PLEPS STUDY OF LABORATORY PRODUCED ODS ALLOYS

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1. Introduction

Structural materials of nuclear power plants (NPP), e.g. reactor pressure vessel steels, are exposed to high doses of irradiation, heat and mechanical stresses, which may reduce their lifetime during NPP operation [1-3]. Much higher radiation and thermal loads are expected in the newest generation of nuclear power plants, such as Generation IV (GEN IV) and fusion reactors, which will be operated at temperatures between 550 - 1 000 °C and will be exposed to irradiation over 100 DPA during planned lifetime, which is more than 60 years [4]. Consequently, the demands on their structural materials are much higher and so the research and development of these materials has to have significant progress in near future.

The advanced structural materials, as oxide-dispersion-strengthened (ODS) steels, are developed for application in cooling systems, reactor pressure vessel or fuel cladding of the GEN IV nuclear power plants. The ODS steels fulfill demands on radiation, thermal and mechanical resistance during operation of nuclear reactor. ODS steels have high thermal corrosion resistance based on alloying by chromium, aluminum, silicon and on formation of dispersion of stable oxides (Y_2O_3) in structure.

The experiments in this paper are focused on the laboratory produced model ODS alloys. The experimental analysis of materials at microstructural level was performed by Pulsed Low Energy Positron System and the nanoindentation system.

2. Materials preparation

Our aim was to produce model ODS alloys through the addition of different alloying elements (Table 1). Production of the powders with specific particle size was achieved by the planetary milling of the iron, chromium and yttrium powders. The material particles were placed in the bowls of the vibrating planetary mills. The balls of austenitic steels (~5mm) were also put in the bowls to intensify the grinding [5]. Finally the isopropyl alcohol was applied to avoid contact with air during milling. The ratio of the amount of powders to the amount of the balls was estimated at 1:5.

Tab. 1: Chemical composition of produced ODS alloys (%wt.)

	ODS-1	ODS-4	ODS-6
Yttrium particles	-	0.3	0.3
Chromium	-	-	9
Iron	100	bal.	bal.

According to the SEM results, the flakes of the particles with the size up to 200 μm, were produced during milling, but more homogeneous structure with smaller particle size (10-40 μm) was expected (fig.1). This effect could be influenced by the time of the milling (48 hours) or by the ratio of the powders and the stainless steel balls.

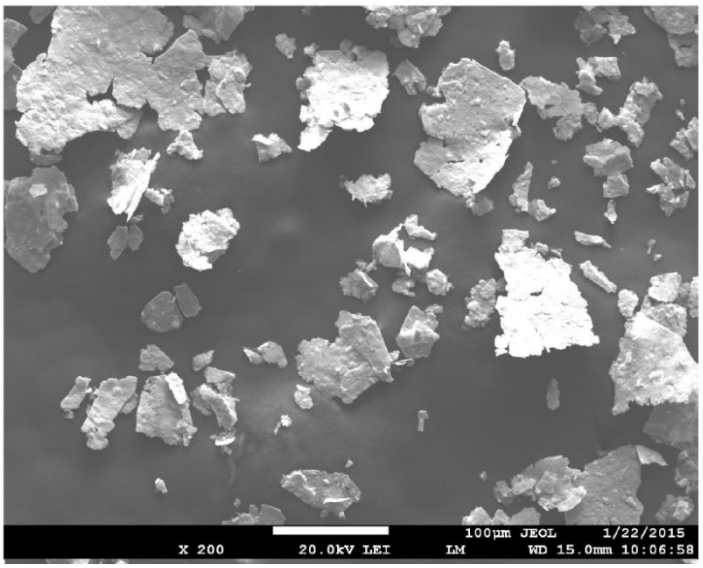

Fig. 1 Structure of the milled iron by SEM.

The next procedures consisted of drying, mechanical pressing, canning, degassing and the final HIPping (Hot Isostatic Pressing) of the mixed powders. The drying was needed as the powders were placed in isopropyl alcohol. The mechanical pressing created discs of powders, which were afterwards canned, degassed and annealed at 650 °C. The final HIP was performed at 1200 °C at the pressure of 160 MPa.

