12. PARTICIPATION IN THE FUSION TECHNOLOGY PROGRAMME¹

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12.1. INTRODUCTION

The participation of the Association EURATOM/IST in the 2004 Fusion Technology Programme was concentrated on the following projects:

- TW3-TTMA-001 Structural materials: SiC/SiC ceramic composites. Deliverable D1: Morphological characterization and impurity studies of SiC/SiC composites;
- TW4- TPP-ERCAR Characterisation of carbon erosion and properties of plasma exposed carbon PFCs under ITER relevant conditions. Deliverable: Characterisation of the surface morphology of ASDEX-Upgrade tiles that have been exposed to large plasma fluences (> 10²⁶ m⁻²);
- TW4: TTBB-005 Helium cooled pebble bed: Breeder and neutron multiplier materials. Deliverable 5: Characterization of two Be₁₂Ti composites;
- Study of beryllides stability, carried out in the frame of underlying technology.

12.2 TW3-TTMA-001: DELIVERABLE D1: MORPHOLOGICAL CHARACTERIZATION AND IMPURITY STUDIES OF SIC/SIC COMPOSITES

One of the main needs in fusion technology is the development of structural materials capable to operate at high temperatures under intense irradiation fields. SiC/SiC composites appear to be the appropriate solution but these materials exhibit under irradiation a loss of their thermal properties. Other key issue in the development of these composite materials is the purity level since the presence of trace impurities with high neutron cross sections could prevent its use.

This task concerns the analysis of two types of SiC/SiC composites produced at ENEA, Italy, with respectively Hi-Nicalon and Tyranno fibres. The surface topography was studied by electron and optical microscopy and ion beam techniques. Particle induced X-ray emission and Rutherford backscattering spectroscopy have been used to study the impurity content and distribution.

The results show that the analysed SiC Hi-Nicalon composites samples have a low degree of contaminants. In particular coated samples present a very high degree of purity with only trace amounts of Ca, Fe, Ni, Cu and Zn (Figure 12.1). The Al, if present, is below the detection limits of the used techniques.



Figure 12.1 – PIXE (upper) and RBS (lower) spectra obtained from sample A during a $2.6x2.6 \text{ mm}^2$ scan.

The detected elements are not uniformly distributed (Figure 12.2). In fact those impurities are concentrated in clusters as found when performing a more detailed scan.

The SiC Tyranno samples were also studied and it was observed a higher elemental contamination in the sample cross-section analysis. However, this result is not due to the purity of the materials used for manufacturing the SiC composite but rather to some surface contamination during the samples cutting procedure. The main surface contaminating elements that have been detected are Ba,

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

Ca, and O, probably from the oil used during cutting and also Fe from the cutting tool. It is also evident the existence of zones with large cracks that should pose heavy porosity problems for its intended use in a fusion reactor.



Figure 12.2 – Elemental distribution maps on a sample obtained after a $2.6x2.6 \text{ mm}^2$ scan.

12.3 TW4-TPP-ERCAR - DELIVERABLE: CHARACTERISATION OF THE SURFACE MORPHOLOGY OF ASDEX-UPGRADE TILES EXPOSED TO LARGE PLASMA FLUENCES (> 10^{26} m⁻²)

The interaction of plasmas with the first wall components is mainly dominated by erosion and re-deposition processes. The study of all these processes is crucial to understand the influence and behaviour of both the plasma and materials during steady-state operation and disrupting conditions. Besides the influence on the properties of plasma facing components the fuel inventory must also be considered. During the re-deposition process some of the T and H are trapped in the first wall materials.

The ITN study reports on erosion/deposition characterisation of ASDEX-Upgrade tiles thatbhave been exposed to large plasma fluences (> 10^{26} m⁻²). The major goal was to compare the surface changes for two series of Carbon tiles placed in different sites inside the chamber.

