12. PARTICIPATION IN THE FUSION TECHNOLOGY PROGRAMME

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12.1. INTRODUCTION

The Portuguese participation in the Fusion Technology Programme during 2003 was concentrated on the following tasks:

- TW1:TTBB–005; Deliverable D9: Surface composition investigations of Li_4SiO_4 and Li_2TiO_3 exposed to long term annealing[1];
- TW3:TTMS–006; Deliverable D4: Detailed Metallurgical Characterisation of the 2 improved ODS batches;
- TW3-TTMA-001; Morphological Characterization and Impurity Studies of SiC/SiC Composites.

12.2. TW1:TTBB–005; DELIVERABLE D9: SURFACE COMPOSITION INVESTIGATIONS OF Li_4SiO_4 AND Li_2TiO_3 EXPOSED TO LONG TERM ANNEALING

Lithium orthosilicate pebbles were analysed by nuclear microprobe techniques in order to account for surface alterations during an annealing experiment performed by the FZK team. The Li pebbles were produced from Li_2CO_3 and SiO_2 and were annealed at 970ºC during 96 days in a gas environment composed of He and 0.1% H_2. After the annealing, the pebbles batch presented two different sub-sets with varying colours: one of those sub-sets with white coloured pebbles and the other one with grey colours. Two pebbles of each sub-set were chosen for characterisation. PIXE and RBS spectra were simultaneously obtained with a 2 MeV proton beam focused to ~3 µm. A secondary electron image system (SEI) was also used for properly positioning the samples. The samples were referenced as belonging to the batch OSi-01/3-3-96. In a previous work we have analysed virgin samples of this batch, referenced as 01/3-3, that will now be used for comparison.

Si X-ray and SEI maps obtained during different beam scans from the several analysed samples are presented in Figure 12.1. The white coloured samples show a higher degree of heterogeneity partially due to higher surface irregularities. Only sample 3 presents elemental inhomogeneity as revealed by the Fe map in Figure 12.2 and is probably due to some contamination during manipulation. The obtained RBS spectra, shown in Figure 12.3, confirm that the white pebbles suffer stronger variations in the constituent elements, in particular a

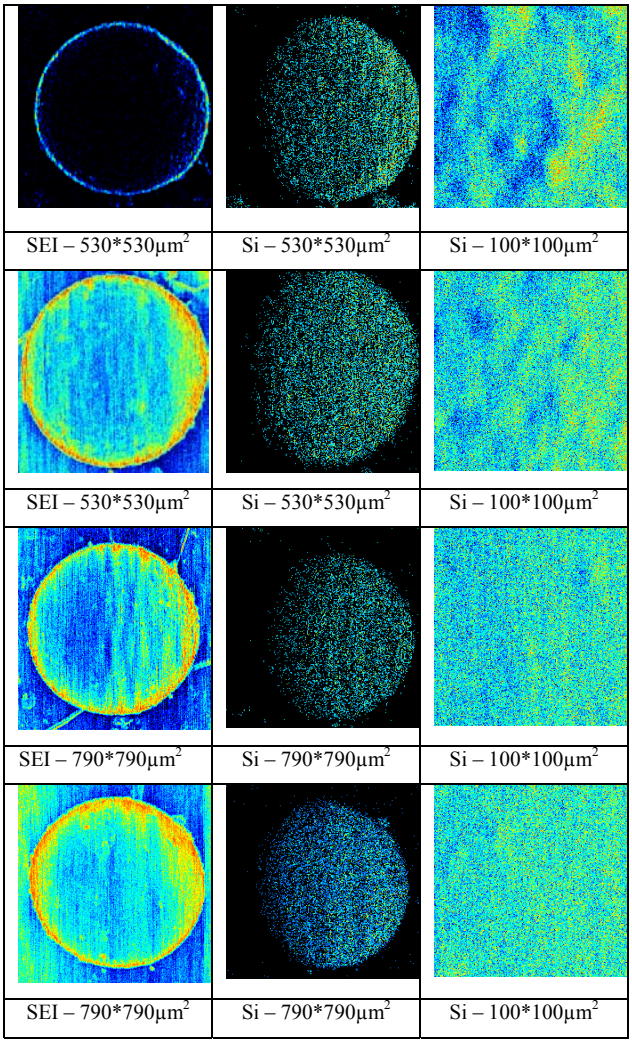

Figure 12.1 - Secondary electron images (SEI) and Si X-ray maps obtained during different amplitude scans on the four analysed pebbles: first two rows in the table (samples S1 and S2) correspond to white coloured pebbles; last two rows in the table (samples S3 and S4) are grey coloured pebbles.

general decrease in the Li average content. Comparison with the virgin sample shows that, in spite of the colour modification, the composition of the grey pebbles are much more similar to the virgin samples than the white ones.

