EFFECT OF THERMOMECHANICAL TREATMENTS ON TA6Zr5D β

GRAIN SIZE EVOLUTION

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Abstract

The response of TA6Zr5D alloy to thermomechanical processing in the β field has been determined. The observations of the deformed prior β grain size reveal no dynamic recrystallization by nucleation and growth for the tested conditions (ε = 2.10⁻³ → 10⁻¹s⁻¹, and ε up to 60 %). The prior β grain size variations have been studied after thermomechanical treatments during a further annealing treatment in the β temperature range and for different deformation parameters. A "static recrystallization" scheme is obtained with a critical deformation dependent on the deformation parameters. The schematic static recrystallization diagram is obtained by the study of the grain size evolution of homogeneously deformed specimens and heterogeneously deformed specimens. The knowledge of the critical deformation, and its variations with the parameters of the thermomechanical treatment allows one to modify the thermomechanical transformation paths in order to enhance the control of the grain size variations.

Introduction

During thermomechanical treatments, the material is subjected to deformation and temperature variations inducing numerous changes in the microstructure. The mechanisms which are at the basis of these changes i.e. deformation, recovery, recrystallization and phase transformations (if cooling in the appropriate temperature range) occur or may occur and can interact, these interactions being quite complex.

In the case of the TA6Zr5D alloy, industrial parts developed large heterogeneities in the prior β grain size, when deformed above the β transus. Indeed, most studies on the thermomechanical behavior were carried out in the α + β temperature range. In this study, we focus on the thermomechanical behavior above the β transus, and the microstructural variations associated with varying deformations in this range.

The mechanical behavior was studied by tensile deformation at different temperatures and deformation rates. The microstructural evolution, i.e. the prior β grain size variations, was studied by optical microscopy for different deformation parameters (ε, T, ε) on two types of specimens, "laboratory" specimens (uniformly deformed) and "massive" specimens (with heterogeneous deformation).
Experimental Procedure

The chemical composition of the alloy is given in Table I.

<table>
<thead>
<tr>
<th>Element</th>
<th>Al</th>
<th>Mo</th>
<th>Zr</th>
<th>Si</th>
<th>C</th>
<th>Fe</th>
<th>N</th>
<th>H ppm</th>
<th>O ppm</th>
</tr>
</thead>
<tbody>
<tr>
<td>wt %</td>
<td>5.97</td>
<td>0.495</td>
<td>5.22</td>
<td>0.220</td>
<td>0.007</td>
<td>0.018</td>
<td>0.003</td>
<td>17</td>
<td>1300</td>
</tr>
</tbody>
</table>

The specimens were taken from a bar, β forged, 200 mm in diameter, which had heterogeneities in the β grain size from the surface to the center. After heat treating at 1050°C, above the β-transus (1020°C), the mean grain size varies from the surface of the bar to the center in a ratio 1 to 3. Specimens for "laboratory" tests were taken so that the tensile axis is parallel to the bar axis and were classified according to their initial β grain size measured on the surface of the etched specimens, before any treatments. Specimens showing large variations in the β grain size on their surface were eliminated. Large massive specimens were obtained from the initial bar by deformation at 1050°C, in order to reduce the bar diameter to about 50 mm. After deformation a thermal treatment was carried out at 1050°C, for one hour, followed by oil quenching. After machining the specimens were 45 mm in diameter and 45 mm in height. The mean grain size of these specimens was 440 ± 50 µm.

Figure 1 represents schematically the thermomechanical and thermal treatments applied to the specimens. The specimens were all β solution treated at 1060°C, 0.5h for "laboratory" specimens and 0.66 to 0.83h for the "massive" specimens.

The "laboratory" tests were carried out on a thermomechanical simulator DITHEM (1), able to apply controlled thermal and mechanical variations. After solution treatment, the specimen is deformed homogeneously at a controlled deformation rate. The deformation level, the deformation rate, and the deformation temperature were variable parameters. The tests were carried out under vacuum.

The massive specimens were isothermally deformed at 1060°C on a hydraulic press whose displacement is controlled, corresponding to a mean deformation rate of 2 × 10^{-3} and 0.15 s^{-1}. The mean deformation is about 24 %, but it is heterogeneous and varies locally between 0 and 60 %. The thermal treatment after deformation was 1050°C for 1.5h.

The microstructures were observed at three states as defined on Figure 1, state 0, prior any deformation, state 1 after deformation and cooling and state 2 after thermal treatment. Mean β grain size corresponds to the mean linear intercept measured by optical microscopy.

