# STUDY OF OXIDE DISPERSION STRENGTHENED STEELS

***Stanislav Sojak, Jarmila Degmová, Jana Šimeg Veterníková, Jozef Snopek, Vladimír Slugeň, Martin Petriska***

*Institute of Nuclear and Physical Engineering, Faculty of Electrical Engineering and Information Technology, Slovak University of Technology, Ilkovičova 3, 812 19 Bratislava, Slovakia*
*E-mail: stanislav.sojak@stuba.sk*

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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 ismore 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 produces model ODSalloys. The experimental analysis of materials at microstructural level was performed by conventional Positron Annihilation Lifetime Spectroscopy (PALS) and the nanoindentation system at Institute of Nuclear and Physical Engineering, Slovak University of Technology.

## 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 achievedby the planetarymilling 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-2 | ODS-3 | ODS-4 | ODS-5 | ODS-6 |
|---|---|---|---|---|---|---|
| Yttriumparticles | - | 0.3 | 0.3 | 0.3 | 0.5 | 0.3 |
| Chromium | - | - | - | - | - | 9 |
| Iron | 100 | bal. | bal. | bal. | 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 (10-40μm) 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.

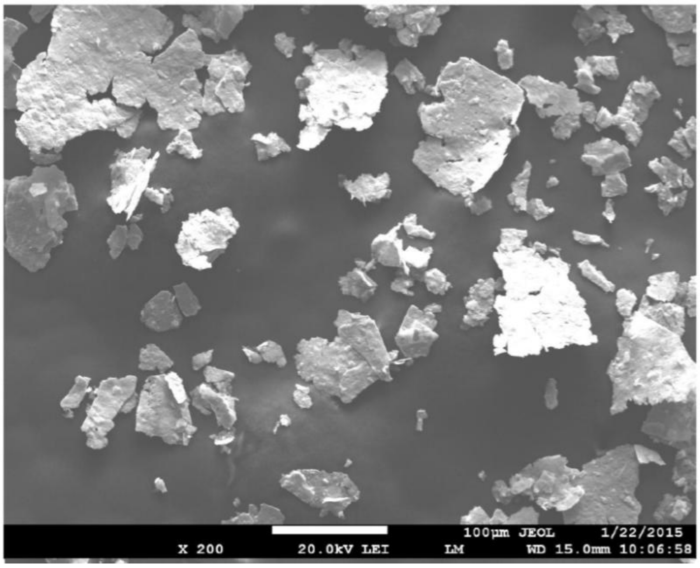


*Fig. 1 Structure of the milled iron by SEM.*

The next procedures consistedof 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 produces 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 xΦ25 mm) were obtained. Specimens for the Charpy and the tensile tests as well as specimens with dimensions of 10x10x1 mm for the nanoindentation 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*

Nanoindentation tests of hardness showed similar results for all specimens (Fig.3,4). The value of hardness of the ODS-5 could be increased by the higher amount of the yttrium oxides but it can be also caused by measurement deviation. The hardness did not change after addition of the chromium in the specimen ODS-6 but the reduced modulus slightly increased.

