In situ characterisation of phase transformations in titanium alloys by high energy X-ray diffraction

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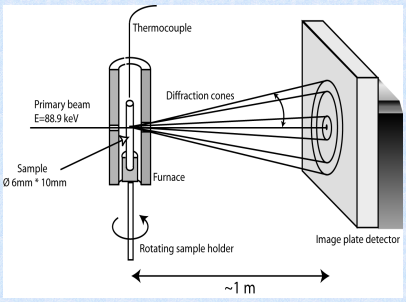
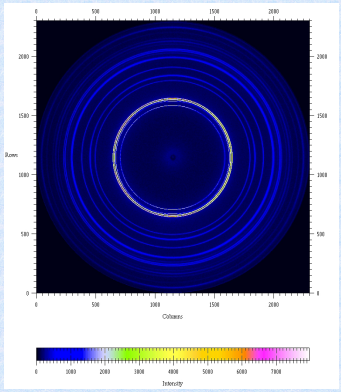
In situ measurements of phase evolutions were performed at ESRF on ID15B beamline using high energy X-ray diffraction in order to quantify the transformations kinetics of Ti17 titanium alloys. Two phases are presents in this alloys α (HCP) and β(CC). The phase transformations were studied on heating to 920°C for a single heating rate (0.5°C/s), on cooling for different cooling rates (0.05, 0.1, 0.61°C/s) and during isothermal stage at 750 and 708°C.

Experimental methods

The transformation kinetics was studied by means of a monochromatic high energy X-ray beam (λ=0.01402nm). The data were recorded with the MAR 345 image plate detector for the acquisition of complete Debye-Scherrer diffraction rings. For the analysis, the intensity was integrated on each ring in order to obtain a diffraction pattern suitable for Rietveld analysis using the software Fullprof. The measurements were obtained each 2min.

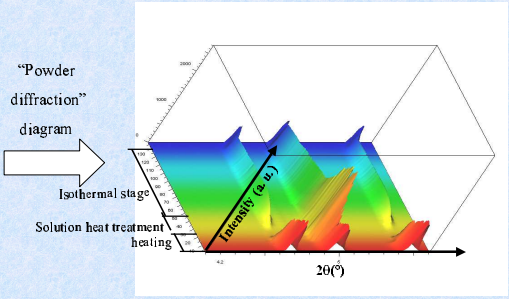


Experimental set up



Debye-Scherrer rings

"Powder diffraction" diagram



3D Representation of diffraction patterns obtained during a the thermal cycle (heating to 920°C – cooling to 750°C - 3h holding at 750°C and further cooling to room temperature

Transformation on heating to 920°C (heating rate=0.5°C/s)

A dissolution of the α phase is observed at T>610°C. Dissolution occurs in two steps which can be associated with a first dissolution of the α lamellar followed by a dissolution of α equiaxed grains.

In the dissolution temperature range, c_α and a_β depend on the temperature and the amount of α phase (the lattice parameter variations are clearly related to a change in chemical composition of the β phase).

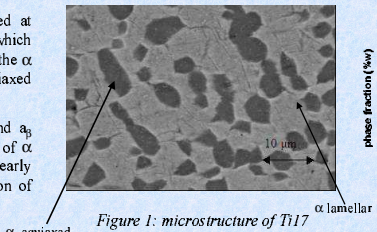


Figure 1: microstructure of Ti17 before heat treatment

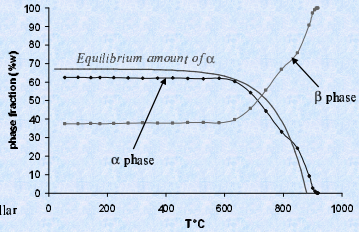


Figure 2 : Evolution of the α and β phase fractions

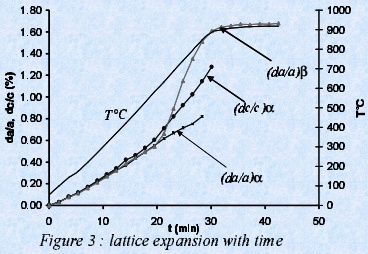


Figure 3 : lattice expansion with time

Transformation on cooling

Isothermal transformation at 750°C and 708°C

Small amounts of α phase are present after the solution heat treatment and the first cooling to the transformation temperature due to surface oxidation.

Isothermal transformations kinetics are measured for both temperatures. A good correlation between the XRD measurements and electrical resistivity variations is obtained. The lattice parameter a_α is constant whatever the phase amount is. a_β decreases as the amount of α increases and remains constant when the transformation is completed.

The final amount of α phase is equal to the equilibrium amount at the transformation temperature (figure 2).

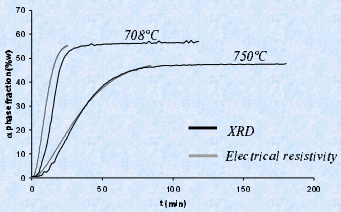


Figure 4 : Evolution of the fraction of α phase versus time

Continuous cooling at 0.05°C/s, 0.1°C/s, 0.61°C/s

Results show that the undercooling necessary for the α phase growth increases as cooling rate increases. For 0.05°C/s, the amount of α phase reaches the equilibrium value. For the higher cooling rates, the amount is lower than the equilibrium one.

A fair correlation is obtained between the XRD measurements and electrical resistivity variations in the both cases (0.1°C/s, 0.05°C/s). A small difference can be noticed for the lower cooling rate which can be partly due to the oxidized surface layer.

As observed for isothermal transformation, a_β decreases as the amount of α increases. a_β is also dependent on the cooling rate.

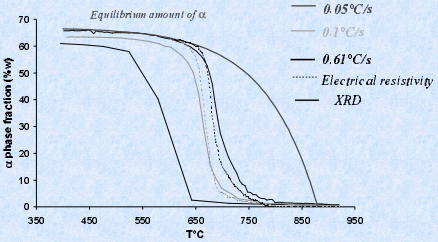


Figure 5: Evolution of the amount of α phase versus temperature for the different cooling rates

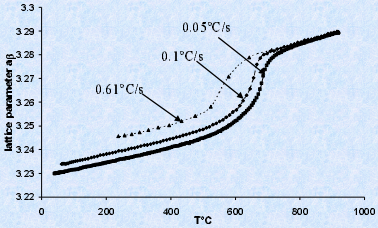


Figure 6 : Evolution of the lattice parameters a_β versus temperature for the different cooling rates

Conclusions

- Phase transformations kinetics of Ti17 were obtained by in situ high energy XRD during heating and further cooling (isothermal transformation and continuous cooling).
- The results are in good agreement with the literature for the given alloy. Additional results obtained by XRD (lattice parameter, Full Width at Half Maximum) reveal the change in composition of the β phase as transformation proceeds. These parameters are dependent on the temperature, the phase amounts, but also the transformation conditions.
- XRD results validate the electrical resistivity method used to characterize the transformation kinetics.