Production of Superplastic Ti–6Al–7Nb Alloy Using High-Pressure Sliding Process*1

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A Ti–6Al–7Nb alloy was processed by severe plastic deformation through high-pressure sliding (HPS) at room temperature for grain refinement. The microstructure consists of grains with sizes of 200–300 nm in diameter having high and low angles boundaries. Superplasticity appeared with the total elongation of more than 400% and this was more likely when the tensile specimen is deformed in the direction parallel than perpendicular to the sliding direction. However, the superplastic elongation is almost the same irrespective of whether the sliding was made in the single direction or in the reversible directions as far as the total sliding distance is the same. The total elongation is invariably higher for the tensile testing at 1123 K than at the other temperatures, reaching the highest elongation of 790% at the initial strain rate of 1 × 10⁻³ s⁻¹. The strain rate sensitivity and the activation energy for the deformation were determined to be more than ~0.3 and 199 kJ/mol, respectively. The microstructural observation reveals that the α phase region covers more than 85% of the tensile specimens after deformation and their grains are equixed in shape. It is concluded that the superplastic deformation is mainly controlled by grain boundary sliding through thermally activation by lattice diffusion. [doi:10.2320/matertrans.ME201924]

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1. Introduction

Ti–6Al–7Nb alloy is well recognized as a good candidate for biomedical application because the biocompatibility of the alloy is superior to a conventional Ti–6Al–4V alloy due to the replacement with non toxic element Nb for toxic element V.¹–³) The Ti–6Al–7Nb alloy is a two-phase alloy consisting of an α phase with the hexagonal closed packed (hcp) structure and a β phase with the body centered cubic (bcc) structure. Because the alloy exhibits excellent mechanical strength as the Ti–6Al–4V alloy,⁴ it is also desired that the Ti–6Al–7Nb alloy has good formability.

Superplasticity is often used to improve the formability in particular of hard-to-deform materials. The superplasticity is a phenomenon in which a massive deformation of 400% or more is achieved without necking under lower pressures when the material undergoes tensile deformation at elevated temperature.⁵⁻⁹) For the advent of the superplasticity, it is important that the grain size should be small enough to increase the contribution of the grain boundary sliding to the total deformation.¹⁰⁻¹⁰)

In order to achieve a fine grained structure, several processes of severe plastic deformation (SPD) are available, where intense strain is introduced to a sample.¹¹,¹²) Because a high density of dislocations are generated, grain refinement occurs through combination of dislocation generation and annihilation.¹³) The major SPD processes include equal-channel angular pressing (ECAP),¹⁴) accumulative roll bonding (ARB),¹⁵) and high-pressure torsion (HPT).¹⁶) Among them, the HPT process is effective for high-strength and low-ductility materials due to a strong constrained condition under high pressure. Thus, the application of the HPT process to a Ti–6Al–7Nb alloy led to an advent of superplasticity.¹⁷) However, there are few reports on the superplasticity of the Ti–6Al–7Nb alloy,¹⁸) and furthermore, with the HPT method, there is a problem in that the sample shape is limited to disks and rings. Therefore, a new SPD process was developed, called the high-pressure sliding (HPS) where the processing of sheet- and rod-shaped samples is possible under high pressure.¹⁹⁻²⁴) Figure 1 shows a schematic of the HPS processing. Samples are placed between two anvils and a plunger. As the plunger is pushed under application of pressure, the shear strain is introduced in the sample, and this leads to grain refinement of the sample.

The Von Mises equivalent strain εeq introduced using the HPS method is given by the following equation.¹⁹)

![Fig. 1 Schematic illustration of high-pressure sliding (HPS).](image-url)
where \( x \) is the sliding distance and \( t \) is the thickness of the sample. As known from this equation, the amount of the strain introduced can be controlled based on the sliding distance as well as the sample thickness.

Recently, the HPS facility was scaled up so that the grain refinement was feasible in high-strength aluminum alloys such as A2024 and A7075 and Ni-based superalloy Inconel718, and it was demonstrated that superplasticity was attained by tensile testing at elevated temperature.\(^{24,25} \)

We also showed that the superplasticity appeared in the present Ti–6Al–7Nb alloy at a testing temperature of 1073 K with an initial strain rate of \( 2 \times 10^{-3} \text{ s}^{-1} \). In this study, the Ti–6Al–7Nb alloy is deformed at various temperatures and strain rates, and the optimum conditions including the mechanism for the advent of superplasticity is examined.