3. Experimental results

Specimens of laboratory produced ODS alloys were studied by destructive as well as non-destructive techniques. Preliminary results of testing are shown in this part and will require more detailed study.

After HIPping of the powders, the specimens in the shape of cylinder (80 x Φ25 mm) were obtained. Specimens for the Charpy and the tensile tests as well as specimens with dimensions of 10x10x1 mm were prepared. The same dimensions were used for non-destructive testing. Results achieved from the tensile tests showed that the materials are fragile as there was no elastic deformation during the tests (Fig.2).

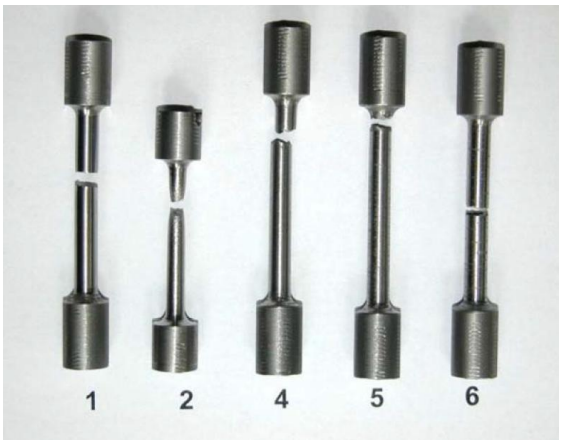

Fig. 2 Tensile tests of ODS alloys

The depth profiling of defects was performed by Pulsed Low Energy Positron System (PLEPS) at FRM II reactor in Garching (Munich) [6, 7], using positron energies, applicable for our materials, in a range from 4 keV to 18 keV corresponding to the positron penetration depth of about 50 – 525 nm. The evaluation of measured spectra was performed by PosWin code [8]. The lifetime components are usually assigned as; τ_1- positron annihilation in free volume/bulk, τ_2- positron annihilation in defects (vacancies, vacancy clusters). Therefore, increase in positron lifetime means increase in the defect size. Another parameter, intensity of positron annihilation in the free volume and defects, respectively, gives information about the amount of the annihilating positrons in studied microstructure.

Figures nr.3 and 4 show the positron lifetimes of studied specimens. The increase of lifetime values with decreasing positron energy in the region of 4-10 keV for all specimens could indicate presence of the oxidation of the surfaces as well as the positron backscattering can be present.

The increase of positron lifetimes τ_1 is recognized with addition of alloying elements. The lifetimes of specimen ODS-Fe are about 100 ps, therefore the bulk can be recognized. In alloyed specimens the lifetime τ_1 increased to about 160 ps, which can indicate presence of the dislocations.

Positron lifetimes of defects τ_2 have also increasing character. Lifetime of 200 ps was measured for ODS-Fe and about 230 ps for ODS-Fe-0.3Y_2O_3 .The intensity of the positrons annihilating in the defects was at about 70% and 40%, respectively. Addition of chromium as alloying element caused increase of τ_2 in ODS-Fe-9Cr-0.3Y_2O_3 to about 400 ps. In this case significant decrease of the intensity of defects to 5% was observed.