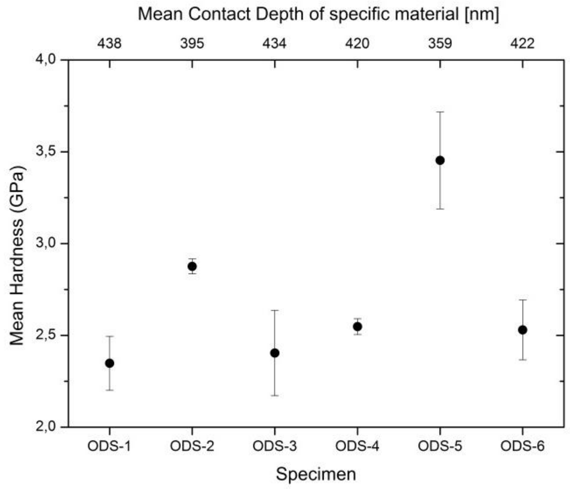


*Fig. 3 Mean hardness*

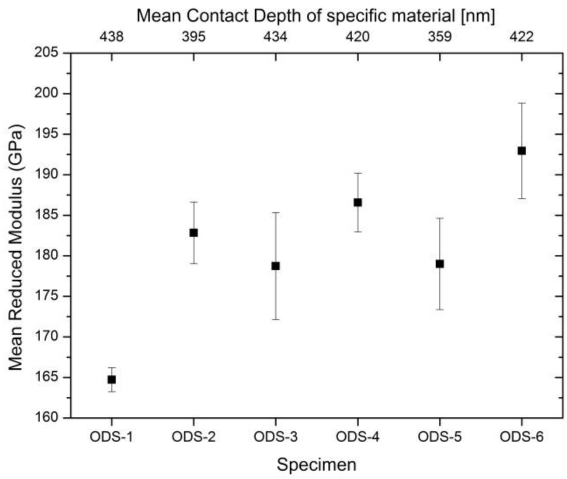


*Fig. 4 Mean Reduced Modulus*

Non-destructive testing of laboratory produced ODSalloys in the as-received state wasperformed by Positron Annihilation Lifetime Spectroscopy (PALS).Final PALS data were evaluated by LT 9 [6]. Results achieved from this software were expressed by positron lifetimes in free volume and in the defects, which define the size of the defect. Therefore, increase in positron lifetime means increase in the defect size. Another parameter (intensity of positron annihilation in the defects) from LT 9 analysis gives information about the amount of the defects in studied microstructure.

Positron lifetimes of defects in the investigated specimens are shown in Figure 3. Bulk positron lifetime in iron based alloys is usually at level of about 100-110 ps.

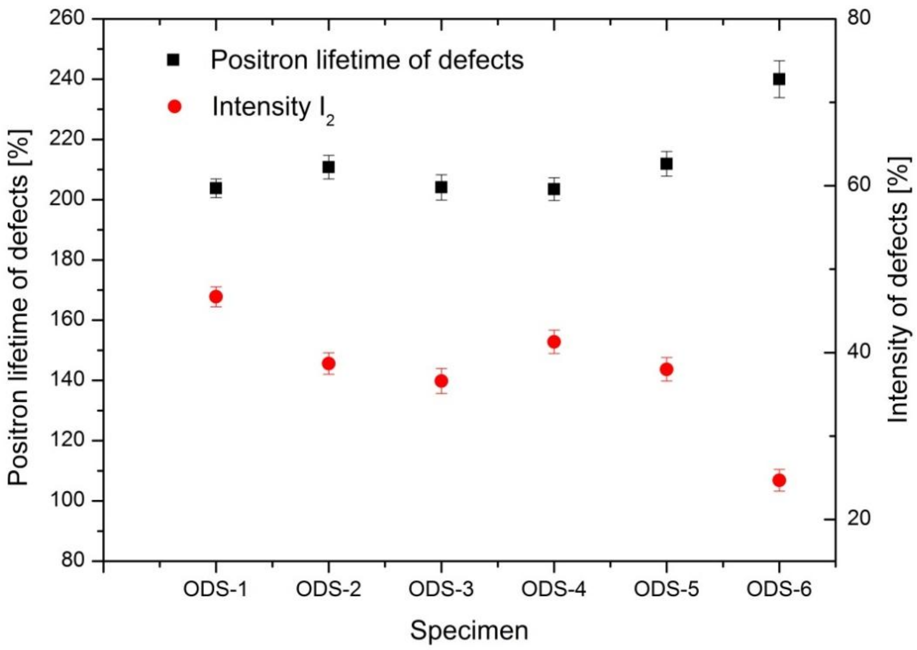


*Fig.3 Positron lifetimes of the defects and their intensities*

Positron lifetimes of defects reached level of about 205 ps. These increased values in comparison to lifetimes of well prepared as-received materials are caused by the presence of the yttrium oxides. In case of the specimen ODS-6, with chromium as alloying element, were the lifetime values at level of 240 ps. Increased lifetimes and therefore increased size of defects can be influenced by chromium, which preventsmovement of the interstitials and the recombination of the defects is less possible. Intensity of the defects was decreased for the ODS-6 (~25%) in comparison to the intensity of the rest of the specimens at level of about 40%.

## 4. Conclusion

In this paper, the laboratory oxide-dispersion-strengthened (ODS) model alloys were studied as one of the promising structural materials for future fission and fusion reactors applications. Specimens of ODSalloys were investigated by destructive nanoindentation technique and non-destructive Positron Annihilation Lifetime Spectroscopy 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.

Nanoindentation showed that the hardness and reduced modulus are at the same level for all specimens. Yttrium oxides particles increased the positron lifetime to about 205 ps. Addition of the chromium in the specimen ODS-6 increased it even more as the chromium blocks the movement of interstitials and therefore the recombination of the atoms is less possible.

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.


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