Microstructural Mechanisms during Dynamic Globularization of Ti-6Al-4V Alloy

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The microstructural mechanisms during dynamic globularization were investigated for ELI grade Ti-6Al-4V alloy with initial martensite microstructure. For this purpose, compression tests were carried out isothermally at 1073 K up to the strains of 1.0 and 1.4 with the strain rates ranging from $10^{-3}$ s$^{-1}$ to 1 s$^{-1}$. Fully dynamically globularized specimen exhibited higher Vickers hardness values due to the finer grain size and higher dislocation density compared to the conventionally produced one, i.e., partial dynamic globularization by subsequent annealing. The strain rate sensitivity ($m$) values of the flow stresses at initial stage ($v \approx 0.05$) and final stage ($v \approx 1.4$) of deformation were $0.15$ and $0.36$, respectively, leading to the conclusion that deformation was controlled by dislocation glide/climb at low strains and grain boundary sliding at high strains. Also, microstructural evolution during dynamic globularization was rationalized by examining the microstructures associated with kinking and fragmentation of lamellar plates. [doi:10.2320/matertrans.L-MRA2008832]

(Received December 26, 2007; Accepted July 1, 2008; Published September 18, 2008)

Keywords: dynamic globularization, microstructural mechanism, titanium-6 Al-4V, martensite

1. Introduction

The two phase Ti-6Al-4V alloy is widely used as an aerospace material due to its high specific strength and good corrosion resistance.1 Generally, the processing of this alloy consists of a series of hot working and heat treatment steps each of which has specific microstructural targets. Ingot breakdown is usually conducted above the beta transus temperature (temperature at which single beta phase $\rightarrow$ alpha phase + beta phase) to produce homogeneous wrought material, resulting in transformed microstructure of which morphology is significantly influenced by the cooling rate. Then, dynamic globularization with subsequent annealing is employed below the beta transus temperature to produce equiaxed alpha + beta microstructure from the transformed beta microstructure.2 Because dynamic globularization is essential to obtain highly formable materials suitable for secondary hot working processes such as forging, extrusion and superplastic forming, many works have been carried out to investigate the microstructural effect as well as transformation kinetics. Shell and Semiatin3) investigated the effect of the alpha plate thickness on the fraction globularized in the three kinds of transformed microstructures which have the different lamellar thickness. They showed that the fraction globularized increases with decreasing the alpha plate thickness. Seshacharyulu et al.4) and Semiatin et al.5) suggested that dynamic globularization of lamellar microstructure (with intermediate lamellar thickness) at relatively low strains ($< 0.8$) is controlled by cross slip and glide/climb of dislocations. So far, most of the earlier works focused on the conventional processing method inducing lamellar microstructures with intermediate and/or thick lamellar thickness. Accordingly, the resulting microstructures were partially globularized microstructure.

Considering the fact that the thinner the alpha platelet thickness, the finer the equiaxed grains after the dynamic globularization, it is worthwhile to studying the dynamic globularization behavior of Ti-6Al-4V alloy using martensite microstructure. Therefore, our aim is to investigate the dynamic globularization behavior at large strains and related microstructural mechanisms using martensite microstructure.

2. Experimental Procedure

The material used in the present work was supplied by President Titanium as bar stock ($\phi = 105$ mm $\times$ L = 350 mm) of ELI grade Ti-6Al-4V alloy. The as-received bar had an equiaxed microstructure with alpha grain size of $\approx 13$ µm (Fig. 1(a)) and a chemical composition (in weight percent) of 6.04 Al, 4.21 V, 0.13 Fe, 0.11 O, 0.016 C and 0.005 N with the balance titanium. The material was beta-solution treated at 1323 K (beta-transus temperature: $\approx 1250$ K) for 2 h and water quenched to obtain the initial martensite microstructure with approximately 400-µm-prior beta grains and 0.3-µm-thick laths (Fig. 1(b)).

To analyze the microstructural mechanisms of dynamic globularization of the martensite microstructure, compression tests were performed by Gleeble 3500. For this purpose, the cylindrical samples ($\phi = 8$ mm $\times$ L = 12 mm) were machined and heated to the test temperature in the vacuum environment, holding 2 minutes to ensure thermal equilibrium. Tests were carried out at 1073 K up to the strain of 1.4 by varying strain rates from $10^{-3}$ s$^{-1}$ to 1 s$^{-1}$. And, according to previous researches, the volume fraction of alpha phase and thickness of alpha lath is about 0.83 and 0.4 µm, respectively, at 1088 K.3,6) The additional samples with equiaxed microstructure were prepared via the conventional processing method shown in Fig. 2, i.e., partial dynamic globularization and additional annealing, to provide an insight into the microstructural differences from fully dynamically globularized specimen. Also, Vickers hardness was measured by a microindentor (Future-Tech FM-700) with a load of 3 N for 10 s.