2. Experiment Procedures

A commercially available Ti–6Al–7Nb (mass\%) alloy in a form of a round rod was cut to sheets with 100 mm length, 10 mm width and 1mm thickness for HPS samples as shown in Fig. 2. The conditions for HPS processing were the applied pressure of \( P = 3 \text{ GPa} \), sliding distances of \( x = 5, 10, 15 \) and 20 mm, sliding speed of \( v = 1 \text{ mm/s} \), and processing temperature at room temperature. In this study, the samples processed through \( x = 10 \text{ mm} \) were mostly used for the examination of the superplastic behavior, but in addition, the samples processed through \( x = 20 \text{ mm} \) in a single way and \( x = 20 \text{ mm} \) with one reciprocating motion with each way of \( x = 10 \text{ mm} \). Vickers microhardness was measured under a load of 0.5 kg for dwell time of 15 s throughout the cross sections at positions \( L = 10, 30, 50, 70, \) and 90 mm, where \( L \) is defined in Fig. 2 as the distance from the front edge. Figure 2 also shows the sampling locations along the HPS-processed sample with the directions of tensile axis. Tensile specimens with the dimensions shown in Fig. 2 were cut from the HPS-processed sample where the tensile axis is parallel to the sliding direction at positions of \( L = 10, 30, 50, 70 \) and 90 mm, and is perpendicular to the sliding direction at positions of \( L = 20, 40, 60 \) and 80 mm. For both cases, the tensile specimens were extracted from the center of the width. Tensile tests were conducted at a selected temperature out of \( T = 1023, 1073 \) and 1123 K, and with an initial strain rate in the range of \( \dot{\varepsilon} = 5 \times 10^{-4} \) to \( 1 \times 10^{-5} \text{ s}^{-1} \). For microstructural observations, 3 mm diameter disks were cut from the center of the width in the HPS-processed sample at \( L = 50 \text{ mm} \). Each disk was thinned to the thickness of 0.1 mm from the center of the specimen at \( L = 50 \text{ mm} \). The surfaces were finished with electropolishing at 25V using an electrolyte of 10% HClO\(_4\) + 90% CH\(_3\)COOH at 10–15°C. The surfaces were observed using a Hitachi H7100 operating at an acceleration voltage of 100 kV. After tensile testing, the gauge area was polished to a mirror surface with buffing, and the surface was finished with electropolishing at 25 V using an electrolyte of 10% HClO\(_4\) + 90% CH\(_3\)COOH at 10–15°C. The surfaces were observed using a Hitachi scanning electron microscope (SEM), SU6600, and the crystal orientation was analyzed using a TSL electron backscatter diffraction (EBSD) analyzer.

3. Results

Figure 3 shows results of the hardness measurements after HPS processing for \( x = 10 \text{ mm} \). When compared to the hardness level of the as-received sample (i.e., 310 Hv), the hardness after the HPS processing increased to 340 Hv, which is constant across the entire length of the sample. This indicates that strain was uniformly introduced throughout the sample by the HPS processing.

Figure 4 shows TEM micrographs after the HPS processing, where (a) is a bright-field image and (b) is a dark field image. This dark-field image was taken using a diffracted beam indicated by the arrow in the selected area electron diffraction (SAED) pattern in the inset of (b). The areas corresponding to bright contrasts indicated by the arrows in the dark-field image represent grains with the same orientations. This analysis reveals that the grain size is refined to 200–300 nm. The grain boundaries are ill defined, which is a unique feature processed by an SPD process.

