Behaviour of Fe-Cr based alloys under neutron irradiation

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Abstract. High chromium (9-12 wt %) ferritic/martensitic steels are candidate as potential first-wall and breeding blanket structural materials for future fusion reactors. Their use for these applications requires a careful assessment of their mechanical stability under high energy neutron irradiation. In this work, the characterization of Fe-Cr model alloys with different Cr content with respect to microstructure and mechanical properties will be presented. Chromium concentration has been shown to be a key parameter which needs to be optimised in order to guarantee the best corrosion and swelling resistance.

Introduction

High-Chromium ferritic-martensitic steels are candidate structural materials for high temperature applications in fusion reactors, Accelerator Driven Systems (ADS) and advanced fission reactors in GEN IV [1]. Cr concentration has been shown to be a key parameter which needs to be optimised in order to guarantee the best corrosion and swelling resistance, together with the minimum embrittlement [2]. Furthermore, the microstructural and metallurgical changes due to neutron irradiation cause a significant alteration of their mechanical properties. Thus, their use requires an in-depth characterization of the microstructure in parallel to a rational comparison between microscopic features and macroscopic properties such as the mechanical ones. The general objective is to investigate the effect of the alloying elements on the irradiation induced defect formation and accumulation with emphasis on Cr content and to provide a well defined experimental database for model validation.

Experimental Details

The materials used in this work were Fe-Cr based model alloys, Fe-2.5Cr (2.36wt%Cr), Fe-5Cr (4.62wt%Cr), Fe-9Cr (8.39wt%Cr) and Fe-12Cr (11.62wt%Cr), and two ferritic martensitic steel, namely the conventional 9Cr1Mo steel (T91) and the European reduced activation steel EUROFER 97. Fe-Cr model alloys were cold worked and then annealed for 3 hours at 1050°C in high vacuum for austenisation and stabilization, thereafter tempered at 730 °C for about 4 hours followed by air cooling. The steel samples were delivered in normalized and tempered condition.

Tensile specimens and transmission electron microscope (TEM) specimens were neutron irradiated in BR2/ Mol-Belgium at 300 ° C to 3 different doses (0.06, 0.6 and 1.5 dpa), with neutron flux (> 1MeV) of 10^{13} n/(cm^{2}s) [3].

TEM specimens were examined using JOEL 3010 EX operating at 300 KeV, using weak beam imaging to observe defects induced by irradiation.

The tensile specimens (with nominal dimensions: length = 27 mm, gage length = 12 mm and diameter = 2.4 mm), were tested with the crosshead displacement rate 0.2 mm/min, corresponding to a strain rate of approximately 2.8×10^{-4} s^{-1}.

Results

Microstructure. Irradiation-induced microstructure changes have been studied by TEM for all materials and for each dose of irradiation. The small defects that are observed as white and black spots in all dark field images are dislocation loops [4]. Size distribution and defect density were measured, while the Burgers vectors were determined where possible. In figure 1 irradiation induced microstructures for all materials after the intermediate dose of irradiation (0.6 dpa) are shown. Most of the observed grains are oriented close to a [111] zone and only occasionally some were oriented close to a [110] zone. Therefore most of the images were taken with different {110} type diffraction vectors as indicated on the TEM micrographs.

At 0.6 dpa it can be observed that the density of defect clusters is higher than at 0.06 dpa. Dislocation loops are present in the matrix as well as near dislocation lines.