Microstructures were examined via optical, scanning-electron and transmission-electron microscopy. Kroll’s re-
agent (5 pct HNO$_3$, 10 pct HF and 85 pct H$_2$O) was used to etch the samples for OM and SEM (JEOL JSM-6330F). Thin foils for TEM were prepared by twin-jet electro-polishing at 233 K using a mixture of 5 pct H$_2$SO$_4$ and 95 pct CH$_3$OH. TEM micrographs were obtained by utilizing a PHILIPS CM30 operating at an accelerating voltage of 200 kV.

### 3. Results

Figure 3 shows the true stress vs true strain curves obtained by compression tests at 1073 K with the variation of strain rate. All the curves exhibited a peak flow stress followed by moderate flow softening. Generally, flow softening of Ti-6Al-4V alloy during compression is known to occur due to deformation heating and microstructural evolution.$^5$ The temperature rise of the samples during the deformation was corrected by the artificial neural network analysis.$^7$ The solid and dashed lines in Fig. 3 represent flow curves with and without correction for deformation heating, respectively. It is noted that at relatively low strain rates of $10^{-3}$ s$^{-1}$ and $10^{-2}$ s$^{-1}$, the two flow curves (compensated and uncompensated) nearly coincide each other. However, the contribution of deformation heating to the flow stress became significant with increasing the strain rate. The compensated flow curves were used in section 4 to obtain more reliable strain rate sensitivity ($m$).

![Figure 3](image3.png)

Figure 3 shows the microstructure of the deformed sample compressed at 1073 K and the strain rate of $10^{-1}$ s$^{-1}$ up to the strain of 1.0 (partially dynamically globularized microstructure as indicated in Fig. 2). As evidenced in the micrograph, the fragmented plates as well as the kinked plates were observed. Seshacharyulu et al.$^8$ described that...
kinking of laths occurs by imposed shear strain and is completed at large strain, and Semiatin et al.\textsuperscript{5) reported that the dynamic globularization takes place preferentially at the kinked plates. The present microstructural observation supports the interpretation of earlier studies on the dynamic globularization, \textit{i.e.}, successive shearing of platelets or kinking results in fragmentation of plates. 

Figures 5(a) and (b) show TEM micrographs of the specimen fabricated by the full dynamic globularization without subsequent annealing (unannealed) and that produced by the conventional processing method involving partial dynamic globularization and subsequent annealing (annealed), respectively. Because the additional heat treatment, which was intended to increase the fraction globularized at reminiscent or kinked alpha plates, resulted in alpha grain growth, the alpha grain size of the conventionally processed specimen ($\approx 3.5 \mu m$) was about twice larger than that of unannealed one ($\approx 1.8 \mu m$). Furthermore, Fig. 5(a) reveals the microstructure with many dislocations around the grain boundaries. In contrast, Fig. 5(b) shows the microstructure with few dislocations both inside the grains and around the grain boundaries due to the additional heat treatment. Accordingly, unannealed specimen exhibited $\sim 20\%$ improvement in Vickers hardness value due to the finer grain size and higher dislocation density compared with the annealed one (Table 1).

4. Discussion

4.1 Strain rate sensitivity

Strain rate sensitivity ($m$) can be estimated from the compensated flow curves in Fig. 3 by the following expression.

$$m = d \log \sigma / d \log \dot{\varepsilon}.$$  \hfill (1)

The slope of Fig. 6 indicates $m$ and the results are summarized in Table 2 with variations of strain and strain rate. The data revealed a measurable increase of the $m$ with strain and its increase was more noticeable for tests at low strain rate. For example, in the strain rate range of $10^{-3} \text{s}^{-1}$ to $10^{-1} \text{s}^{-1}$, $m$ increased from $\sim 0.15$ to $\sim 0.36$ at the strain of 1.4. However, the $m$ which was obtained in the relatively high strain rate range of $10^{-1} \text{s}^{-1}$ to $1 \text{s}^{-1}$ still lies in a low values of $\sim 0.07$ to $\sim 0.17$ even at a large strain level ($\varepsilon \approx 1.4$), leading to the conclusion that deformation would be characterized by dislocation glide/climb processes. This result is consistent with early study for dynamic globularization of a commercial grade Ti-6Al-4V alloy having transformed microstructure with the lath width of $\sim 1 \mu m$\textsuperscript{5)} On the other hand, it seems that the deformation mechanism at low

<table>
<thead>
<tr>
<th>Grain size</th>
<th>Disl. density</th>
<th>#1</th>
<th>#2</th>
<th>#3</th>
<th>#4</th>
<th>#5</th>
<th>Average</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unannealed</td>
<td>1.8 $\mu m$</td>
<td>397.2</td>
<td>402.6</td>
<td>400.0</td>
<td>392.5</td>
<td>389.9</td>
<td>396.4</td>
</tr>
<tr>
<td>Annealed</td>
<td>3.5 $\mu m$</td>
<td>340.4</td>
<td>348.3</td>
<td>326.9</td>
<td>342.0</td>
<td>332.6</td>
<td>338.0</td>
</tr>
</tbody>
</table>

Fig. 5 TEM micrographs of the specimens fabricated by (a) the present method (unannealed) and (b) the conventional method\textsuperscript{2)} (annealed).
strain rates is somewhat different from that at high strain rates. Dynamic globularization takes place by penetration of beta phase into newly formed alpha/alpha interface at kinked point and the penetration rate is dependent on diffusion process.9) In this regard, fraction globularized increases with decreasing strain rate since low strain rate at high temperature is more beneficial to migration of the interface by diffusion. Also, the earlier studies for both thin and thick lamellar microstructures show the fraction globularized increases with decreasing strain rate.8,10) If the fraction globularized increases, grain boundary sliding would occur at boundaries between globular grains. Therefore, higher m at low strain rate is reasonable.