Figure 5 shows results after tensile testing of the specimens extracted in the direction (a) parallel (longitudinal),
and (b) perpendicular (traverse) to the sliding direction. For both cases, nominal stress-elongation curves are displayed on the upper side and the specimens after deformation to failure on the bottom side. For comparison, undeformed and as-received specimens after tensile testing are also included. The maximum flow stress of the as-received specimen was 120 MPa, but after the HPS processing, the maximum stress decreased to 90–60 MPa in the specimens extracted in the longitudinal direction and to 80 MPa in the traverse direction. Elongation to failure was 240% for the as-received specimen, whereas after the HPS processing, it exceeded 400% at \( L = 10, 30, 50 \) and 70 mm in the specimens extracted in the longitudinal direction, demonstrating the advent of superplasticity. The appearance of the tested specimens confirms that the superplastic deformation occurred uniformly throughout the entire length of the specimens. Inspection of the specimens extracted in the traverse direction reveals that superplasticity of 400% or more was achieved only at \( L = 20 \) mm, whereas it was below 400% at \( L = 40, 60, \) and 80 mm. In other words, superplasticity occurred more likely

**Fig. 4** (a) TEM bright-field image, (b) TEM dark-field image with SAED pattern after HPS processing for \( x = 10 \) mm. Arrow indicates diffracted beam for dark-field image.

![Fig. 4](image)

![Fig. 5](image)

**Fig. 5** Stress-strain curves (upper) and appearance of tensile specimens (lower) after deformation to failure including undeformed specimen (lower), where tensile specimens were extracted at distances of \( L \) along (a) longitudinal direction and (b) transverse direction.
in the specimens extracted in the longitudinal direction than in the transverse direction; thus, this comparison suggests that anisotropy exists in the HPS-processed samples.

Figure 6 plots the total elongation to failure as a function of the sliding distances where the tensile specimens were extracted at the center of the HPS-processed sample ($L = 50 \text{ mm}$). When the sliding distance exceeds 10 mm, the total elongation to failure becomes saturated. It is concluded that the elongation to failure does not increase with the sliding distance but it remains constant.

Figure 7 shows results of the tensile testing for samples processed through (a) $x = 20 \text{ mm}$ in a single direction and (b) $x = 10 \text{ mm}$ with one reciprocal motion. For both cases, as in Fig. 5, nominal stress-elongation curves are displayed on the upper side and the specimens after deformation to failure on the bottom side. For comparison, undeformed and as-received specimens after tensile testing are also included. In the sample processed through the reciprocal motion, the total elongation of 630% was achieved at $L = 10 \text{ mm}$; however, there is little difference between the samples processed in the single direction and those processed through the reciprocal motion. The appearance of the specimens after the tensile testing supports the advent of superplasticity where the uniform deformation occurs throughout the entire length of the tensile specimens.

Figure 8 shows results of the tensile testing performed with an initial strain rate of $5 \times 10^{-4} \text{s}^{-1}$–$1 \times 10^{-2} \text{s}^{-1}$ at (a) 1023, (b) 1073, (c) and 1123 K, for specimens extracted at around $L = 50 \text{ mm}$. In all cases, nominal stress-elongation curves are delineated on the upper side and the appearance of the tensile specimens are displayed on the lower side. For reference, an undeformed specimen is included. As the testing temperature is higher, the elongation to failure increases and reaches a maximum value of 790% at 1123 K with $1 \times 10^{-3} \text{s}^{-1}$. It is confirmed from the nominal stress-
 elongation curves and appearance of the tested specimens that uniform deformation occurs throughout the gauge length when superplasticity appears.

Figure 9 summarizes the elongation to failure at each testing temperature as a function of the initial strain rate. At all testing temperatures, if the initial strain rate is decreased, the elongation to failure increases and then reaches maximum values followed by decrease with a further decrease in the strain rate. The reason for the presence of the maxima in the total elongation is discussed in the following section.

4. Discussions

The strain rate $\dot{\varepsilon}$ in high-temperature deformation is generally expressed through the following equation:

$$\dot{\varepsilon} = A \left( \frac{b}{d} \right)^P \left( \frac{\sigma}{G} \right)^n \exp \left( - \frac{Q}{RT} \right) \text{ (s}^{-1})$$

where $A$ is a constant, $b$ is the Burgers vector, $d$ is the grain size, $\sigma$ is the flow stress, $G$ is the shear modulus, $Q$ is the activation energy for deformation, $R$ is the gas constant, $T$ is the testing temperature, $p$ is the grain size exponent, and $n$ is the stress exponent.

