Analysis of the role of plastic deformation on the transformation kinetics of the metastable $\beta$-Cez titanium alloy.

E. LAUDE, E. GAUTIER, D. DELANNOY, P. ARCHAMBAULT

Laboratoire de Science et Génie des Matériaux Métalliques - UMR CNRS / INPL / UHP 7584
École des Mines Parc de Saurupt 54042 Nancy Cedex France

ANNOTATION
The effect of a plastic deformation on the $\beta$-$\alpha$ transformations of the $\beta$-Cez has been studied for transformation temperatures ranging from 830 to 600°C. Applying a deformation in the metastable $\beta$ range led to an accelerated isothermal transformation kinetics. The effect of the deformation is essentially to modify the nucleation rate of the grain boundary $\alpha$ phase. This accelerating effect is larger for deformation temperatures close to the transformation temperatures, and for the lowest undercoolings. In isothermal transformation conditions the $\alpha_{WGB}$ width remains constant indicating that the growth rate is nearly unaffected. For intragranular $\alpha$ phase, the nucleation rate is also increased. In consequence the aspect ratio (width/length) of the $\alpha$ phase is increased. In the case of transformation during continuous cooling, the previous deformation strongly accelerates the $\beta \rightarrow \alpha_{GB} + \alpha_{WGB}$ transformation.

Key words: $\beta$ metastable titanium alloys, thermomechanical treatments, $\beta \rightarrow \alpha$ phase transformation, kinetics

1. INTRODUCTION
In order to optimize the mechanical properties of titanium alloys it is necessary to know the relationships between microstructures and mechanical properties as well as the relationships between thermomechanical treatments and their resulting microstructures. We thus have studied the influence of thermomechanical treatments on the transformations occurring during cooling from the $\beta$ temperature range. Deformation is known to influence the transformation kinetics as observed for different alloys and different transformations [1-7]. In order to quantify these effects, the influence of a plastic deformation on the $\beta \rightarrow \alpha$ transformation during isothermal transformation or during continuous cooling has been investigated.

2. MATERIAL. EXPERIMENTAL METHODS
The alloy studied was the $\beta$-Cez alloy [8] which chemical composition in wt % is: 6% Al, 2% Sn, 4% Zr, 4% Mo, 2% Cr, 1% Fe, bal Ti. The $\beta$ transus temperature is 890°C. Tests were performed on a thermomechanical simulator DITHEM [9,10]. Temperature variations, deformation rate and deformation amount were computer controlled all over the test. For the reported cases, the deformation conditions are 25% deformation and a deformation rate $\dot{\varepsilon} = 10^{-2}$ s$^{-1}$. Deformation was applied either in the stable $\beta$ temperature range or in the metastable $\beta$ temperature range.

In order to reach the transformation progress, in-situ electrical resistivity measurements were performed during the test: A four point method was used; the specimen was submitted to a constant current ($I = 3A$), and the potential variations were recorded and related to the electrical resistance variations due to temperature and structural changes. Measurements of the potential variations were performed with two pure-Pt wires, each one being spot welded 8mm far from the middle of the specimen's gage length.

In order to study the microstructures evolutions, the specimens were helium quenched at different transformation content. The amount of phase formed was quantified by XR diffraction. The microstructures were observed by optical and scanning electron microscopes allowing quantitative analysis of different morphological parameters.

3. RESULTS
3.1. ISOTHERMAL TRANSFORMATION KINETICS
Transformation kinetics were determined considering either the $\beta \rightarrow \alpha_{GB} + \alpha_{WGB}$ transformation, or the $\beta \rightarrow \alpha_{W1}$ transformation. The transformation temperatures were between 830 °C and 600°C.
The kinetics of electrical resistivity variations and the amount of α phase measured by XR diffraction analysis are reported Fig. 1, for two transformation temperatures, 790 and 700°C. Some discrepancies are observed between the different measurements. Globally, the kinetics could be considered as comparable. However, the amount of phase formed by XR diffraction is always larger than the one obtained from electrical resistivity measurements. The difference between the two methods increases as the transformation temperature decreases as shown for the transformation temperature of 700°C, which could be attributed to further transformation during quenching. But the rapid helium quenching should avoid any further transformation as it was observed for very fast cooling from 920°C. For lower transformation temperatures (700, 600 and 500°C), the β lattice parameters evolutions are delayed to longer transformation times [11, 12]. We could thus assume that for the lowest temperatures and mainly for the α_w1 formation, an HCP phase is formed which chemical composition is not the equilibrium one; as transformation time increases, equilibrium composition can be reached. Electrical resistivity could thus be sensitive to the chemical composition factor in addition to the structural one. In consequence, considering the study on the influence of the deformation on the transformation kinetics, electrical measurements will be considered for the β → α GB + α_w1 and XR measurements will be considered for the β → α_w1 transformation.

