PERSPECTIVE STEELS FOR GENERATION IV AND FUSION REACTORS

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1. Introduction

Oxide dispersion strengthened (ODS) ferritic/martensitic (F/M) steels are promising materials for high temperature applications. Hardening due to oxide dispersion in steels is discussed in detail for decades. It has been studied through various computational models and some theoretical models have been already proposed but are based on simplified microstructures [1]. Experimental studies have shown that the complexity of microstructure in steels further complicates the understanding of the hardening due to oxide dispersion. The analysis of the interaction phenomena has led to the development of a number of dislocation models, which describe the mechanical properties as a function of particle size and density [2] as in the well-known ‘‘dispersed barrier hardening’’ model [3].

In this study we focus on the F/M steel Eurofer, the European candidate material for the future fusion reactor and for the strengthening we consider oxides of yttrium. The oxides of yttrium and complex yttrium titanium oxides reinforce the material by forming more or less stable obstacles to dislocations, and by promoting grain refinement by pinning grain boundaries. It appears that part of the yttrium titanium oxides particles dissolves from about 600°C while pure yttria particles are stable at least to 1000°C in the steel

The aims of this study are the following: 1) Prove the positive effect of strengthening by yttrium oxides. 2) Measure the hardness of base Eurofer and ODS version by Vickers hardness test (HV). 3) Investigate the behaviour of steels at different annealing temperatures and the changes in strength. 4) Assess defects in microstructure by Coincidence Doppler Broadening (CDB) and Positron Annihilation Lifetime Spectroscopy (PALS) at chosen annealing temperatures.

2. Experimental details

Chemical composition of studied materials is in Tab. 1. The ODS version of Eurofer has an addition of Y_2O_3 particles.

Tab. 1. *Chemical composition of Eurofer and ODS Eurofer [4].*

%	Cr	W	Mn	Ta	V	S	Si	N	C	Co	P	Y_2O_3
Eurofer	9,25	1,1	0,4	0,07	0,2	0,005	0,04	0,06	0,105	0,006	0,005	-

The measurements were carried out in the Laboratory of Low Temperature Physics, Charles University, Prague. First we made a series of HV test at room temperature to determine the integrity of the material surface. HV under different load was basically the same for individual samples however it is evident that ODS Eurofer achieved higher hardness than its base material.

To study the temperature dependence of the materials hardness, we annealed the samples at different temperatures for 50 minutes, cooled in water for 3 minutes, and removed the oxide layer using a solution of 10 vol% Nital. If needed, the samples were mechanically polished. The samples were annealed from 400- 850°C by a 50°C step. Fig. 1 presents HV as a function of annealing temperature.

