Phase transformation kinetics in a near-β titanium alloy

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Central European PhD Students
Research in Materials Science

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1- Goal

2- Experimental
   2.1. Material
   2.2. Techniques:
      - In Situ High Energy Synchrotron X-ray Diffraction (XRD)
      - Differential Scanning Calorimetry (DSC)

3- Results
   3.1. Bimodal microstructure
   3.2. Lamellar microstructure
   3.3. Beta quenched microstructure

4- Summary
To study the phase transformation kinetics during continuous heating of a near-β titanium alloy with different initial microstructures

Methods

*Differential Scanning Calorimetry (DSC)*
Identification of precipitation and dissolution processes

*In situ High Energy Synchrotron X-ray diffraction (XRD)*
Identification AND quantification of microstructural phases
2- Experimental

2.1. Material

Near-β alloy

Ti-10V-2Fe-3Al

β transus Temperature

<table>
<thead>
<tr>
<th>β transus Temperature</th>
<th>Mo eq. (wt.%) β Stabilizers</th>
<th>Characteristics</th>
<th>Applications</th>
</tr>
</thead>
<tbody>
<tr>
<td>808°C</td>
<td>10.81%</td>
<td>• Excellent Forgeability</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>• High strength</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>• High toughness (air, salt water)</td>
<td></td>
</tr>
</tbody>
</table>

Aerospace industry (variety of components)

Handbook of Titanium Alloys, 2007
2- Experimental

2.1. Material: Initial microstructures

**Bimodal**

- **Ar Atmosphere**
  - $T_{\beta\text{TRANSUS}}$
  - $T_{\alpha\text{TRANSUS}}$
  - 763°C / 2 h
  - 510°C / 8 h

**Lamellar**

- **Large $\alpha$ Lamellae**
- 40µm

**Beta quenched**

- **Ar Atmosphere**
- 5 k/min
- 510°C / 8 h

- **Water quenching**

**Primary $\alpha$**

- 50µm

**Secondary $\alpha$-lamellae**

- 3µm

**Small $\alpha$ Lamellae**

- 10µm

**Metastable $\beta$ grains**

**$\beta$ martensite**

**$\omega_{\text{ath}}$**
2- Experimental

2.2. Techniques: In Situ High Energy X-ray Diffraction

ID15-ESRF
E = 87keV
\( \lambda = 0.1426\) Å
Slit size = 0.3x0.3 mm²
Acquisition time = 2s
Readout time = 1s
Sample size = 4x4x10 mm³

Ar Atmosphere
900°C

\( T_{\text{TRANSUS}} \)
20 k/min

Data processing

2D analysis
(calibration/integration/image analysis)

Image processing

Quantitative phase analysis
(Rietveld)

E. Aeby-Gautier, JOM 2007

fit2d

ImageJ

MAUD
2- Experimental

2.2. Techniques: DSC

![Diagram showing experimental setup for DSC analysis with Ti1023 Sample, Alumina Crucible, Reference Sample Alumina, Ar Atmosphere, and temperature-time graph.](image)
3- Results

Bimodal

Lamellar

Beta quenched

\( T (\degree C) \)

\( T_{\text{TRANSUS}} \)

\( f(T) \)

\( \alpha' + \beta \rightarrow \beta \)

\( \alpha \)

\( \alpha \)

\( \beta \)

\( \alpha \)

\( (100) \)

\( (002) \)

\( (110) \)

\( (101) \)

\( 2\text{-Theta (}\degree\text{)} \)

\( \alpha' \)

\( \alpha' \)

\( \beta \)

\( \alpha' \)

\( (100) \)

\( (002) \)

\( (110) \)

\( (101) \)

\( (110) (020) \)

\( (110) (002) (111) \)

\( \alpha'' \)

\( \alpha'' \)

\( \alpha'' \)

\( \alpha'' \)
3- Results

3.1. Bimodal microstructure

Two dissolution processes may indicate sequential dissolution of α secondary → α primary