Ca and Fe are the main impurities found in all the PIXE spectra (cf. Figure 12.4) in µg/g level

[1] As a result of a delay on pebble supply and the long annealing studies this task last more than one year.

concentrations (<30µg/g for Ca and <10µg/g for Fe). Zn and Pt that were found previously in the virgin sample were not detected in any of these samples, suggesting an initial surface contamination that was removed during the annealing procedure. On the other hand, Gd and Ca and Fe are the main impurities found in all the PIXE spectra (cf. Figure 12.4) in µg/g level concentrations (<30µg/g for Ca and <10µg/g for Fe). Zn and Pt that were found previously in the virgin sample were not detected in any of these samples, suggesting an initial surface contamination that was removed during the annealing procedure. On the other hand, Ti that could be found in some of the virgin pebbles are still present in the annealed pebbles in concentration bellow 5µg/g.

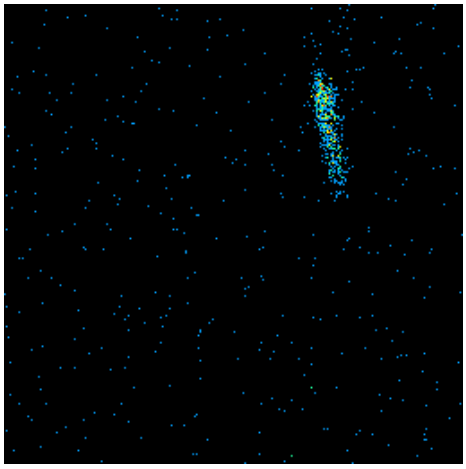

Figure 12.2 - Fe X-ray map obtained from sample S3 during a scan of 100×100 µm^2.

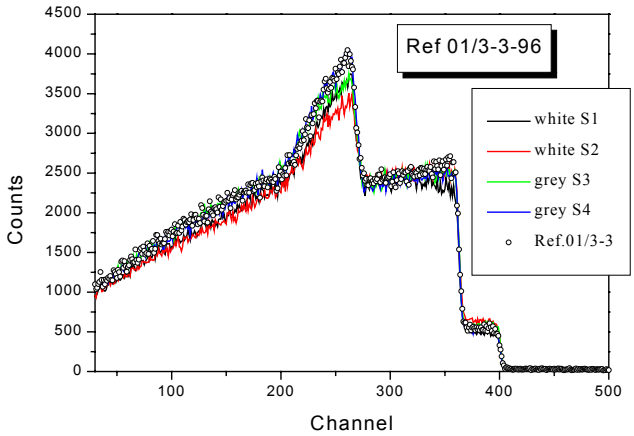

Figure 12.3 – RBS spectra obtained from the four annealed pebbles and the virgin sample.

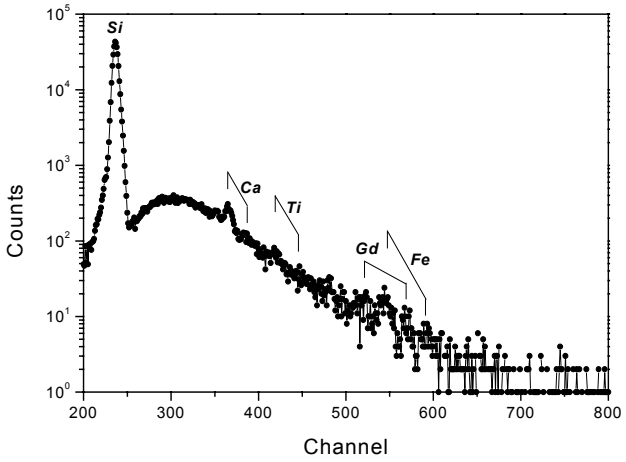

Figure 12.4 – Representative PIXE spectra of the annealed pebbles.

12.3. TW3:TTMS–006; DELIVERABLE D4: DETAILED METALLURGICAL CHARACTERISATION OF THE 2 IMPROVED ODS BATCHES

Three steel ODS samples were analysed by ion microprobe techniques in order to evaluate surface composition homogeneity. Two of the samples were obtained from CEA, Grenoble in the form of plates, one of them with 5 mm and the other with 10 mm thickness and the third sample was manufactured by CRPP, Swiss, in the form of a disk with 12 mm thickness.

The samples surface was polished and bombarded with a 2.0 MeV proton beam focused to ~3 µm, for simultaneous PIXE and RBS analysis.

The elemental distribution maps (Cr, Fe, W and Y) for each of the analysed samples are shown in Figure 12.5. It can be noticed that only the Y map presents heterogeneities that are noticed in a 260×260 µm^2 scan area.