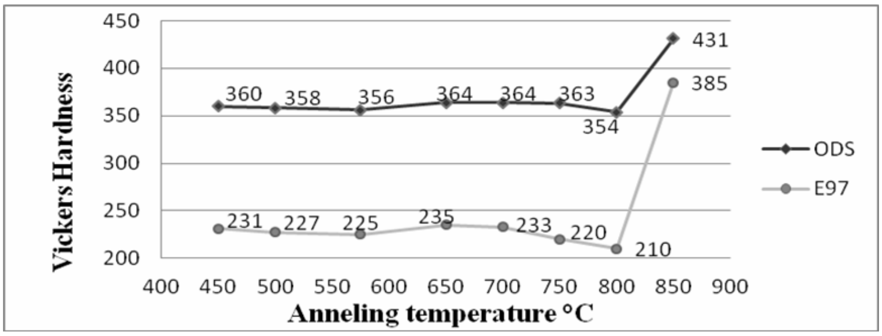


Fig. 1: *HV as a function of annealing temperature for Eurofer and ODS Eurofer.*

It should be noted that in all graphs the solid lines through the data points are provided solely as eye guide. As can be seen from Fig. 1 the alloys hardness remains almost constant to 575°C.A slight upraise can be noticed from 575-650 °C. This temperature range corresponds to the diffusion of alloying elements that leads to the precipitation of secondary precipitates. The secondary precipitation can be understood by considering the solubility limits of carbon. The solubility of carbon is less at 600 °C than at 760°C which is the tempering temperature in ferrite. Hence, the volume fraction of precipitates will be higher at 600°C as compared to that of 760°C. When the normalized and tempered steel is treated at 600°C, the carbon further precipitates as carbides to maintain the solubility limits. After exceeding 700°C, a significant decrease in hardness occurs with a minimum at 800°C. This softening could be due to coarsening of laths and precipitates resulting in the conversion of tempered martensite into coarse grained ferrite. However after 800°C the hardness rapidly increases. This behavior can be explained by an assumption that an annealing temperature of 800°C and subsequent cooling is sufficient to induce a martensitic phase transition. The microstructure is that of as quenched martensite, with thin laths and a high density of dislocation which were observed by TEM (Transmission electron microscope) [5]. In the case of ODS Eurofer the results correlate with those of its base material. ODS Eurofer presents a typical morphology of tempered martensite [5]. For the samples heat treated at more than 800°C, the HV also increases although not that significantly as in Eurofer. It is assumed that this is linked with the formation of the as quenched martensite microstructure. In Fig. 2 the results from CDB are presented. The CDB curves pertain to pure annealed iron. Curves for Eurofer show a higher contribution of annihilations with valence electrons with low momentum and lower for annihilations with core electrons with high momentum. The curve is in the area of high momentum ($p > 10 \times 10^{-3}\ m_0c$) is approximately constant, which shows that a fraction of positrons in Eurofer annihilate trapped in dislocations.

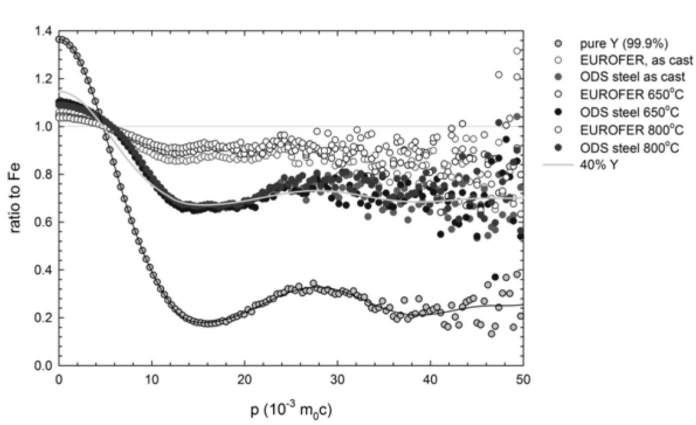
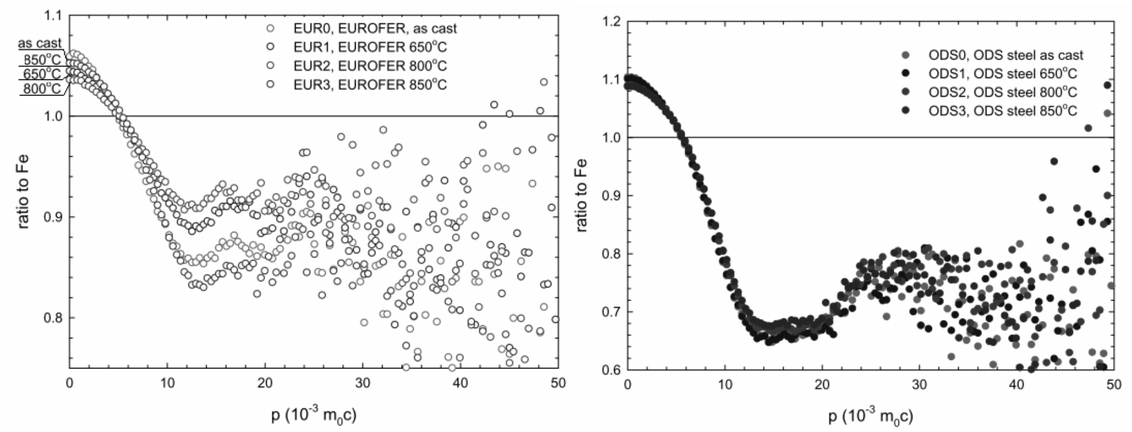


Fig. 2: *CDB results of Eurofer and ODS Eurofer as cast, 650°C and 800°C and 40%, 99.9% yttrium.*



(a) (b)

Fig.3: *CDB results in detail for Eurofer (a) and ODS Eurofer (b) as cast, 650°C, 800°C and 850°C.*

