Results report : Experiment MA-1917

J. BAILLIEUX, B. MALARD, D. POQUILLON

CIRIMAT – CNRS/UPS/INPT, 4 allée Emile Monso - BP 44362 - 31030 TOULOUSE cedex 04, France

Objective: Observation of α-titanium crystallographic evolution after oxygen diffusion using synchrotron X-ray diffraction

Introduction

Above 600-700°C, the oxidation rate in titanium alloys is parabolic before to transform into a linear rate. This oxidation, leads to formation of an oxide scale (OL) on the surface with an oxygen diffusion zone (ODZ) in the metal. The solubility of oxygen in the hexagonal-close-packed (hcp) structure of α-titanium can reach 30% at. During oxidation, oxygen diffusion and dissolution behind OL cause development of ODZ. The depth of the ODZ increases with increasing temperature and exposure duration. It is well known that oxygen prefers to occupy the octahedral sites of the hcp structure which leads to model diffusion with direct octahedral-to-octahedral. Because of the oxygen insertion, lattice parameters of hcp titanium are modified and it has been observed a significant increase of c parameter compared to a parameter.

The objective of this experiment was to study the effect of pure titanium oxidation on the lattice parameters. Instead of preparing and analyzing alloys with different oxygen content as it was studied before, the ODZ has been characterized directly after oxidation on pure titanium sample using x-ray synchrotron diffraction method which allows studying precisely a zone thin enough to quantify the effect of oxygen diffusion.

Experiment

For our investigation, we chose the Ti50A which is a commercially pure titanium alloy (ASTM grade 2). Four parallelepiped samples ($7mm_{rolling\ direction}$×5mm×2mm) has been cut in the sheet and polished. Then the oxidation has been performed at 600°C and 700°C in a Carbolite® furnace as described in table 2:

Table 1 : Heat treatment conditions (temperature, duration and environment).

Sample	S1	S2	S3	S4
Heat treatment	700°C 52h AIR	700°C 75h AIR	600°C 100h AIR	700°C52h VACUUM

After heat treatment, the two largest parallel surfaces have been polished to remove OL and ODZ which grew on these faces. At the end, transversal observation allowed to observe the different layer of each sample as represented in figure.1.The synchrotron x-ray measurements method is also described in figure 1.

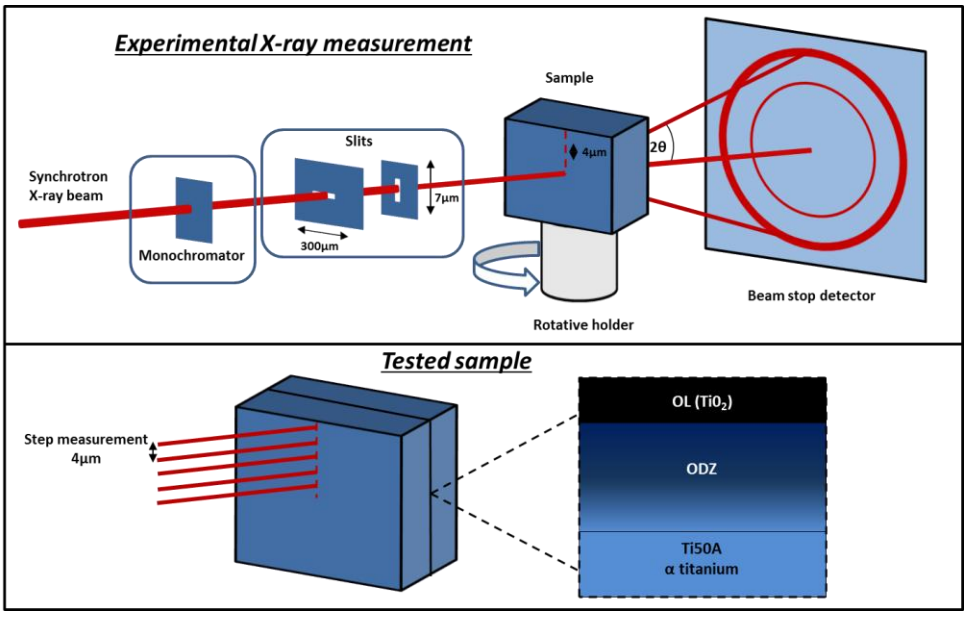

Figure 1: Synchrotron X-ray diffraction measurement and experimental method.

