Johnson–Cook models to predict the magnitude of stress reduction caused by an electric current and the associated temperature increased by Joule heating. To design an effective microforming process assisted by RH, the authors [28] predicted the flow stress for a material considering not only the effect of an electric current but also the effect of the strain rate on the material properties.

However, the RH-assisted method cannot eliminate size effects. Siopis and Kinsey [29] investigated the effects of the grain size and current density on annealed pure copper during electrically assisted forming and found that the reduction of the flow stress decreased with increasing grain size. Fan et al. [30] studied the effect of the grain size and grain boundaries on the behavior of brass under electrically assisted deformation during tensile tests. The results indicated that decreasing the material grain size caused an increase in the reduction rate of the flow stress. Regarding the related research on pure Ti, by conducting RH-assisted microbending tests using foils with different thicknesses, the authors [31] found that thinner foils exhibited less springback than thicker ones at high temperatures, whereas the opposite tendency occurred at room temperature. In addition, by conducting RH-assisted tensile tests at a strain rate of 10^{-3} s^{-1}, the reduction of the stress at elevated temperatures was found to be higher for the foils with larger grain sizes [33]. Although the specimen size effect on the springback of pure Ti foils in RH-assisted microbending has been confirmed to be dominated by the ratio of surface area [32], the mechanism of grain size effect on mechanical properties of the material has not yet been clarified.

The aim of this study was to clarify the mechanism of grain size effect on mechanical behavior of thin pure Ti foils at elevated temperatures and various forming strain rates. To achieve this, RH-assisted uniaxial tensile tests and microbending tests were conducted at different temperatures of 298, 433, 573, and 723 K using 0.05 mm-thick pure titanium (Ti) foils with various grain sizes of 2.7–42.4 μm. The effect of the grain size on the tensile properties and springback behavior was investigated. In particular, to figure out the mechanism of grain size effect on material deformation at elevated temperatures with a higher strain rate, a composite constitutive model involving statistically stored dislocations (SSDs) and geometrically necessary dislocations (GNDs) was built, which is expected to contribute to the design of microforming processes assisted by RH.

2. Experiments

2.1. Materials

Thin pure Ti foils with a thickness of 0.05 mm (JIS TR270C-H) were used. To obtain specimens with different grain sizes, the foils were annealed at different temperatures with different holding time under a controlled atmosphere of argon gas. Table 1 shows the annealing conditions and the corresponding grain sizes of the foils. Fig. 1 shows images of the microstructures (rolling direction-normal direction (RD-ND) plane) of the annealed foils taken with a scanning electron microscope (SEM; HITACHI, SU-70) after ion milling (HITACHI, IM-4000). As can be seen from Fig. 1, Ti foils with different grain sizes were successfully obtained by controlling the annealing temperature and holding time.

Table 1
Annealing conditions and grain sizes of thin pure Ti foils.

Annealing temperature	773 K	823 K		923 K	
Heating time	15 min	20 min		20 min	
Holding time	1 min	10 min	10 min	30 min	60 min
Grain size (*d*)	2.7 μm	4.5 μm	14.7 μm	24.5 μm	42.4 μm

2.2. Tensile tests

The tensile specimens were cut by machining along the rolling direction. The dimensions of the tensile specimens were the same as those in Ref. [28]: the gauge length was 16 mm while the gauge width was 4 mm. All specimens were polished to remove major burrs to prevent fracture at undesired locations. Tensile tests were performed at temperatures of 298, 433, 573, and 723 K and at different strain rates (from 10^{-3} s^{-1} to 10^{-1} s^{-1}) by using a tensile testing system incorporating RH method. Details of this system can be found in Ref. [28]. The tests were conducted three times under each condition to ensure repeatability.

2.3. Microbending tests

The width of bending specimens was 5 mm while the length was 10 mm. Micro air bending tests were conducted on the annealed foils at temperatures of 298, 433, 573, and 723 K along the rolling direction by using a RH-assisted microbending system. Fig. 2 shows the illustration of the tests. The die used as the electrode was connected with the power supply (see Fig. 2(a)). The power supply was turned on when the punch moved down and contacted with the blank as shown in Fig. 2(b). After the temperature at the center of the blank increased to the forming temperature (see Fig. 2(c)), the punch moved down and the bending test was conducted (see Fig. 2(d)). Finally, the power supply was turned off as soon as the punch moved up as shown in Fig. 2(e). Fig. 2(f) shows the definition of springback angle. More details of the microbending system can be found in the previous study [31, 32]. The radius of the punch and die was 1.25 mm while the clearance between the punch and die was 0.1 mm. The velocity of the punch was 10 mm/s. To ensure repeatability, microbending tests were conducted five times under each condition.

3. Experimental results

The effect of the grain size on the tensile properties and springback behavior of thin pure Ti foils at elevated temperatures are presented in Sections 3.1 and 3.2, respectively.

3.1. Tensile properties

3.1.1. True stress–strain curves

Fig. 3 shows the true stress–strain curves of the annealed foils at a strain rate of 10^{-3} s^{-1} for grain sizes of 4.5 and 42.4 μm. Fig. 4 shows the true stress–strain curves of the annealed foils at different strain rates for grain sizes of 2.7, 14.7, and 24.5 μm. Owing to the decrease in the strength of the material after annealing, the flow stress decreases with increasing grain size for a given strain rate and temperature [34]. For example, at the strain rate of 10^{-3} s^{-1} and the temperature of 573 K, when the true strain is 0.1, the flow stresses of the material are 252.4,