Fig 1: Comparison of relative electrical variations and α phase amount measured by XR diffraction at 790 and 700°C.

3.2. EFFECT OF THE PLASTIC DEFORMATION ON THE ISOTHERMAL TRANSFORMATION KINETICS

3.2.1. Overall transformation kinetics

Deformation of the β phase leads to an acceleration of the transformation kinetics. Both the incubation time and the duration of the transformation are shortened (Table 1).

Table 1: Influence of the deformation on the beginning time (t_b) and duration (t_90 - t_0) of the β phase transformation

<table>
<thead>
<tr>
<th>Transformation temperature (°C)</th>
<th>Influence of</th>
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</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>ε on t_b</td>
<td>ε on (t_90 - t_0)</td>
<td>ε on t_b</td>
<td>ε on (t_90 - t_0)</td>
<td>ε on t_b</td>
<td>ε on (t_90 - t_0)</td>
</tr>
<tr>
<td>830</td>
<td>- 71 %</td>
<td>- 0 %</td>
<td>- 67 %</td>
<td>- 16 %</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>790</td>
<td>- 46 %</td>
<td>- 62 %</td>
<td>- 43 %</td>
<td>- 54 %</td>
<td>- 80 %</td>
<td>- 86 %</td>
</tr>
<tr>
<td>750</td>
<td>0 %</td>
<td>- 43 %</td>
<td>0 %</td>
<td>- 51 %</td>
<td>- 50 %</td>
<td>- 80 %</td>
</tr>
</tbody>
</table>

The duration of the transformation is expressed as (t_90 - t_0) time for 90 and 10% transformation respectively. These results show that the reductions are function of the deformation temperature, as of the amount of the deformation [13, 14]. Concerning the incubation time, for a same deformation temperature, the reduction is larger for lower undercoolings. The reduction in the incubation time is 71% for the transformation temperature of 830°C.
830°C while it is of 46% for 790°C and nearly 0 for 750°C. If the deformation temperature is modified these effects are larger i.e. for a deformation at 790°C the incubation time is reduced by 80% for a transformation at 790°C and by 50% for a transformation at 750°C.

For the duration of the transformation it is shown that the reductions are the larger for the lower transformation temperatures.

3.2.2. Effect of a pre-deformation on the microstructure formation

Fig 2 are SEM micrographs for different specimens transformed at 790°C. For a non deformed specimen, $\alpha_{GB}$ is not observed at all grain boundaries, and the $\alpha_{WGB}$ phase is formed on the $\beta$ grain boundaries where $\alpha_{GB}$ precipitated. For a specimen deformed at 920°C, mostly all grain boundaries present $\alpha_{GB}$ phase, and on a large part of them $\alpha_{WGB}$ is observed. At last, for a deformation at 790°C, $\alpha_{GB}$ and $\alpha_{WGB}$ are formed on almost all grain boundaries with a same amount of $\alpha_{WGB}$. Thus the effect of deformation is to increase the nucleation rate of the $\alpha_{GB}$ phase. For these deformation conditions, nucleation occurs mainly at previously deformed $\beta$ grain boundaries. The differences between deformation at 920 and 790°C (a lower increase in the nucleation rate) are mainly attributed to a recovery of the deformation structure when cooling from 920 to 790°C, limiting thus the effect of the deformation.