Data have been analyzed using the xrd software Diffrac^{plus} Topas®. Lattice parameters of hcp titanium have been calculated and refined using a full pattern matching approach.

Results

Lattice parameters c and a of hcp titanium have been calculated on each sample. As it was expected, an increase of theses parameters has been observed gradually when x-ray beam approached the oxidized surface. This fact is only true for samples which have been heat treated under air (S1, S2 and S3) but no lattice parameters modification has been noticed on sample heat treated under vacuum (S4). So the heat treatment under air is indirectly linked to lattice parameters modification with a diffusion phenomenon. Results of c/a ratio in function of oxidation distance for S1, S2 S3 and S4 are presented in fig 2.

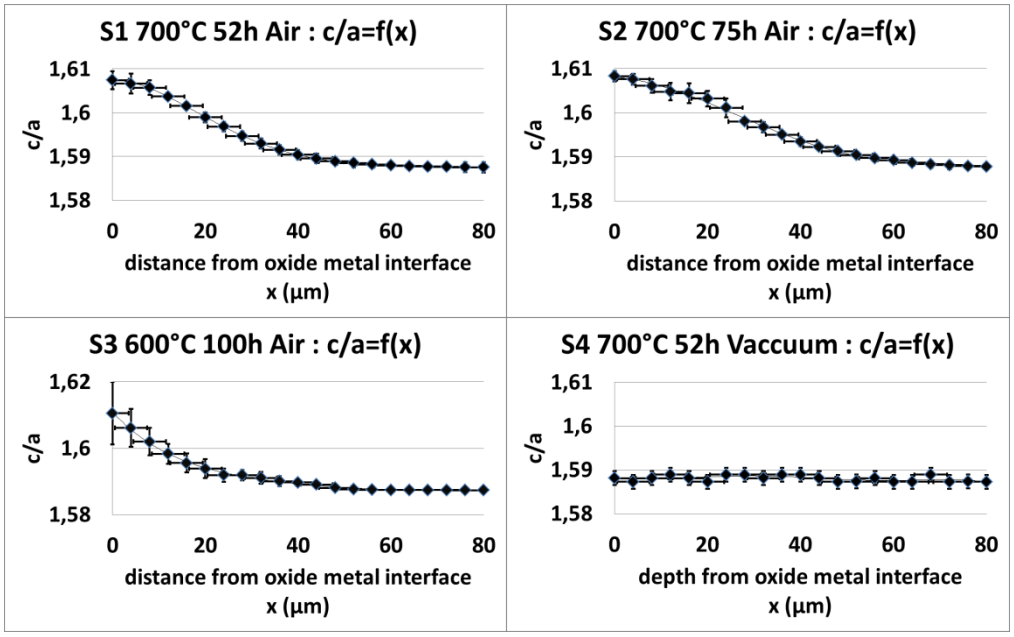

Figure 2: c/a ratio evolution of the hcp titanium in function of the oxygen diffusion depth

The temperature and time dependency of the oxygen diffusion is shown by the difference between S1 and S3 results. Even if exposure time is more important for S3, the diffusion distance is higher for S1 (50µm) than S3 (40µm) because of the higher exposure temperature. For a given temperature (700°C), the increase of the diffusion distance is confirmed when exposure time increase (60µm for S2). For each sample oxidized, ratio c/a and volume of hcp increase as it gets closer to the surface. Compared to lattice parameter a, a significant increase of lattice parameter c is observed. At the oxide/metal interface, ratio c/a seems to stabilize to a limit value which depends on temperature oxidation.

Acknowledgement

We gratefully acknowledge the ESRF for the welcome and Jonathan Wrigh for his help with synchrotron measurement.

Conclusion

Synchrotron x-ray diffraction allowed us to observe lattice parameters modification directly after the oxidation of titanium sample. The significant increase of c parameter as it gets closer to the surface oxidized confirms the oxygen diffusion and insertion through the octahedral sites of the hcp structure. This diffusion is directly linked to the hcp structure deformation which increases hardness consequent of dislocation and interstitial oxygen interaction.