In Fig.3 is a closer visual of individual samples. From Fig.3a we can observe that until 800°C the contribution of positrons annihilating with valence electrons decreased with higher temperature suggesting that the dislocations were annealing out of the sample. However at 850°C the contribution of positrons annihilating with valence electrons raised suggesting formation of more defects. The shape of the curves for ODS Eurofer is markedly different. It is significantly lower in the high momentum area and presents a local minimum at $p \approx 15 \times 10^{-3}$ m_0c and local maximum at $p \approx 25 \times 10^{-3}$ m_0c. A similar behavior shows the curve representing pure yttrium. Therefore a considerable amount of positrons annihilate in defects that are surrounded by Y_2O_3 precipitates. It is probable that clusters of vacancies in ODS steels are in the vicinity of Y_2O_3 precipitates. The solid line in Fig. 3 represents 40% contribution of positrons which annihilate with electrons of yttrium atoms. In the high momentum area the curve correlates well with the experiment. ODS Eurofer also seems to be resistant to higher temperature since the behavior of curves did not change significantly (Fig.3b).

The PALS equipment is introduced in length in [6]. In the digital setup, ultra fast 8-bit digitizers Acqiris DC 211 is used. The timing resolution of 146 ps was achieved [6].

The particular lifetimes of positrons at various annealing temperatures and their relative intensities of components are presented in Fig. 4. The spectrum of Eurofer consists of two components: contribution of free positrons with $\tau_1 < 100$ ps and positrons trapped in dislocations with lifetime $\tau_2 \approx 155$ ps. With the temperature increasing until 800°C the density of dislocations decreases, therefore decreasing intensity I_2 of positrons trapped in dislocations. After exceeding 800^{o}C we can observe an increase in the intensity of dislocations. This corresponds well with the HV tests which assessed that at 850^{o}C the strength is higher because of the phase transformation to quenched martensite which would also explain the higher intensity of dislocations. From CDB results we can see an increase of dislocations in the structure of Eurofer which serve as obstacles for defect movement and hence reinforce the strength.

Lifetime spectrum of ODS Eurofer consists of three components: free positrons with $\tau_1 < 100$ ps, positrons captured in dislocations with $\tau_2 \approx 155$ ps and positrons captured at vacancy clusters with $\tau_3 \approx 265$ ps, which corresponds to clusters of four vacancies. In contrast to Eurofer, the intensity of defects I_2 in ODS Eurofer stays practically the same up to 850^{o}C.

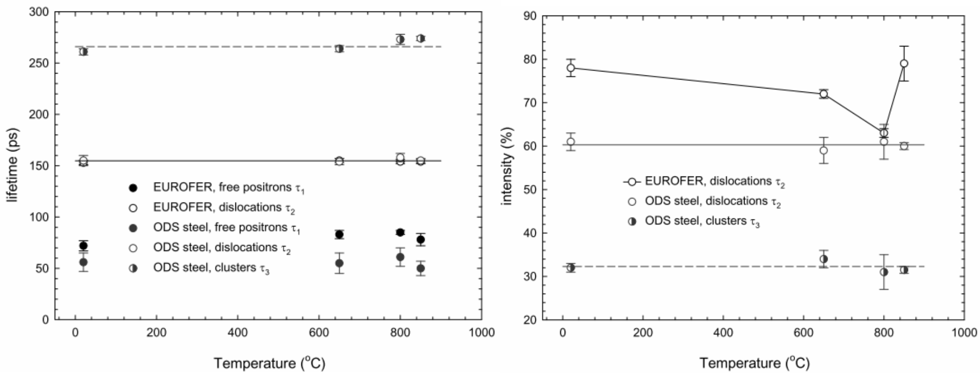


Fig. 4: *Results from PALS a) lifetimes of positrons, b) intensities.*

3. Discussions

The different behavior of ODS steel and its base material were proven by various methods. Vickers Hardness test shown significantly higher strength of the ODS steel. The PALS supports the results from HV and CDB in the full range of annealing temperatures. All methods indicate that a phase transformation to quenched martensite occurs at 850^{o}C and, in the case of Eurofer, results in a higher intensity of defects (from CDB, PALS) mainly dislocations (from PALS) which serve as barriers for movement and thus increase the strength (from HV). In ODS Eurofer the transformation also occurs (Fig.2) although a change in the behavior of the steel does not show significantly in either of the applied methods. Therefore we can assume that the ODS variant is resistant to higher temperatures than base Eurofer. From CDB we can assume that an amount of defects are in the vicinity of Y_2O_3 particles and positrons annihilate with them rather than with grain boundaries.

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4. References

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