Additional experiments revealed that for deformation and transformation at 750°C, new nucleation sites on sub-boundaries were are activated [15]. This increase of the nucleation rate on grain boundaries as on sub boundaries due to the pre deformation was also observed by Chaussy et al [16] for larger pre deformation. Moreover, Chaussy mentioned that nucleation on grain boundaries is more efficient than that on sub boundaries.

In order to further quantify the effect of the deformation on the microstructures, different morphological parameters were determined: mean width of $\alpha_{GB}$, $\alpha_{WGB}$ and $\alpha_{W1}$. Fully transformed specimens were investigated for different transformation temperatures. Variations of $\alpha_{GB}$ mean width with transformation temperature are reported figure 3. We observe that the $\alpha_{GB}$ mean width decreases with the transformation temperature for the non-deformed or the deformed specimens. The pre deformation has no effect for transformation temperatures $\leq 790°C$, but induces a diminution of about 20 for deformed specimens transformed at 830 °C. However we have to mention that the standard deviation is quite large at that transformation temperature because $\alpha_{GB}$ is not present on all grain boundaries.

As $\alpha_{GB}$ formation is diffusion controlled, the variations of $\alpha_{GB}$ mean width with transformation temperature is coherent considering that the diffusion is larger at the higher temperatures. The lower width obtained for deformed specimens, may be explained by the higher occupation of grain boundaries which lead to a more rapid formation of $\alpha_{WGB}$.

The mean width of the $\alpha_{WGB}$ and $\alpha_{W1}$ lamellae was also measured on specimens fully transformed. Results are reported Fig. 4. This parameter decreases as the transformation temperature decreases. If one considers the effect of deformation, we can only notice a very small difference compared to the non deformed specimen.
This first group of results does not reveal a strong influence of the pre deformation on the morphological parameters of the $\alpha$ phase. In order to further quantify the microstructural changes observed Fig.2, we measured the number of colonies / grain for transformation temperatures where the $\beta \rightarrow \alpha_{GB} + \alpha_{WGB}$ was observed.

The number of colonies/grain was determined at different transformation times and for different transformation temperatures. If we consider first a transformation without previous deformation, the number of colonies /grain increases as transformation progresses and reaches a maximal value before the transformation is completed. A decrease in the transformation temperature leads to an increase in the total number of colonies/grain (Fig.5). Moreover, the variations of the number of colonies/grain versus transformation time are faster at lower transformation temperatures. In the case of a transformation at 790°C, when deforming the sample, the total number of colonies / grain is increased by a factor 3. This number is quite similar for the two deformation conditions and equivalent to the one obtained at 700°C without previous deformation. The deformation conditions modify mainly the kinetics of colonies formation which are more rapid when deforming at 790°C. For a transformation temperature of 700°C, the total number of colonies / grain for deformed and non deformed samples were the same [14]; the main difference concerns the evolution of the number of colonies versus time.

Considering the formation of $\alpha_{GB} + \alpha_{WGB}$, microstructural observations and quantitative measurements show that the nucleation of $\alpha_{GB}$ is accelerated. The mean width of $\alpha_{GB}$ and $\alpha_{WGB}$ are not modified in a large amount by the previous 25% deformation, indicating that the growth rate is poorly affected. A larger number of $\alpha_{WGB}$ colonies is observed. The effect of the deformation is comparable to an increase in the driving force (i.e. a lower transformation temperature). This increase in the number of colonies / grain can be directly related to the increase of $\alpha_{GB}$ nucleation rate. Indeed, for low undercoolings, all grain boundaries will not be saturated. $\alpha_{WGB}$ forms on
$\alpha_{GB}$, and grows in the grain. Transformation could be completed without site saturation. As undercooling increases, driving force for the transformation increases and site saturation occurs previously; formation of new $\alpha_{WGB}$ colonies on $\alpha_{GB}$ is more rapid and thus the number of colonies increases.