From the above maps, location of points for quantitative analysis were chosen, some of them in the region that showed less amount of Y. The obtained results are summarized in Table 12.1.

These results are just preliminary, once only the major and minor elements were investigated, while it is expected that the trace elements contents can also be determined. A cross section sample analysis is also planned for investigating the composition of the first 50 µm. This work will continue during this year.

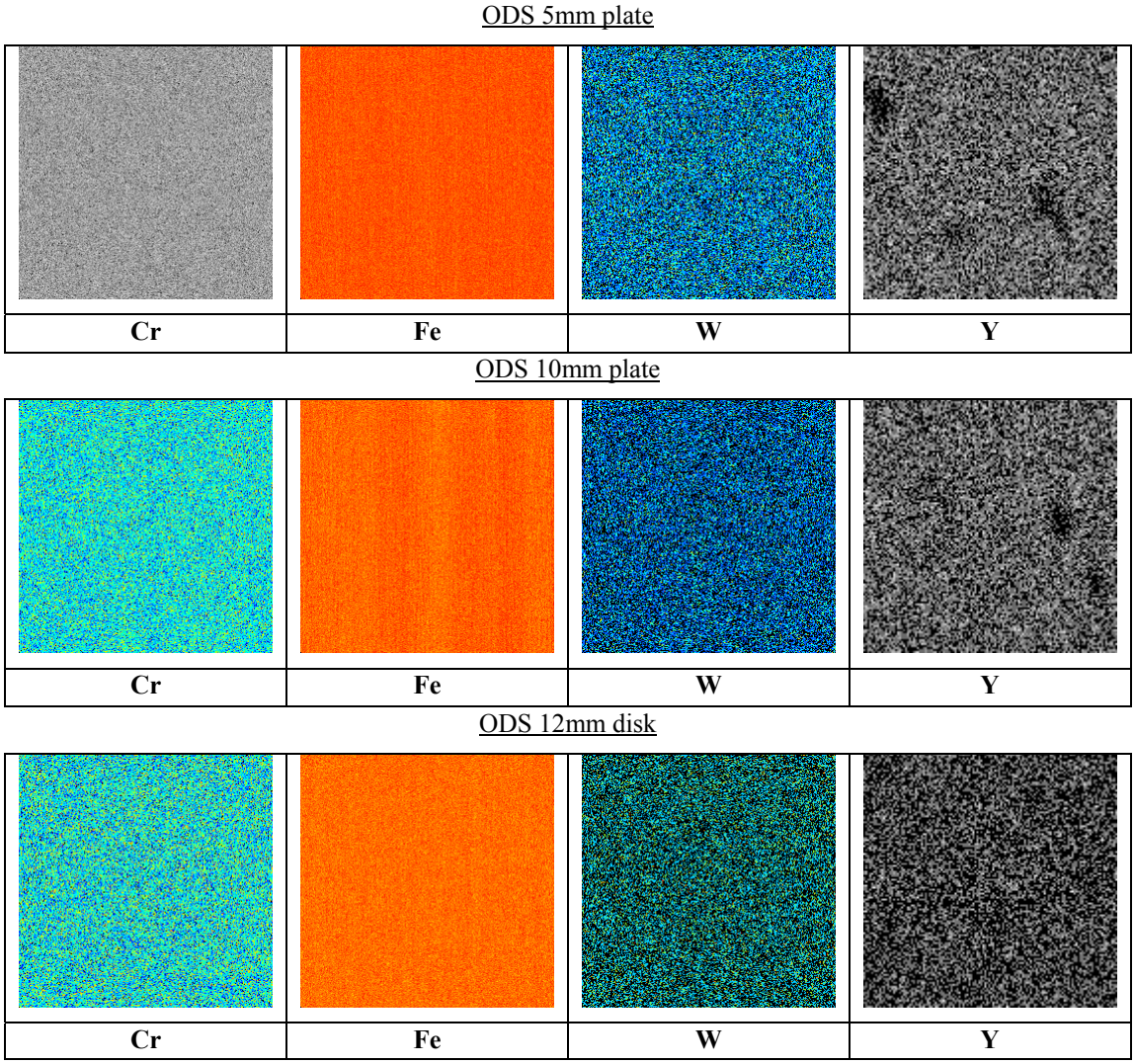

Figure 12.5 – Cr, Fe, W and Y elemental distribution maps obtained from the analised ODS samples, during a proton beam scan of 260×260 μm². The darker regions on the Y map correspond to lower contents of the element.

12.4. TW3-TTMA-001; MORPHOLOGICAL CHARACTERIZATION AND IMPURITY STUDIES OF SIC/SIC COMPOSITES.

There was a significant delay on the supply of the SiC/SiC composite. The studies of the new composites are now under way.