Ti64, P. Barriobero Vila, Master’s Thesis

Start α+β → β
DSC T~ 425-450 °C
XRD T~ 450-500°C

End α+β → β
DSC T~ 875°C

$\mathbf{a}_\beta$ shows a sudden increase at T=500°C

Diffusion of alloying elements
3- Results

3.2. Lamellar microstructure

Assymetric endotermic peak may indicate sequential dissolution of smaller and larger lamellae

Ti17, E. Aeby-Gautier, JOM 2007

Start $\alpha+\beta \rightarrow \beta$

DSC $T \sim 500-550^\circ C$

XRD $T \sim 575^\circ C$

End $\alpha+\beta \rightarrow \beta$

DSC $T \sim 875^\circ C$

$a_\beta$ shows a sudden increase at $T=500^\circ C$

Diffusion of alloying elements

Stabilization of alpha content between $\sim 400<T<575^\circ C \rightarrow$ increase from $\sim 65wt\%$ to $\sim 75wt\%$
3- Results
3.3. Beta quenched microstructure

Start $\alpha+\beta \rightarrow \beta$
DSC $T \sim 575 ^\circ C$

End $\alpha+\beta \rightarrow \beta$
DSC $T \sim 855 ^\circ C$

XRD: Beta quenched microstructure shows the presence of stable $\alpha$ and $\beta$ plus metastable $\omega_{\text{ath}}, \omega, \alpha''$, $\alpha''_{\text{iso}}$ phases at different stages during heating

$\omega_{\text{ath.}}(TEM)+\beta+\alpha''$
$\omega+\beta +\alpha''$
$\omega+\beta +\alpha''$
$\beta + \alpha''_{\text{iso}}$
$\beta+\alpha$
$\beta$

References:
- Ivasishin, Materials Science and Engineering 2005
3- Results

3.3. Beta quenched microstructure

20°C

390°C

425°C

540°C

Heat Flow (mW/mg)

Temperature (°C)

5 k/min
4- Summary

• **In Situ High Energy XRD:**
  Reveals the phases and its weight/volume fraction as f(T)
  The faster increase of the $a_\beta$ cell parameter at a threshold temperature of 500°C may be due to the diffusion of alloying elements into β phase (Al, Fe and/or V)

• **DSC:**
  Reveals that the α phase (identified by XRD) dissolves at different stages depending on its morphology → fine lamella start to dissolve earlier than coarse lamellae or globular α

  - The **bimodal and lamellar** microstructures show the presence of α and β phases at different stages during heating (no metastable phases were observed)
  
  - The **beta quenched** microstructure presents a much more complex phase transformation kinetics as a consequence of the formation of the metastable phases $\alpha''$, $\alpha''_{iso}$, $\omega_{ath}$, ω
**4- Summary**

<table>
<thead>
<tr>
<th>Microstructure</th>
<th>Start $\alpha+\beta \rightarrow \beta$ Temperature (°C)</th>
<th>$\alpha_B$ Increasing temperature (°C)</th>
<th>End $\alpha+\beta \rightarrow \beta$ Temperature (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>DSC</td>
<td>XRD</td>
<td></td>
</tr>
<tr>
<td>Bimodal</td>
<td>425-450</td>
<td>450-500</td>
<td>500</td>
</tr>
<tr>
<td>Lamellar</td>
<td>500-550</td>
<td>575</td>
<td>500</td>
</tr>
<tr>
<td>Beta quenched</td>
<td>575</td>
<td>-</td>
<td>-</td>
</tr>
</tbody>
</table>

*Bimodal* starts earlier due to the presence of smaller $\alpha$-lamellae $\rightarrow$ higher surface energy

**Outlook:**
- Metallography of microstructures frozen at different stages during heating to confirm the dissolution sequence of the different $\alpha$ morphologies
- The complex phase transformation kinetics of the Beta quenched microstructure will be quantified by Rietveld analysis.
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