Figure 1 - Schematic thermomechanical treatments
Results

Evolutions During Deformation

From the laboratory tests, we obtain the stress/strain curves in the β temperature range for different deformation rates and temperatures (Figure 2). At 1060°C, for 0.15 s⁻¹, the flow stress continuously increases up to a constant value of 34MPa. At lower strain rates, a small softening occurs at the beginning of the deformation. For a deformation rate of $2 \times 10^{-3}$ s⁻¹ the stress strain curves display similar behavior at different test temperatures. A small peak is obtained followed by smooth softening as deformation increases, whatever the temperature.

![Stress-strain curves](image)

Figure 2 - Stress strain curves obtained at 1060°C for various deformation rates (a) and obtained at different temperatures for a deformation rate of $2 \times 10^{-3}$ s⁻¹ (b).

The results can be expressed by the power law equation:

$$\sigma = A \dot{\varepsilon}^m \exp(mQ/RT)$$

were $m$, the sensitivity to the deformation rate, is about 0.29 and $Q$ the apparent activation energy 255 KJ/mole.
The microstructural evolution during deformation is observed after deformation for two
deformation rates $2 \times 10^{-3} \text{s}^{-1}$ and $0.15 \text{s}^{-1}$, at different deformation levels, after cooling the
specimen. We give Figure 3 two micrographs for each deformation rate at 15% and 30% deformation. The microstructures consist of lamellar $\alpha$ phase obtained during cooling; if we focus on the prior $\beta$ grain, we observe serrated grain boundaries, with an increase in the
serration level as the deformation increases. The serrations are less severe for the deformation rate of $2 \times 10^{-3} \text{s}^{-1}$ than for $0.15 \text{s}^{-1}$. No new $\beta$ grains are observed for the applied deformation levels. Measurements of the prior $\beta$ grain size reveal no variation of the grain size between undeformed specimens and deformed ones.

![Figure 3 - Microstructural evolutions after different deformations for two deformation rates](Image)

Figure 3 - Microstructural evolutions after different deformations for two deformation rates $0.15 \text{s}^{-1}$ and $2 \times 10^{-3} \text{s}^{-1}$

The macrography of the "massive" specimen deformed 28% at $2 \times 10^{-3} \text{s}^{-1}$ is presented Figure 4. The "massive" specimens present heterogeneities in the microstructure after deformation, with equiaxide prior $\beta$ grains at the surface of the specimen and high deformed serrated grains at the center. A calculation of the iso-deformation map by code VULCAIN estimates the deformation level at the surface near 0, while it reaches 60% at the center. Again no new $\beta$ grains were observed, even in the most deformed area and for the two deformation rates. These results, clearly show that no dynamical recrystallization for the $\beta$ phase occurs in the deformation range studied.

**Microstructural Evolution during Thermal Treatments**

The prior $\beta$ grain size evolution was determined for the different treatments without any
defformation. These results, Table II, show that the grains grow during the two treatments, depending on the initial grain size (at state 0).
For a mean initial grain size of 510 µm the high temperature β grain grows and the standard deviation increases. For a higher initial β mean grain size (1050 µm), the mean grain size remains constant; however the standard deviation increases. Also the normal growth leads to a spreading of the grain size distribution, and an increase of the mean size essentially for medium initial values of β grains.

Table II: β grain size evolution during thermal treatments

<table>
<thead>
<tr>
<th>mean grain size (µm) and [standard deviation (µm)]</th>
<th>stage 0</th>
<th>stage 1</th>
<th>stage 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>510 and 220)</td>
<td>640 [340]</td>
<td>970 [560]</td>
<td></td>
</tr>
<tr>
<td>1050 [550]</td>
<td>1030 [550]</td>
<td>1050 [620]</td>
<td></td>
</tr>
</tbody>
</table>

In order to put out the effect of the deformation, the grain size variations are given as a difference between sizes at state 2 and state 0. Figure 5 presents the variations of the β grain size versus the applied deformation and for three couples of deformation parameters (, T). The results are given as a mean value with dispersion brackets (95 % of the measured mean values are inside the brackets). We observe that the difference of size between state 2 and state 0 is the same up to a critical value. At that value, a maximal increase of the difference occurs, which lowers as the deformation increases. The deformation at which the maximal increase is noticed, called the critical deformation, is dependent on the high temperature deformation parameters (Table III).