12.5. UNDERLYING TECHNOLOGY; SUB-TASK REPORT

One of the major drawbacks of beryllium for fusion applications it is the reactivity with steam. In the case of a loss of coolant accident (LOCA), the use of beryllium in combination with pressurised water as coolant can lead to excessive hydrogen production due to the reaction Be + H_2O = BeO + H_2 + heat. Because of the explosion hazard associated with this phenomenon, the hydrogen generation rate during a LOCA should be reduced as much as possible. Therefore, it is important to develop mitigation methods for the beryllium/steam reaction. With this in mind we start to study the influence of the presence of other elements (Ca and Al) on the reactivity of beryllium. We study the doping effect on the chemical reactivity of beryllium in steam. Beryllium samples were implanted with aluminium and calcium ions and exposed the samples to steam at 650°C in an experimental facility containing TG and mass spectrometry equipment. The ion-implantation resulted in a reduction of the chemical reactivity by a factor 2 for aluminium-doped material and by a factor 4 for calcium-doped material.

ODS 5mm plate								
	V (wt%)	Cr (wt%)	Mn (wt%)	Fe (wt%)	Ta (wt%)	W (wt%)	Y (wt%)	
	0.22	9.17	0.49	88.85	0.05	1.17	0.07	*
	0.20	9.14	0.50	89.04	0.09	1.05	0.29	
	0.22	9.11	0.52	88.99	0.09	1.08	0.27	
Average	0.22	9.14	0.50	88.96	0.08	1.10	0.21	
STD	0.01	0.02	0.01	0.08	0.02	0.05	0.10	
ODS 10mm plate								
	V (wt%)	Cr (wt%)	Mn (wt%)	Fe (wt%)	Ta (wt%)	W (wt%)	Y (wt%)	
	0.20	9.10	0.52	88.90	0.07	1.08	0.29	
	0.19	9.15	0.56	88.67	0.13	1.12	0.33	
	0.19	9.07	0.54	88.88	0.04	1.09	0.02	*
Average	0.20	9.11	0.54	88.82	0.08	1.10	0.21	
STD	0.00	0.03	0.01	0.10	0.04	0.02	0.14	
ODS 12mm disk								
	V (wt%)	Cr (wt%)	Mn (wt%)	Fe (wt%)	Ta (wt%)	W (wt%)	Y (wt%)	
	0.19	8.96	0.40	89.07	0.09	1.12	0.30	

*Table 12.1 - Elemental concentration results obtained by PIXE, for several point analysis, as well as the average concentration and the standard deviation value. The symbol * refers to the point analysis in the observed distribution map zones were the Y content is lower.*

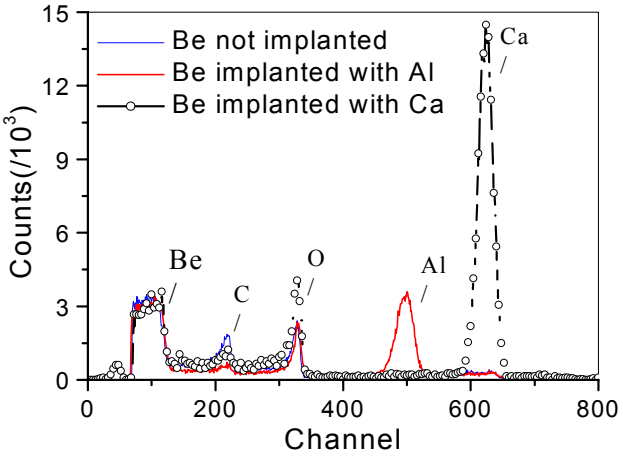

Figure 12.6 - RBS spectra of the implanted samples and a reference RBS spectrum of a non-implanted beryllium sample.

Under the underlying technology task we also continue our research activities on the chemical compatibility between Li ceramic breeders (e.g. Li_4SiO_4, Li_2TiO_3) and reactor structural materials. In this work, Eurofer samples were placed inside a Li ceramic pebble bed and kept at 600ºC under a reducing atmosphere obtained by the flow of a purging gas (He+0.1% vol. H_2) to study their compatibility.

Chemical compatibility experiments between Eurofer and Li ceramic pebbles were performed using ion beam techniques for the characterisation of the pebbles. The orthosilicate pebbles in the pebble bed that were in close contact with the Eurofer indicate a clear in-diffusion of the constituent elements from the Eurofer. The heterogeneities found also reveal the different formation and localisation of compounds. For the Li titanate pebbles, a diffusion of Eurofer elemental constituents was not found. Apart from an increase in the irregularities of the pebble surface, chemical composition alterations that could justify the pebble colour change above 500 h of exposure were not found. This could be an indication that the coloration is related with phase transformations including the formation of different Ti oxides.