For the formation of $\alpha_{w1}$, transformation kinetics and morphological parameters were measured too. The overall kinetics is accelerated [13]. Considering $\alpha_{w1}$ mean width, no major effect of a pre deformation is observed as shown figure 4 for the transformation temperature of 600°C and figure 7 where micrographs are presented. For this transformation, we characterized the variations in the mean ratio width/length of the $\alpha$ plates. The results are reported figure 8. An increase in the ratio width/length is observed when transformation progresses; these variations are mainly due to the simultaneous decrease of the width and the length of the plates the later being larger than the mean width variations. Applying a pre deformation to the samples leads to an increase in the mean width/length ratio. These small variations are essentially due to a modification of the plate length because the mean width is not modified (Fig. 4).

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![Figure 7: SEM micrographs of specimens transformed at 600°C for a complete transformation](image)

- a) without deformation
- b) 25% pre deformation at 920°C

![Figure 8: Variations of the mean ratio width/length for $\alpha_{w1}$ plates formed at 600°C for non deformed and deformed specimens.](image)

**3.3 TRANSFORMATION DURING CONTINUOUS COOLING.**

At last we studied the effect of a 25% pre deformation at 920°C on the further transformation during continuous cooling. Microstructures obtained for a cooling rate of 600°C/h are shown Fig. 9. These micrographs show that the morphological parameters of the $\alpha$ phase as the relative amounts of $\alpha_{WGB}$ and $\alpha_{w1}$ are modified.
Measurements of these morphological parameters were performed for the non deformed and deformed specimens. The variations of $\alpha_{\text{GB}}$ mean width with the cooling rate are plotted Fig. 10. For non-deformed specimens, $\alpha_{\text{GB}}$ mean width decreases as cooling rate increases. Applying a deformation leads to an increase in the $\alpha_{\text{GB}}$ mean width for cooling rates of $1^\circ\text{C/s}$ and $600^\circ\text{C/h}$. For the lower cooling rate of $150^\circ\text{C/h}$ a small decrease in the mean width is obtained. In the case of isothermal transformation conditions, we observed that a pre deformation increases the nucleation rate of the $\alpha_{\text{GB}}$. For continuous cooling, the increase of $\alpha_{\text{GB}}$ mean width is due to the occurrence of the transformation at higher temperatures. Diffusion being faster, the $\alpha_{\text{WGB}}$ mean width is thus increased. This increase will be observed until reaching the conditions for $\alpha_{\text{WGB}}$ formation. For the lowest cooling rates, the decrease in $\alpha_{\text{GB}}$ width can be compared to the decrease observed in isothermal transformation conditions for a transformation at $830^\circ\text{C}$ where $\alpha_{\text{WGB}}$ is formed earlier.

![SEM micrographs](image)

**Figure 9:** SEM micrographs for specimens cooled at a constant cooling rate of $600^\circ\text{C/h}$:
- a) non-deformed,
- b) deformed 25% at $920^\circ\text{C}$.

![Graph](image)

**Figure 10:** Variations of $\alpha_{\text{GB}}$ mean width versus cooling rate for non deformed and deformed specimens

The mean widths of the $\alpha_{\text{WGB}}$ and $\alpha_{\text{W1}}$ lamellae were measured for the different transformation conditions (Fig. 11). Increasing the cooling rate leads to a decrease in the $\alpha$ mean width. When the specimens are deformed, we observe that the mean width is increased for a same cooling rate.
Figure 11: Variations of $\alpha_{WGB}$ mean width versus cooling rate for non-deformed and deformed specimens

At last, considering $\alpha_{W1}$ formation, we determined the variations in width/length ratio for a cooling rate of 1°C/s. These values are reported Table 2. A pre-deformation of the sample leads to an increase of the mean/width ratio by nearly a factor 2.

<table>
<thead>
<tr>
<th>Cooling rate (°C/s)</th>
<th>Mean width (μm)</th>
</tr>
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<tbody>
<tr>
<td>0.01</td>
<td>0.8</td>
</tr>
<tr>
<td>0.1</td>
<td>0.6</td>
</tr>
<tr>
<td>1</td>
<td>0.4</td>
</tr>
<tr>
<td>10</td>
<td>0.2</td>
</tr>
</tbody>
</table>

Table 2: Variations of the mean width/length ratio of the $\alpha_{W1}$ plates for a 1°C/s continuous cooling.