To examine the strain rate sensitivity ($m$ value ($= 1/n$)) under the condition where the superplasticity appears, Fig. 10 shows a double logarithmic plot of the maximum flow stress against the initial strain rate. The value of $m$ is obtained from the slope of this graph. Close analysis reveals that when the superplasticity appears, $m$ takes a value of 0.3 at 1023 K and 1073 K, and 0.4 to 0.5 at 1123 K. These values of $m$ meet the
condition where the superplasticity appears due to grain boundary sliding.\(^{26}\)

The activation energy during superplastic deformation was determined as follows: First, the initial strain rates which correspond to the flow stress at 100 MPa but where superplastic flow appears with \(m \geq 0.3\) are obtained by calculating the best fit line using a least-squares method for each temperature. Then, the initial strain rates so obtained are plotted against the reciprocal temperature in Fig. 11 (so called in the Arrhenius relation). There is a good linear relationship and the slope of this graph represents \((-Q/R)\), and the activation energy was determined to be 199 kJ/mol. Because the activation energy for the lattice self-diffusion in \(\alpha\)-Ti is 204 kJ/mol,\(^{27}\) this determination suggests that the superplastic deformation is controlled by the lattice diffusion.

Figure 12 shows SEM images near the fracture tips after tensile deformation to failure with initial strain rates of (a) \(1 \times 10^{-2}\) s\(^{-1}\), (b) \(1 \times 10^{-3}\) s\(^{-1}\) and (c) \(5 \times 10^{-4}\) s\(^{-1}\) at 1123 K. The bright contrast areas correspond to the \(\alpha\) phase. The grain sizes of the \(\alpha\) phase measured using EBSD were 2.6, 4.5 and 5.9 \(\mu\)m, respectively. The measurement indicates that grain coarsening occurs as the initial strain rate decreases. However, because the grains were not elongated in the deformation direction (which is the horizontal direction in the figure), it is concluded that grain coarsening as well as the grain boundary sliding contributes to superplastic elongation.

Finally, let us discuss the reason why maxima appear in the plots of elongation to failure against initial strain rate as observed in Fig. 9. Grain boundary sliding responsible for superplasticity occurs more likely when the initial strain rate decreases. However, the slower strain rate gives a longer time of exposure to high temperatures. Thus, as shown in Fig. 12, grain coarsening occurs during tensile testing. Because the density of grain boundary decreases due to the grain coarsening, the fraction that the grain boundary sliding contributes to the overall deformation decreases, and thus this in turn decreases the total elongation to failure. Therefore, the maxima appear due to the balancing of these two factors.

5. Summary and Conclusions

(1) A Ti–6Al–7Nb alloy was severely deformed using a process of high-pressure sliding (HPS). The grain size including high- and low-angle grain boundaries was refined to 200–300 nm.

(2) The HPS-processed samples were subjected to tensile testing at a selected temperature in the rage of 1023–1123 K and superplastic elongation more than 400% was attained. Optimization of the HPS condition led to a maximum elongation of 790% at 1123 K with an initial strain rate of \(1 \times 10^{-3}\) s\(^{-1}\).

(3) The total elongation to failure was larger when tensile tests are conducted in the tensile direction parallel to the sliding direction than perpendicular to the sliding direction. It was then suggested that the anisotropy developed for the advent of superplasticity. However,
the superplastic elongation remains the same even the sliding distance extended twice or the sliding direction reversed as long as the sliding distance is more than 10 mm.

(4) The strain rate sensitivity was measured to be more than $\sim 0.3$ for the initial strain rates in the range of $5 \times 10^{-4} - 1 \times 10^{-2} \text{s}^{-1}$ and for the testing temperatures in the range of $1023 - 1123 \text{K}$. The activation energy for the superplastic deformation was determined to be 199 kJ/mol which was almost the same as the activation energy for lattice diffusion. Observations by scanning electron microscopy revealed that grain growth occurred during superplastic deformation but the grains remain equiaxed, suggesting that the grain boundary sliding occurs.

(5) It was thus found that the HPS-processed Ti-6Al-7Nb alloy exhibited superplasticity which was controlled by grain boundary sliding through lattice diffusion.

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