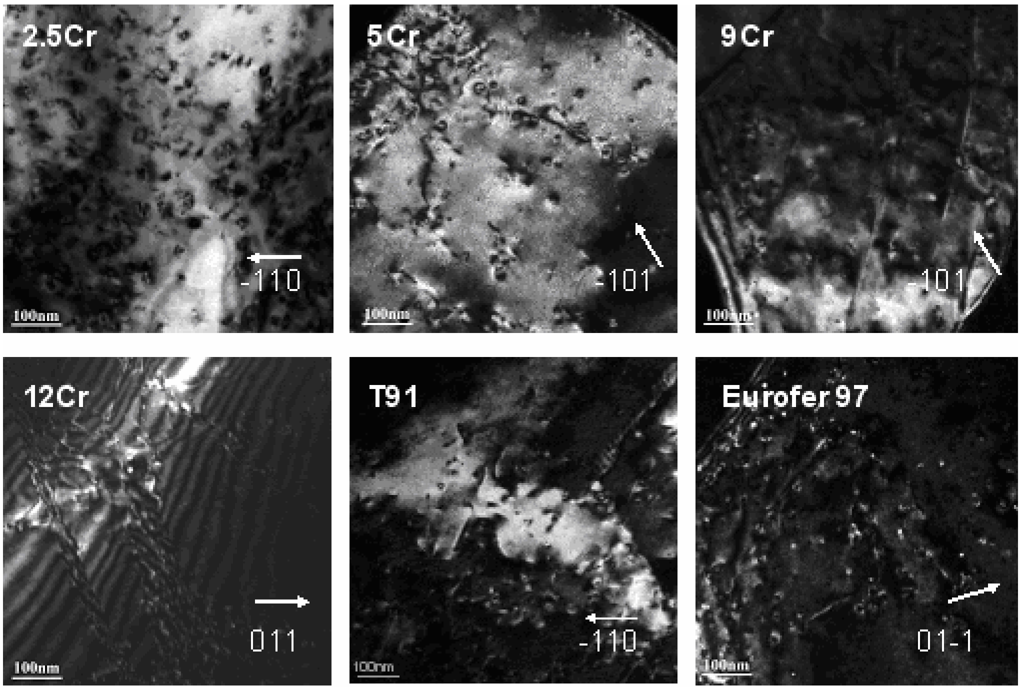


Fig. 1. Irradiation induced microstructure for Fe-Cr model alloys and steels at 0.6dpa

It was shown that the microstructure didn't change after irradiation apart from the formation of radiation induced dislocation loops [5]. The size and density have been measured and it was observed that the size of cluster loops increases with dose, but decreases with Cr concentration. The density of the defect clusters increases with doses up to 0.6 dpa, but tends to saturate at higher doses. Dislocation loops observed in Eurofer 97 are much larger than in T91 and model alloys at high dose [6,7]. Defect density and loop size for all materials at all doses of irradiation are summarised in Fig. 2. The filled numbers in the graph refer to the defect density and the values are indicated on the left Y-axis. It shows how the defect density decreases with increasing Cr concentration and that a maximal density is reached at a dose of 0.6 dpa, after which the density of defects seems to saturate. The open symbols refer to the loop size and the values are shown on the right Y-axis. It was observed that the size increases with irradiation dose but decreases with Cr concentration.