The roughness is visible in all the samples as a consequence of the ion irradiation (Figure 12.3). Moreover the pits observed in samples A and C are an indication for a stronger erosion process. This process could also be responsible, despite the roughness, for the smother surfaces of samples A and C. The higher content of T and H measured in these samples is another indication for preferential erosion/re-deposition processes along the tiles. It seems also that the erosion/re-deposition processes occur with different intensities in modules 10Z and 14Z. The AFM images allow concluding that the module 14Z has been located in a region with higher flux of particles. This explains the high concentration of T and H in the samples from this tile. However to produce any conclusive explanation of all the processes occurring it is mandatory to have reference samples for comparison and detailed knowledge of the places inside the plasma chamber.



Figure 12.3 - AFM images of sample series 10Z (left) and 14Z (right)

12.4. TW4: TTBB-005 - DELIVERABLE 5: CHARACTERIZATION OF TWO Be₁₂Ti COMPOSITES

In the water cooled blanket design there are safety concerns in the case of a loss-of-coolant accident (LOCA). The exothermal reaction between hot beryllium and steam, producing hydrogen gas (Be + H₂O \rightarrow BeO + H₂ + heat) must be prevented. At low temperatures (up to approximately 600°C) the oxidation of beryllium forms a protective oxide layer on the surface of the metallic beryllium and the oxidation rate decreases with time. When the temperature exceeds a critical value (typically around 700°C) oxidation becomes non-protective and proceeds until the beryllium is depleted. In view of the intrinsic safety of ITER and DEMO whose blanket will withstand temperatures in the 600 to 900 °C range, beryllides will appear as a viable alternative to metallic beryllium.

A detailed study of structural stability of titanium beryllides and the oxidation behaviour under air annealing has been performed. Both high resolution X-ray diffraction and micro-beam techniques were used to follow the evolution of the composition and phases. The microstructure was studied with scanning electron microscopy.

Beryllium-titanium inter-metallic compounds were produced using a nominal composition of Be-5at%Ti and Be-7at%Ti. In the as cast samples, $Be_{10}Ti$ was the major phase formed in the Be-7at%Ti sample and $Be_{12}Ti$ for the Be-5at%Ti sample. The Be-5at%Ti alloy reveals intra-grain regions with high concentration of impurities (O, Fe and Ni) and Ti depletion. During thermal treatments up to 800 °C for 1 hour the oxidation occurs preferentially at the beryllide grain boundaries, but a continuous increase of oxygen was found in the beryllide grains (Figure 12.4).

12.5. STUDY OF BERYLLIDES STABILITY

Beryllides are potential candidates to replace Be in future fusion power plants due to their improved properties. Among the beryllides one of the most promising is Ti beryllide due to its lower chemical reactivity. Although, fabrication routes and properties of Beryllium are well established a lack of knowledge still exists for beryllides.

This work presents the study of $Be_{12}Ti$ and $Be_{10}Ti$, provided by JAERI in the frame of the IEA Agreement, using a large number of techniques. A composition of Be-5at%Ti was used to produce the samples. The Ti and Fe elemental distribution maps obtained for the Be-5at%Ti are shown in Figure 12.5. Although the front face and cross section analysis reveal different type of structures, they are both composed of Ti rich phases surrounded by Be rich regions that also present higher impurity amounts of Cr, Mn, Fe, Ni and Cu. Elements such as Zr and U were also detected by the PIXE technique and seem to be homogeneously distributed all over the entire sample.



Figure 12.4 - RBS spectra obtained from a $Be_{12}Ti$ region and a Ti depleted region for the Be-5%Ti alloy before and after annealing. The Increase of the oxygen signal is evident and more pronounced on the region with low Ti content.



Cross section



Figure 12.5 Ti and Fe elemental distribution maps obtained from a Be-5at%Ti sample surface and sample cross-section analysis. (Scanned area: 530×530µm2).

The behaviour of the beryllide was followed during the annealing up to 800 °C. In situ X-ray analysis and RBS measurements were done to obtain information on phase stability and oxidation behaviour. Figure 12.6 shows the X-ray spectra obtained during the annealing at different temperatures in vacuum. It is evident the growth of the $Be_{17}Ti_2$ phase. No evidence was found for the presence of BeO phase during the annealing in vacuum.



Figure 12.6 - X-ray spectra showing the phases present in the sample during the annealing in vacuum.