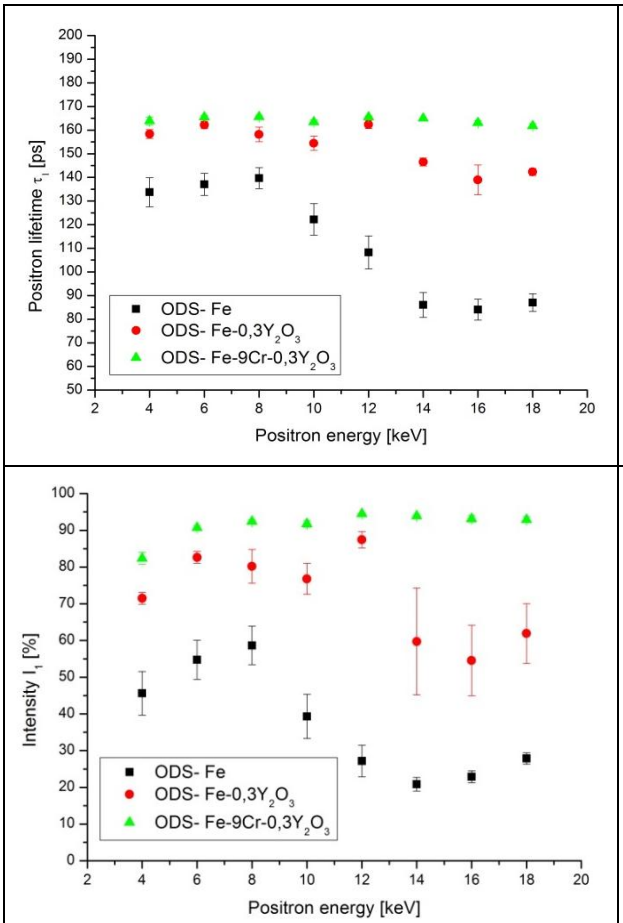

Fig. 3 *Positron lifetimes τ_1 and their intensities.*

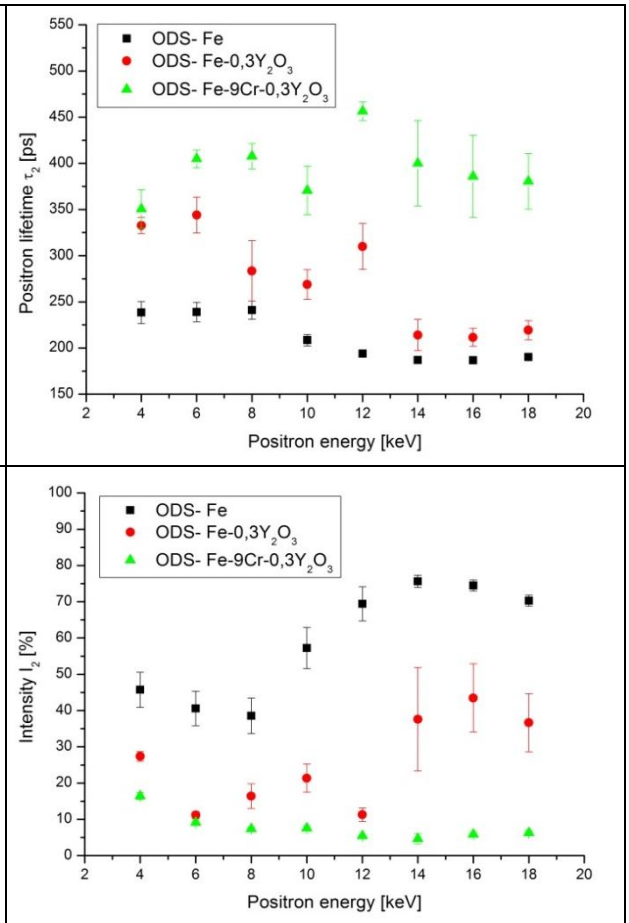

Fig. 4 *Positron lifetimes τ_2 of defects and their intensities.*

Addition of chromium as alloying element had a good influence on decrease of greater defects formation and mostly (~95%) the dislocations are present.

Investigation of these alloys and their microstructure behaviour is still in progress as well as some fitting by the PosWin software is performed. Therefore the particular components as positron lifetime in bulk and defects can provide more specific information. The results indicate that during the preparation process of alloys could be introduced some defects, especially for the ODS-Fe, where the lifetime of about 107 ps (bulk lifetime) should be measured with higher intensity.

4. Conclusion

In this paper, the laboratory produced oxide-dispersion-strengthened (ODS) model alloys were studied as one of the promising structural materials for future fission and fusion reactors applications. Specimens of ODS alloys were investigated by destructive techniques and non-destructive Pulsed Low Energy Positron System at different chemical composition.

According to the destructive testing results, especially tensile tests, was shown that the ODS alloys are brittle. EDX (Energy Dispersive X-ray) analysis showed great amount of the carbon ~11% in the structure, which can be responsible for the embrittlement of ODS alloys. Its origin is not known yet but it will be connected with the preparation procedure.

In general, the results showed that by us produced oxide dispersion strengthened alloys have their limits and till this moment do not fulfill the requirement for the commercially produced ODS steels. Some more experiments concerning the production starting with the milling have to be performed.

Acknowledgement

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