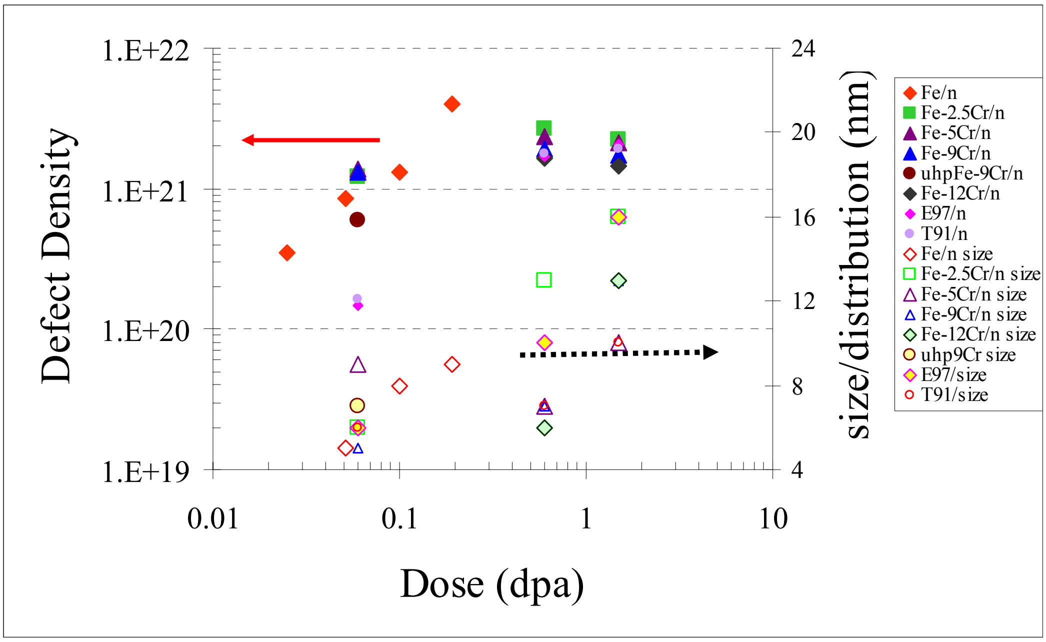
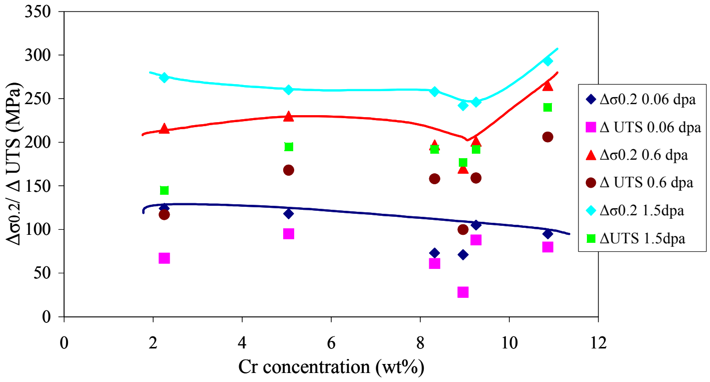


Fig. 2. Density and size dependence on irradiation dose for Fe, FeCr-model alloys and steels under investigation.

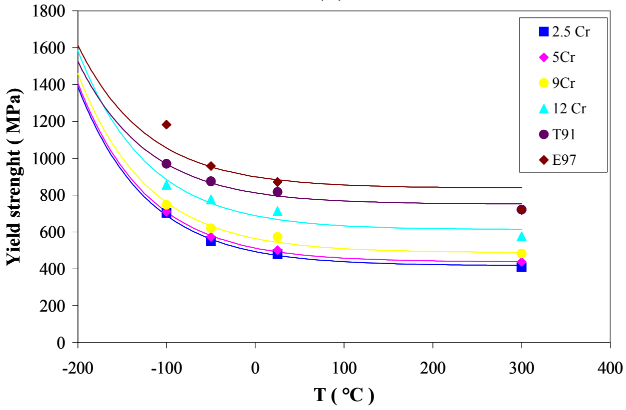
Tensile properties. Tensile tests for all doses of irradiation have been performed in the same temperature range (from -160°C to 300°C). The presence of Cr strongly influences radiation-induced hardening already at 0.06 dpa. But at higher doses, the effect of hardening is more evident and a minimum is found around 9 % Cr. In terms of ductility after irradiation, the model alloys are more ductile than the steels, but they are less tough, as it was observed before irradiation as well [6]. Reduction of uniform and total elongation starts to be more pronounced at 0.6 dpa. Yield strength for model alloys increases with increasing Cr concentration. However at this dose, it is observed that the steels T91 and Eurofer 97 do not have similar behaviour as was observed at 0.06dpa. For Eurofer 97, after reaching the yield, softening is observed, while in case of T91 retains some work hardening or uniform elongation [7,8].

Hardening dependence with Cr concentration, expressed as $\Delta\sigma$ and ΔUTS is shown in Fig. 3a.