<table>
<thead>
<tr>
<th>$\epsilon$</th>
<th>Mean value ($10^{-2}$)</th>
<th>Mean value ($10^{-2}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>4.2 ± 0.4</td>
<td>7.9 ± 0.7</td>
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</table>

4 DISCUSSION.

The results obtained in isothermal transformation conditions, clearly show that the plastic deformation accelerates the phase transformation kinetics. For $\beta \rightarrow \alpha_{GB} + \alpha_{WGB}$ transformations the deformation will increase the nucleation rate of $\alpha_{GB}$. For all the microstructures observed in our deformation conditions, the effect of the deformation leads to a more rapid site saturation of the nucleation sites, i.e. the $\beta$ grains boundaries. Similar results were obtained for the ferritic or pearlitic transformation of steels [2, 3, 17] or for discontinuous precipitation in Cu-Cd alloys [18]. When transformation progresses, $\alpha_{GB}$ width does not increase. However the formation of $\alpha_{WGB}$ is observed earlier and the number of $\alpha_{WGB}$ colonies / grain increases if site saturation does not occur for the considered transformation temperature. The $\alpha_{WGB}$ width is not modified by the previous deformation whatever the deformation temperature is. This indicates that dislocations do not increase the cellular type growth rate of $\alpha_{WGB}$. Chaussy showed that a rapid recovery occurs in the deformed $\beta$ structure [16] leading to the formation of sub-boundaries as deformation increases. Considering the increase in the $\alpha_{GB}$ nucleation rate, the effect of the deformation can be related to an increase in the driving force due to the elastic energy stored in the matrix and associated with the defects. However, this contribution is generally low. Moreover, the observations of $\alpha_{GB}$ on the previous $\beta$ grain boundaries tends us to rather relate this increase to the serration of the grain boundaries as proposed by Umemoto et al [2], and to the nature of the deformed boundary which lowers the energetic barrier and allows the formation of $\alpha_{GB}$ allotriomorphs.

For the $\beta \rightarrow \alpha_{W1}$ transformation, the kinetics is also accelerated. The morphological changes are mainly an increasing ratio width/length due to a lowering of the plate's length. This decrease can be related to the size of the sub grains, which can limit the plate growth, or to an increased number of intra-granular nucleation sites.

Considering the transformation on continuous cooling, large changes in the microstructure are observed. As the formation of $\alpha_{GB}$ is accelerated, it will be able to grow at larger extent, which favours the formation of $\alpha_{WGB}$, depending on the amount of the deformation and on the cooling rate. The amount of $\alpha_{W1}$ can thus be lowered. This is clearly the case for specimens deformed 25% at 920°C and cooled at 1°C/s.

These results show that for the prediction of the microstructure in titanium parts during thermomechanical treatments, it is necessary to describe the transformation kinetics depending on both the cooling rate and the previous deformation (i.e. the deformed structure). Models describing the kinetics of different $\alpha$ morphologies [14, 18], have thus to consider the nucleation and growth rate of each morphology.
CONCLUSIONS

We studied the effect of a 25 % plastic deformation on the $\beta \rightarrow \alpha$ transformation of the $\beta$-CeZ alloy. Plastic deformation in the $\beta$ temperature range accelerates the $\beta \rightarrow \alpha_{\text{GB}} + \alpha_{\text{WGB}}$ or $\beta \rightarrow \alpha_{\text{W}}$ transformation kinetics. By quantitative measurements of different morphological parameters, we showed that the major effect is to increase the nucleation rate of the $\alpha_{\text{GB}}$, leading to an acceleration of the $\alpha_{\text{WGB}}$ formation.

In isothermal transformation conditions, the width of $\alpha_{\text{GB}}$ or $\alpha_{\text{WGB}}$ lamellae is poorly modified. However, for continuous cooling, the transformation occurs at higher temperatures and the width of $\alpha_{\text{GB}}$ or $\alpha_{\text{WGB}}$ is increased.

Moreover, the respective amount of $\alpha_{\text{WGB}}$ and $\alpha_{\text{W}}$ could be largely modified depending on the cooling rate and on the deformation conditions.

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