Table III: Variation of the critical strain versus high temperature deformation parameters

<table>
<thead>
<tr>
<th>T(°C)</th>
<th>= 2 10^-3 s^-1</th>
<th>0.15</th>
<th>2 10^-3</th>
</tr>
</thead>
<tbody>
<tr>
<td>1010</td>
<td>7-8 %</td>
<td></td>
<td></td>
</tr>
<tr>
<td>1060</td>
<td>15-16 %</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The observations of the massive specimens deformed and heat treated, reveal a similar scheme (Figure 6). Near the surface, small grains are observed, whose size increases and then decreases when observing along the deformation axis. The superposition of the calculated
deformation pattern on the microstructure reveal that the large grains are observed between the isodeformation lines of 10 and 20% for the deformation rate of $2 \times 10^{-3}$ s$^{-1}$ and 5-10% for the deformation rate of 0.15 s$^{-1}$.

Figure 5 - Variations of the $\beta$ mean grain size between state 2 and state 0 versus the applied deformation for three deformation conditions.
Discussion

Stress-strain curves yield to a mean value of \( m \) equal to 0.29 and an apparent activation energy of 255KJ/mole for the \( \beta \) phase. The \( m \) value is large compared to the one obtained by Malcor (2) on TA6V4 alloy (0.18 at 1010°C), but less differences are noticed when comparing to Hanaki (3) (\( m = 0.24 \) for TA6V4 at 1050°C), or to Côme-Dingremont (4) (\( m = 0.29 \) for TA6V4 at 1015°C). The larger values are obtained when studying a range of deformation rate including low deformation rates (10\(^{-3}\) to 10\(^{-4}\)s\(^{-1}\)) and more generally using tensile tests (for Malcor the deformation was obtained by torsion). For the apparent activation energy, the value obtained is larger than the one measured on TA6V4 alloy above the \( \beta \) transus. The microstructural observations of the deformed specimens revealed that the alloy does not dynamically recrystallize by nucleation and growth, in the deformation range studied. The small softening observed is then related to dynamic recovery.

After deformation and further thermal treatment, the grain size variations observed for different primary deformations, follow a scheme similar to the one obtained for different materials during static recrystallization (5). For titanium alloys very few results were reported on recrystallization above the \( \beta \) transus. In our case, even if the size variations are sometimes low, both the laboratory and the massive specimens present these variations and the "critical deformation" is dependent on the deformation parameters. In order to approach the recrystallization kinetics, one sample was maintained 180s after deformation (5%) and no structural change was observed compared to the sample cooled just after deformation. Also the mechanism of that static recrystallization has been further studied on a TA6V4 alloy (4,6) showing that recrystallization occurs by migration of the existing \( \beta \) grain boundaries.

In order to explain the difference in the \( \beta \) grain size between "laboratory" and "massive" specimens, some specimens were heat treated after deformation under a constant applied stress of 1.5MPa. For the non deformed specimen, no increase in grain size was observed compared to the specimen heat treated without applied stress; however an increase in the grain size is observed, especially for the 15% deformed specimen (\( \dot{\varepsilon} = 2 \times 10^{-3}s^{-1}, T = 1060°C \)) that
deformation corresponding to the "critical deformation". The mean grain size and the maximal size for the "laboratory" 15% deformed specimens and for the "massive specimen" in the 15% deformed area, are reported Table IV.

Table IV: Variations of grain size for specimens heat treated with or without applied stress after a pre-deformation of ~15%.

<table>
<thead>
<tr>
<th></th>
<th>Laboratory specimen</th>
<th>Massive specimen</th>
<th>Laboratory specimen with constant stress</th>
</tr>
</thead>
<tbody>
<tr>
<td>mean size (µm)</td>
<td>1400</td>
<td>2400</td>
<td>1900</td>
</tr>
<tr>
<td>maximal size (µm)</td>
<td>2700</td>
<td>4000</td>
<td>3500</td>
</tr>
</tbody>
</table>

The larger grain size obtained when the last thermal treatment occurs under an applied stress can be related to a higher velocity of the grain boundary as observed on TA6V4 alloy (4) or in other studies (7). Also the difference between the "massive" and the "laboratory" specimens can be partly related to an internal stress state during static recrystallization which will lead to an increase in the β grain boundary mobility. Another factor can be a slower recrystallization kinetics as it can be suggested regarding the kinetics results versus the recrystallization temperature obtained by Côme-Dingremont and al. (4,6).

Conclusion

The mechanical behavior of TA6Zr5D alloy has been studied by tensile testing in the β phase field. The results show that dynamical recrystallization does not occur for the deformation range studied (0-30%). During further heat treatment, the β mean grain size varies with the previous deformation level according to a static recrystallization scheme. The critical strains are obtained for different deformation parameters. Modifications of the thermomechanical path can then be planned in order to enhance the control of the mean grain size.

Acknowledgments

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References


6. N. Côme-Dingremont, E. Gautier and A. Simon, "Grain growth of TA6V β phase after thermomechanical treatments" (this conference).