Yield strength dependence with temperature after irradiation at 1.5 dpa is presented in Fig. 3b. Values of tensile strengths have been fitted as a function of test temperature using the empirical formula ($\sigma_{ath}+\sigma_0 e^{(-\alpha T)}$), where σ_0 and α are regression coefficients and σ_{ath} is the athermal stress calculated from the fitting. It can be noticed that the athermal stresses increase with Cr concentration. Ferritic martensitic steels have different temperature dependence of the yield strength compared to the model alloys. According to this it is evident that these two steels have different hardening behaviour; the athermal part seems to be similar, while the temperature dependent stress is different. However microstructural observations at the 3 doses of irradiation show that after 0.6 dpa the density of defects induced by irradiation starts to saturate. Therefore an increase of athermal stresses is more evident at this highest dose of irradiation [9].



(a)



(b)

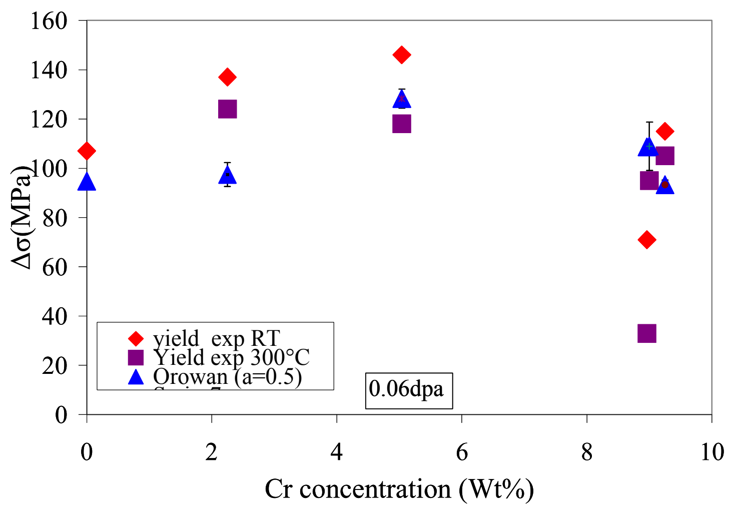
Fig. 3. a) Irradiation hardening dependence with Cr concentration at 0.06 dpa, 0.6 dpa and 1.5 dpa and b) yield strength dependence with temperature at 1.5 dpa

Discussion

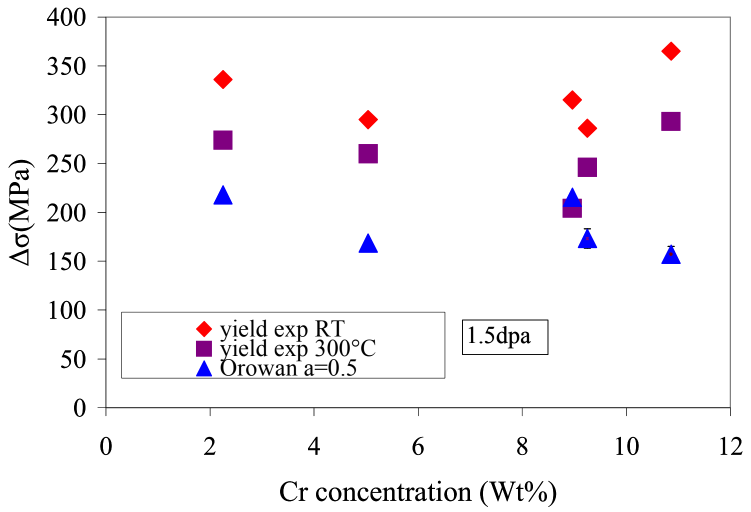
Hardening mechanisms have been studied by correlating microstructure observations from TEM to mechanical property measurements using an Orowan-type mechanism. The Orowan model is an important contribution to the theory of yield stress of alloys containing non-shearable particles. The Orowan model is given by the following Eq. 1 [10]:

