12. PARTICIPATION IN THE FUSION TECHNOLOGY PROGRAMME

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12.1. INTRODUCTION

The Portuguese participation in the Fusion Technology Programme during 2002 was concentrated on the following tasks:

- TW1:TTMS-002; Deliverable D21: Impurity measurements on Eurofer 97¹
- TW1:TTBB-005; Deliverable D9: Surface composition investigations of Li4SiO4 and Li2TiO3 exposed to long term annealing¹
- TW2:TTBB–07; Deliverable D13: Report on study of the impact of irradiation on electrical conductivity of Be pebble beds²
- TW2:TTMS-002a; Deliverable D4: Eurofer microstructure characterisation: Correlation with macroscopic properties²
- TW2:TTMS-006a; Deliverable D4: Detailed Metallurgical Characterisation of the 2 improved ODS batches²

12.2. TW1:TTMS-002; DELIVERABLE D21: IMPURITY MEASUREMENTS ON EUROFER 97

Several Eurofer heats (E83694, E83698 and E83699) with different shapes were analysed with ion beam techniques (PIXE and RBS), Instrumental Neutron Activation Analysis (INAA) and Ion Cyclotron Resonance (ICR). Undesired elements in the Eurofer batches were detected in concentrations well above the radiological desired values even for some elements whose identification should be very difficult due to their expected very low contents. Figure 12.1 shows a typical PIXE spectrum revealing most of the elements presents in one of the analysed batches and reveals that Nb is well above the 10 ppb limit (>7 ppm) and the same happens for Co (limit 1 ppm but concentrations above 30 ppm were determined).

Concerning U, Eu and Ag its presence was not detected with confidence above the detection limits.

More measurements are being performed to determine with accuracy these elements. The results, although encouraging, point out the need for a better control of the trace elements in the Eurofer steel for the future fusion power plants.



Figure 12.1 - Typical PIXE spectra obtained with and without X-ray filter.

12.3. TW1:TTBB–005; DELIVERABLE D9: SURFACE COMPOSITION INVESTIGATIONS OF LI₄SIO₄ AND LI₂TIO₃ EXPOSED TO LONG TERM ANNEALING

Microbeam analysis was used to characterise Li ceramic pebbles whether they were manufactured by different procedures (orthosilicate Li pebbles with and without ⁶Li enrichment by FZK) or after annealing under different conditions (up to 3 months vacuum annealing at 970° of Li titanate pebbles by CEA; chemical compatibility of titanate or orthosilicate Li pebbles with Eurofer samples annealed for several periods of time ranging from 50h to 2000h by ENEA).

For the orthosilicate pebbles produced at FZK, the ones referenced as 01/3-3 (Am 4 in this work) reveal that the C compound layer that was found at the surface of all the other type analysed samples was successfully removed. Sample referenced as 01/2 (Am 2 in this work) was the only one to present elemental surface distribution inhomogeneities of K, that at the same time presents high concentrations of this element that is barely found in any of the other FZK analysed samples (Figure 12.2).

The nominal 20% enrichment on ${}^{6}Li$ in sample referenced as 00/2-3 (Am 1 in this work) was measured to be achieved (Figure 12.3).

The Li titanate samples annealed at CEA during different amounts of time do not present any significant

¹ Task of the 2001 Fusion Technology Programme.

² Task of the 2002 Fusion Technology Programme.

change in composition except some tendency of the Si content to increase in the zones were the SEI (secondary electron image) signal is more intense. This SEI contrast appears as a dendritic type formation (Figure 12.4), whose spatial extension increases with annealing time, endeavouring the formation of different material phases.

Chemical compatibility experiments between Eurofer and Li ceramic pebbles were performed at ENEA and ion beam techniques used at ITN for the characterisation of those pebbles. The same type of C compound layer was observed on top of the Li orthosilicate material. It was observed that this layer, that extends up to ~800nm from the pebble surface, is gradually removed as a function of annealing time. This effect is stronger for the orthosilicate pebbles in the pebble bed than for the ones that were removed directly from the surface of the Eurofer sample. The pebbles that were in close contact with the Eurofer indicate a clear diffusion into the pebbles of the constituent elements from the Eurofer. The heterogeneities found also reveal the different formation and localisation of compounds.

For the Li titatanate 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. Ce contamination of unknown origin was found in one of the pebbles.



Figure 12.3 – Obtained RBS spectra from FZK Li orthosilicate pebbles: sample Am 1 (⁶Li enriched) and Am 4 (natural ⁶Li enrichment). The spectra main difference is due to the different amount of ⁶Li and ⁷Li in the samples.

