

11. PARTICIPATION IN THE FUSION TECHNOLOGY PROGRAMME¹

E. Alves (Head), A. Paúl, L.C. Alves, M.R. da Silva, N. Franco

11.1. INTRODUCTION

The Portuguese participation in the Fusion Technology Program was focused in 2005 on the *micro-structure characterisation of ODS-RAFMs steels and titanium beryllide samples*, both candidate materials for the ITER and DEMO nuclear fusion reactors.

Three different Eurofer-ODS samples have been characterized: 5 and 10 mm thick plate fabricated by CEA and 12 mm disk fabricated by CRPP. Samples were analysed before and after annealing experiments and data comparison was carried out.

Two titanium beryllide alloys with a nominal composition of Be-5 at % Ti and Be-7 at % Ti were produced at JAERI, Japan, and its structural stability and oxidation behaviour under air annealing was followed.

11.2. TW5-TTMS-006: HIGH PERFORMANCE STEELS DELIVERABLE D2: CHARACTERIZATION OF REFERENCE EU ODS-EUROFER BATCH.

Small pieces were cut from the as received material and analysed before and after the annealing conditions. The samples were initially heated at 700 °C during several hours in a He/0.1% H gas mixture followed by long-term annealing also, at 700 °C, in an oxidizing atmosphere (dry air). The total annealing time was of 5000 h. Before and after the aging procedure the samples were characterized by means of SEM, TEM, XRD and nuclear microprobe techniques in order to detect changes in the microstructure. The elemental distribution maps obtained after the heat treatment at the edge of the samples are shown in Figure 11.1. For the CRPP sample the oxidation altered the surface layer to an extension of 30 µm and presents a bilayered structure with very sharp interfaces (Figure 11.1a)). The layer in the oxide/atmosphere interface has an extension of ~15 µm where Cr is the major metal component. This layer also indicates the diffusion of Mn (~3 wt.%) and strong Fe depletion (~2 wt.%). The innermost layer in the metal/oxide interface also has ~15 µm thickness and a main metal composition of Cr and Mn with a strong increment in the Mn content, reaching a 2:1 ratio for the Cr/Mn amount. In this layer there is also depletion on Fe, W and Y. The oxidized layer of the 5 mm thick CEA sample has a much larger and irregular

extension than the one observed for the CRPP sample, reaching 120 µm (Figure 11.1b)). The scale formed in this sample doesn't have the same multilayer structure seen in the CRPP specimen and the interface separating the scale and bulk has an irregular shape. Mainly Cr forms the outer region but there is presence of Fe in the inner parts of the scale. Besides, Y has an uneven spatial distribution with some Y segregation to the mid scale.

Although the studied alloys have the same nominal composition, fabrication route and heat treatment, SEM, TEM and XRD analysis also reveal that the samples present different microstructures and appreciable differences in the ferritic-martensitic phase transformation temperatures as well as on the distribution of the yttria dispersion.

11.3. MATERIAL CHARACTERIZATION USING NUCLEAR TECHNIQUES: STRUCTURAL AND OXIDATION STUDIES OF BERYLLIUM ALLOYS.

Small pieces (about 2 mm³) were cut from the as-cast material and studied before and after annealing at 600 and 800 °C in dry air. Both high resolution x-ray diffraction and micro-beam techniques were used to follow the evolution of the composition and crystalline phases as well as the microstructure. The impurity content and oxide layer were monitored with Rutherford Backscattering Spectrometry (RBS) and Particle Induced X-ray Emission (PIXE) techniques. The image shown in Figure 11.2 for the Be-5at%Ti alloy shows a granular distribution of the beryllide phase surrounded by a beryllium rich region containing several impurities as revealed by PIXE. The Be-7at%Ti is more homogeneous as shown by the titanium distribution map (Figure 11.2) and the impurity content is significantly lower compared to the other alloy. On the other hand the presence of uranium in both types of compounds needs to be taken into account when applications are foreseen.

The identification of the beryllide phases present in the samples was done using high resolution X-ray diffraction. The Be₁₂Ti intermetallic compound is present in both Be-5at%Ti and Be-7at%Ti alloys whereas the Be₁₀Ti compound could only be detected in the Be-7at%Ti alloy.

¹ Work carried out by "Instituto Tecnológico e Nuclear" (ITN).