$$\Delta\sigma_y = M\alpha\mu b(Nd)^{½} \qquad (1)$$

where the Taylor coefficient is M=3.06, α=0.3-0.5 represents the strength of obstacles, the shear modulus is $\mu=8.3\mathrm{x}10^4$ MPa, and the Burgers vector is b=0.286 nm, N is the density of defects and d is the defect size. Fig. 4. shows the hardening measured by tensile tests and calculated using the Orowan relationship as an example for the lowest and the highest doses. For all materials and doses of irradiation, α was taken to be equal to 0.5. The predicted hardening from the Orowan mechanism is in good agreement with the experimental values measured in tensile tests for the lowest dose of 0.06dpa [11]. This observation clearly shows that the loops are the features that are contributing to Δσ and their strength is a function of their size. At higher doses it can be seen that the visible loops cannot be the only responsible for the measured irradiation induced hardening. In fact, the hardening predicted by the Orowan mechanism decreases at higher doses, while hardening measured by tensile tests increases with dose and Cr concentration [7]. A relative minimum is found around 9% Cr, while hardening drastically increases for the 12 Cr alloy.



(a)



(b)

Fig. 4. Hardening effect and comparison of the experimental results with those calculated using the Orowan expression for (a) 0.6dpa and (b) 1.5dpadose.

Conclusions

The microstructure and the tensile properties of irradiated Fe-Cr model alloys and ferritic martensitic steels were presented after irradiation at 300°C three different doses. The effect of dose and Cr concentration up to tensile properties and microstructure of model Fe alloys has been studied. It was shown that the microstructure didn't change after irradiation apart from the formation of radiation induced dislocation loops. The size and density have been measured and it was observed that the size of cluster loops increases with dose, but decreases with Cr concentration. The density of the defect clusters increases with doses up to 0.6 dpa, but tends to saturate at higher doses. For the tensile properties it is clearly shown that the presence of Cr strongly influences hardening versus dose. Hardening mechanisms have been studied by correlating microstructural observations from TEM to tensile property measurements using an Orowan-type mechanism. It can be seen that the Orowan mechanism is valid for low doses of irradiation, while by increasing doses there are significant differences. It is shown that the observed loops are the features that are contributing to $\Delta\sigma$.

References

[1] R. L. Klueh and Donald R. Harries " High-Chromium Ferritic and Martensitic steels for Nuclear Application " 2001, Library of Congress Cataloguing in Publication data by American Society for Testing and Materials (ASTM).

[2] A. Kohyama, A. Hishinuma, D. S. Gelles, et al, J. Nucl. Mater. 233-237 (1996) 138-14.

[3] M. Weber "Mire-Cr irradiation report", NT.57/F040112/0/MW/MM/AA.rw, SCK•CEN, internal report 2004.

[4] M. L. Jenkins, M. A. Kirk , "Characterisation of Radiation Damage by Transmission Electron Microscopy " Institute of Physics Series in Microscopy in Materials Science, 2001.

[5] M. Matijasevic, A. Almazouzi, "Modellisation of irradiation effects: Modeling oriented experiments on Fe-Cr model alloys ", SCK•CEN-R-4196, 2005.

[6] M. Matijasevic, A. Almazouzi, "Effect of Cr on the mechanical properties of Fe-Cr model alloys ", J. Nucl. Mater., submitted.

[7] M. Matijasevic, A. Almazouzi, " Effect of the chemical composition on the mechanical and microstructural behaviour of Fe-9Cr steels after n-irradiation at 300°C", J. Nucl. Mater., to be published.

[8] M. Matijasevic, E. Lucon, A. Almazouzi, "Behavior of ferritic-martensitic steels after n-irradiation at 200°C and 300°C ", J. Nucl. Mater., submitted.

[9] M. Matijasevic, Ph.D. UGhent (2007).

[10] A. L. Bement, Fundamental materials problems in nuclear reactors, in: Proceedings of the ICSMA 2 Conference, vol. 2, 1970, p.693.

[11] M. Matijasevic, A. Almazouzi, in :"Structural and Refractory Materials for Fusion and Fission Technologies", MRS Proceedings Vol. 981E (2006) 0981-JJ07-06.