12.4. TW2:TTBB-07; DELIVERABLE D13: REPORT ON STUDY OF THE IMPACT OF IRRADIATION ON ELECTRICAL CONDUC-TIVITY OF BE PEBBLE BEDS

Several 1mm Be pebble beds were filled with different atmospheres (He+ 0.1%H₂ and Ar-) or under vacuum (P<10⁻⁶ mbar) and irradiated at the ITN Nuclear Reactor. The resistivity as function of the irradiation γ field was measured for all the pebble



Figure 12.2 – Si and K x-ray elemental maps obtained from sample Am 2 for two different scanning sizes. Lighter colours represent higher concentrations.



Fig. 12.4 – Secondary electron image (SEI) and Ti map from a Li pebble annealed for 30 days

beds. The structural and composition characterization of the pebbles reveals the presence of a certain amount of BeO distributed on the pebble external surface as well as along the grain boundaries, whose contribution is the increase of the effective electrical resistivity of the pebble bed. At room temperature, without radiation, the electrical resistivity measurements indicate the presence of two regimes for the electrical resistivity of the Be pebble bed as we have shown previously. The first one corresponds to the mechanical arrangement of the pebble bed and results in a drastic decrease in the pebble bed resistivity. The second regime is linear with the pressure and is probably related with the increase in the contact area between the pebbles under the effect of the external pressure. After a mechanical cycling the bed shows an irreversible behavior and the electrical resistivity never reaches its initial value for zero pressure.

Although it is impossible to reproduce the envisaged conditions of the fusion power reactor, experimental results on the electrical conductivity of Be pebble bed suggest that it is the purging gas present the main responsible for the conductivity in the presence of ionizing radiation. The resistivity reveals a nearly linear decrease with a very small slope with the increase of the γ dose rate (Figure 12.5). This allows us to extrapolate a value of at a typical γ dose rate

of a fusion power reactor, which is still several orders of magnitude higher than the resistivity value of RAFM steel, used as structural material, eliminating any possibility of significant electromagnetic loads in the beryllium pebble beds. According to this study the extrapolated resistivity of the Be pebble bed to real operating conditions, is high enough to prevent any meaningful current flowing in the Be pebbles in case of plasma disruptions even under irradiation conditions.



Figure 12.5 – Resistivity of a Be pebble bed filled under an atmosphere of $He+ 0.1\%H_2$ and compressed to 0.13 MPa as a function of gamma dose rate.

12.5. TW2:TTMS-002A; DELIVERABLE D4: EUROFER MICROSTRUCTURE CHARACTE-RISATION: CORRELATION WITH MACROS-COPIC PROPERTIES

Metallographic studies show the presence of non-metallic inclusions and small pores in all the Eurofer samples. The most numerous are seen in grey colour and where identified as Fe-Mn sulphides by electron microscopy (Figure 12.6).



Figure 12.6 - Backscattered electrons image of Fe-Mn sulphides (black) and Ta-rich inclusions (bright spheres), left and of Si-Ti rich inclusions, right.

The presence of Ti-rich inclusions were also found which is surprising as the chemical analysis reveals Ti levels below detection limits in all the specimens. Also Ta and Si-rich inclusions where seen during the observation in the scanning electron microscope. Those are seen as white spheres in the backscattered electron image and where detected in all the specimens.

The structure is fully lath martensite with small prior austenite grain size. No δ -ferrite was detected in any of the samples by light microscopy. Carbides are dispersed homogeneously throughout the martensitic structure. The high temperature XRD experiments show that the austenite starts to form at 755 °C and that at 780 °C the structure is fully austenitic as seen by this technique (Figure 12.7).



Figure 12.7 - 2D spectrum plot of the intensity of the austenite (left) and (right) ferrite peaks during the cooling experiments.

TW2:TTMS-006A; DELIVERABLE 12.6. D4: METALLURGICAL CHARACTE-DETAILED **RISATION OF THE 2 IMPROVED ODS BATCHES** Preliminary studies by ion beam analysis and electron microscopy were performed on Eurofer ODS alloys produced by Plansee. Samples were prepared with 0.3 and 0.5 wt% yttria. The presence of carbides and porosity was found in all the samples. Due to porosity the mechanical properties of these alloys are behind the expectations. Further and detailed studies are being carried out on new improved ODS batches produced by CRPP Swiss and CEA Grenoble.