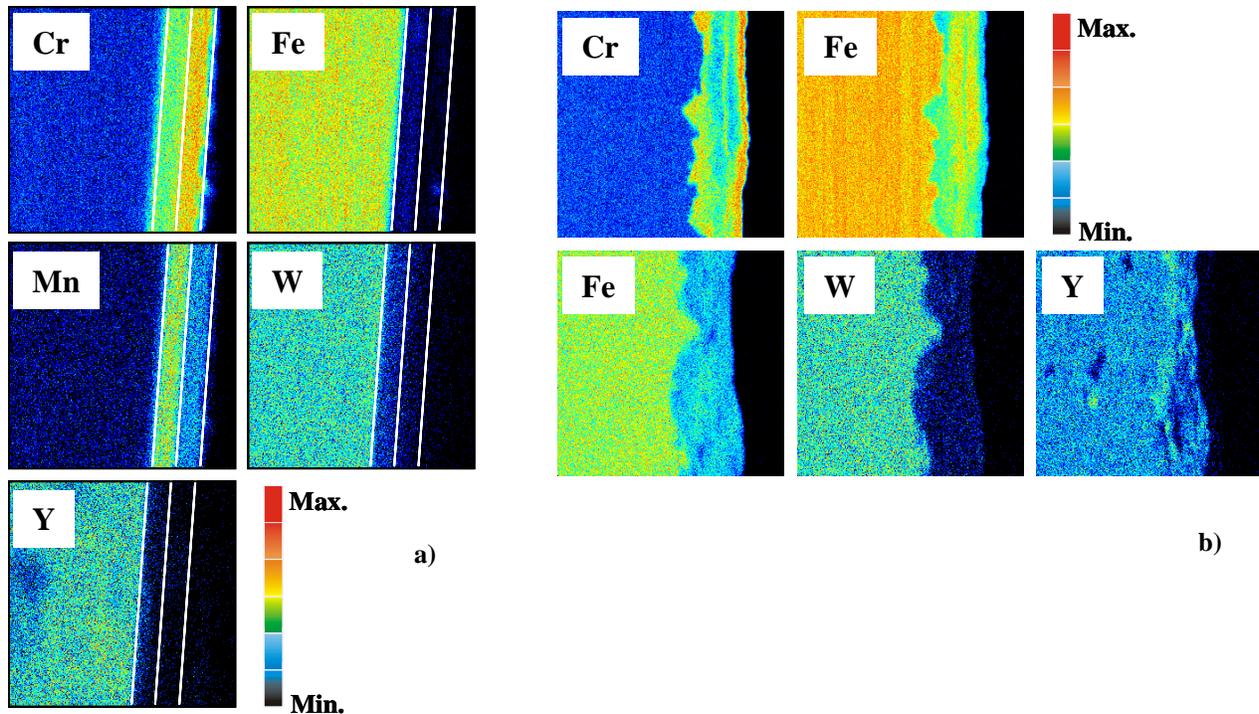


Figure 11.1 - Elemental distribution maps obtained for the CRPP sample after the thermal treatment, in a cross section near the sample surface. The maps have dimension of $130 \times 130 \mu\text{m}^2$. The white lines shown in all the elemental distribution maps indicate the layers referred in the text and are used to guide the eye; b) - Elemental distribution maps obtained for the 5 mm thick CEA sample after the thermal treatment, in a cross section near the sample surface. The maps have dimension of $530 \times 530 \mu\text{m}^2$. The elemental maps at the bottom row of the figure 10.1b) were obtained in a position different from the one used to obtain the maps shown in the top row. In both rows the Fe map is shown as a reference

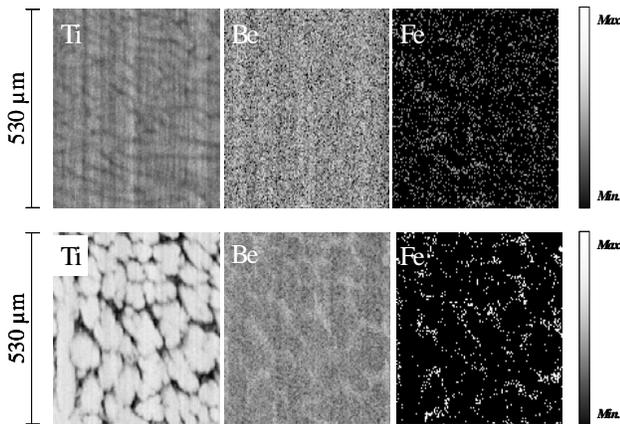


Figure 11.2 - $530 \times 530 \mu\text{m}^2$ Ti, Be and Fe elemental maps obtained from as cast Be-7at % Ti (top) and Be-5at % Ti (bottom) compounds.