Also, it is noted that m of thin lath microstructure is higher than that of thick one. S. L. Semiatin et al.5) and T. Sechacharyulu et al.8) suggested that dynamic globularization of Ti-6Al-4V alloy with thick lath microstructure is controlled by dislocation glide/climb process with relatively low m of 0.25 and 0.27, respectively even at low strain rate region of $10^{-3}\text{s}^{-1}$ to $10^{-1}\text{s}^{-1}$. However thin lath microstructure contains more alpha plates than thick one at the same volume fraction, providing more kinking sites at alpha plates. Also, thin lath makes it easy to penetrate beta phase through alpha/alpha interface. Therefore it seems appropriate to observe higher m value (0.36) in the thin lath (martensitic) microstructure than in the thick lath microstructure at the strain rate of $10^{-1}\text{s}^{-1}$ to $10^{-1}\text{s}^{-1}$. Consequently, considering the above results, deformation of Ti-6Al-4V alloy with martensite microstructure during dynamic globularization at low strain rate is mainly controlled by grain boundary sliding rather than dislocation glide/climb process when the fraction globularized exceeds a certain critical quantity.

4.2 Microstructural evolution

Microstructural evolution during dynamic globularization would be visualized through the following steps which are schematically shown in Fig. 7. First, kinking of the plates occurs as shown in Fig. 4 and the kinking frequency is proportional to the imposed strain. At this process, colonies which are favorably oriented with respect to the applied stress will more take part in the kinking process and the rest colonies which are unfavorably oriented will rotate to accommodate further kinking.11) Second, dislocations of both signs are generated along the shear line preferentially at the kinked point. Then annihilation of opposite sign dislocations by glide/climb process occurs, resulting in the accumulation of same sign dislocations which increases interfacial energy of the shear line.8) Third, some globules are formed, as mentioned in section 3, by surface migration to reduce the interfacial energy. If the amount of the globularized grain exceeds a critical quantity, overall deformation is mainly controlled by grain boundary sliding rather than dislocation glide/climb. Finally, fully equiaxed microstructure (Fig. 5(a)) with the fine grain size and high dislocation density in the vicinity of grain boundaries is obtained.

5. Conclusions

The microstructural mechanisms during dynamic globularization of an ELI grade Ti-6Al-4V alloy with initial martensite microstructure were established by conducting high temperature isothermal compression tests. The following conclusions are drawn in this study.

Table 2 Strain rate sensitivity (m) of ELI grade Ti-6Al-4V alloy with variations of strain and strain rate at 1073 K.

<table>
<thead>
<tr>
<th>Strain Rate (s⁻¹)</th>
<th>$10^{-3}$ to $10^{-1}$ s⁻¹</th>
<th>$10^{-1}$ to $10^{-1}$ s⁻¹</th>
</tr>
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<tbody>
<tr>
<td>ε = 0.05</td>
<td>0.15</td>
<td>0.07</td>
</tr>
<tr>
<td>ε = 1.4</td>
<td>0.36</td>
<td>0.17</td>
</tr>
</tbody>
</table>
(1) The true stress vs true strain curves obtained by compression tests with strain rates of $10^{-3}$ s$^{-1}$ to 1 s$^{-1}$ at 1073 K exhibited a peak flow stress followed by moderate flow softening. The contribution of deformation heating to the flow stress became significant with increasing strain rate.

(2) Fully dynamically globularized alloy has higher Vickers hardness value ($H_v: \approx 396.4$) due to the finer grain size and higher dislocation density compared to the conventionally produced one ($H_v: \approx 338.0$), i.e., partial dynamic globularization with subsequent annealing.

(3) The strain rate sensitivities of the flow stresses at the initial stage ($\varepsilon \approx 0.05$) and final stage ($\varepsilon \approx 1.4$) of deformation at the relatively low strain rate region were $\approx 0.15$ and $\approx 0.36$, respectively, leading to the conclusion that deformation was controlled by dislocation glide/climb at low strains with low fraction globularized, and grain boundary sliding at high strains with high fraction globularized.

Acknowledgement

This work was supported partly by the National Research Laboratory Program (ROA-2003-000-10309-0) of the Ministry of Science and Technology, Korea, and partly by the Dual Use Technology Project (4.0000812), Agency for Defense Development and the Ministry of Science and Technology, Korea.

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