The thermal stability of the intermetallic beryllides was studied after annealing at $800 \text{ }^\circ\text{C}$ in a tubular furnace with a dry-air atmosphere and in-situ up to $750 \text{ }^\circ\text{C}$ in vacuum. The changes were not pronounced and only for the Be-7 at % Ti compound the presence of the $\text{Be}_{17}\text{Ti}_2$ could not be excluded.

The oxidation of the beryllides was studied in the range of 600 to $800 \text{ }^\circ\text{C}$ in dry air atmosphere but only BeO could be identified.

Ion beam RBS technique was also used to extract information on the oxygen profile. We observe that before annealing only a small peak indicating the presence of oxygen was found in the beryllium rich regions of the samples. The oxygen profiles after the annealing are shown in Figure 11.3; Figure 11.3a shows the profile obtained in a Ti rich region for the Be-5 at % Ti alloy. As expected the oxidation process starts at low temperature and a pronounced oxygen peak at the surface region is visible immediately after 1h annealing at $600 \text{ }^\circ\text{C}$. Further annealing at $800 \text{ }^\circ\text{C}$ almost doubles the peak intensity and a broadening into deeper regions. Extending the annealing for longer time the height of the peak remains constant but the profile continues to widen, indicating the diffusion of oxygen into the bulk. The analysis in the Ti depleted region (Figure 11.3b) indicates higher oxygen content concentration.

These results suggest an enhancement of the oxidation rate in the Be rich region. For the Be-7 at % Ti the oxidation is clearly inhibited in the Ti rich regions of the sample (Figure 11.3c). The difference in the oxidation rate for the Be-5 at % Ti and Be-7 at % Ti is most probably related with the different microstructure of these alloys.

The more homogeneous distribution of Ti in Be-7 at % Ti could retard or prevent the oxidation. On the contrary the intra grain Be rich regions in Be-5 at % Ti could enhance the oxygen diffusion.

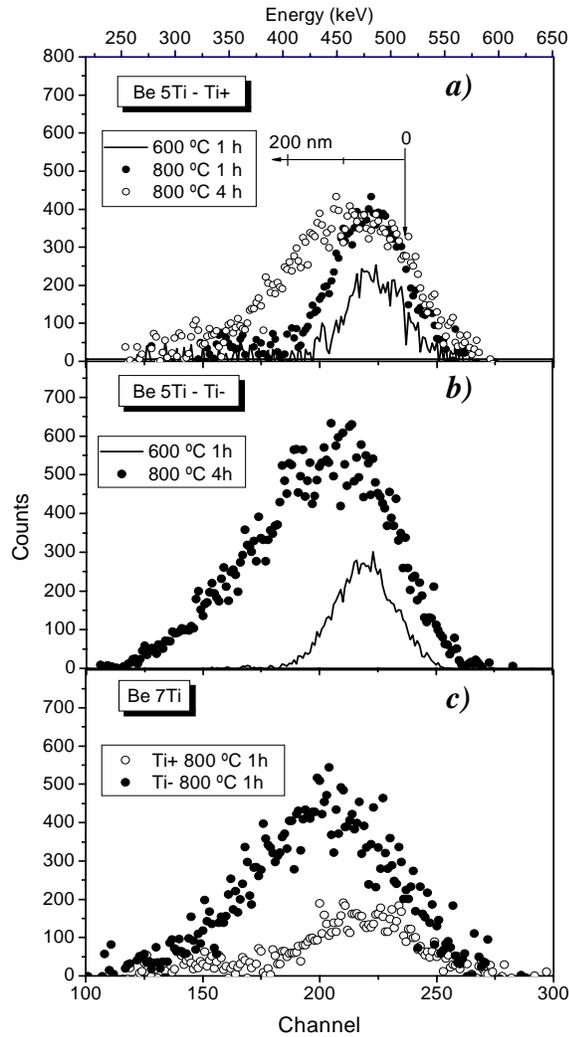


Figure 11.3 – RBS oxygen profiles obtained from a $Be_{12}Ti$ region (Ti+) (a) and a Ti depleted region (Ti-) (beryllium rich) (b) for the Be-5 % Ti alloy after annealing. The oxygen profiles for the Be-7% Ti alloy are shown in (c).