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Effect of Cr on the mechanical properties and microstructure of Fe–Cr model alloys after n-irradiation

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A B S T R A C T

High-chromium ferritic–martensitic steels are candidate structural materials for high-temperature applications in fusion reactors and accelerator driven systems (ADS). Cr concentration has been shown to be a key parameter which needs to be optimized in order to guarantee the best corrosion and swelling resistance, together with the minimum embrittlement. The behavior of Fe–Cr model alloys with different Cr concentrations (0, 2.5, 5, 9 and 12 wt%Cr) has been studied. Tensile tests have been performed in order to characterize the flow properties in the temperature range from −160 °C to 300 °C. The trend of the yield strength with temperature shows that the strain hardening is the same for all materials at low temperatures, even though they have different microstructures. The same materials have been neutron-irradiated at 300 °C in the BR2 reactor of SCK·CEN, up to three different doses (0.06, 0.6 and 1.5 dpa). The results obtained so far indicate that even at these low doses, the Cr content affects the hardening behavior of Fe–Cr binary alloys. Using the Orowan mechanism, the TEM observed microstructure provides an explanation of the obtained hardening but only at the very low dose, 0.06 dpa. At higher doses, other hardening mechanisms are needed.

1. Introduction

High-chromium (9–12 wt%) ferritic/martensitic steels are candidates as potential first-wall and breeding blanket structural materials for future fusion reactors [1] as well as for fuel cladding and the spallation target in the accelerator driven systems [2]. Their use for these applications requires a careful assessment of their mechanical stability under high energy neutron irradiation [3] and their compatibility with the cooling media [4]. Thus, their chemical composition and thermo-mechanical treatments have been optimized over decades of experimental investigations to resist the expected harsh conditions. Indeed, it was found that the steels containing 9 wt% Cr are resistant to swelling [5], less brittle [6] and reasonably immune to erosion corrosion [7] in the temperature range of 300–500 °C. The physical understanding of these empirical findings is the aim of this investigation.

Although Cr-steels became very popular just after World War I, as the consequence of the scarcity of Tungsten [8]. Already in the 1940s, it was established that the behavior of the binary alloy Fe–Cr depends strongly on Cr content in terms of magnetic behavior and electrical resistivity [9]. It is, however, only very recently that the effect of Cr on the mixing enthalpy of the alloys [10] and on their resistance to swelling have been elucidated theoretically [11]. Furthermore, the effect of Cr and temperature on defect accumulation was studied for some time [12–16]. As described by Yoshida et al. [12], dislocation loops nucleate and grow slowly under irradiation, especially at lower temperatures. At higher temperatures loops are larger and their density decreases. In the presence of Cr they nucleate and grow faster and with increasing temperature their density decreases while their size increases. Cr has a strong effect on defect stability. Thus, while in Fe defects start shrinking or disappearing at $T > 750$ K, when adding Cr, the shrinkage and the motion of the observed loops are significantly suppressed [14]. However, the effect of Cr concentration on the microstructural changes and their influence on the mechanical behavior has never been studied thoroughly.

The general objective of this work is to investigate the effect of Cr on the irradiation-induced defect formation and accumulation in Fe–Cr based model alloys with well defined chemical compositions and microstructures both to examine the microstructure changes and the hardening induced by well controlled neutron irradiation and also to help the theoreticians to bench-mark their codes.

In this paper, the properties of the investigated materials will be described in Section 2. In Section 3, the microstructural changes due to neutron irradiation are described in detail. The tensile data obtained are summarized in Section 4 and finally the relationship between the microstructures and the hardening of the investigated four binary alloys, are discussed in terms of Orowan mechanism.

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2. Materials

The investigated materials have been laboratory-fabricated, thermo-mechanically treated and thoroughly analyzed in terms of microstructure and tensile properties.

2.1. Composition and heat treatment

The materials used in this work were Fe–Cr based model alloys with 2.5%Cr (2.36 wt% Cr), 5%Cr (4.62 wt%Cr), 9%Cr (8.39 wt%Cr) and 12%Cr (11.62 wt%Cr) obtained by furnace melting of industrial pure Fe and Cr. After casting, the obtained ingots were cold worked under protective atmosphere to fabricate plates of 9 mm in thickness. Similar to the standard practice of ferritic–martensitic steels, Fe–Cr model alloys were treated at 1050 °C, for 1 h in high vacuum for austenisation and stabilization. Thereafter, the tempering was done at 730 °C for 4 h, followed by air cooling. The final product was chemically analyzed using the adequate techniques to measure both substitutional and interstitial impurities. The total amount of impurities did not exceed 300 wt ppm [17] as reported in Table 1.

2.2. Electrical resistivity

Electrical resistivity measurements were performed between liquid helium temperature (4.2 K) and room temperature (300 K) with and without a saturated longitudinal magnetic field of 0.5 T. The measured resistivities (μΩ cm) obtained as a function of Cr content at the lowest (full symbols) and the highest (open symbols) temperatures are shown in Fig. 1, together with those found in the literature for binary Fe–Cr alloys. It should be mentioned here that the magnetic field had no effect on the measured resistivity in the investigated alloys (therefore, the results obtained with, are omitted here for figure clarity). In the figure, the values of the resistivity obtained by using the empirical model proposed by Maury et al. [18] are also reported. As it can be noticed, the results obtained here are very comparable to those reported in earlier investigations [18–20]. In fact, one can distinguish clearly two domains: below and above 9%Cr. This variation is believed to be one of the reasons affecting the response of this system after irradiation as well. The absolute values found here are of the same order of magnitude as those reported in the literature [18–20] demonstrating that the investigated alloys can be considered as binary systems with negligible amount of interstitial (N, C) impurities.

2.3. Microstructure for non-irradiated material

Specimens of 3 mm diameter that were first cut by an EDM machine with a diameter of 1 mm, fine mechanically polished to about 100 mm, were then prepared using the conventional jet polishing technique and examined using a TEM (JOEL 3010 EX) with an acceleration voltage of 300 KeV. The microstructure of the as-prepared Fe–Cr model alloys is illustrated by the images in Fig. 2. It can be seen that with increase of Cr content, the microstructure

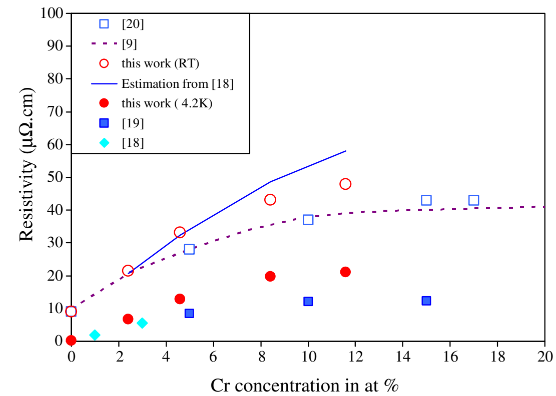

Fig. 1. Electrical resistivity of Fe–Cr binary alloys. The low temperature (full symbols) and the room temperature (open symbols) obtained without magnetic field are compared with those reported in the literature [18–20].

is changing from fully ferritic to ferrite and bainite. Thus, TEM bright field images of Fe–2.5%Cr and Fe–5%Cr alloys show massive amount of ferrite grains with randomly oriented grain boundaries that are the signature of fast cooling which prevents the formation of equiaxed ferrite. The dislocation density for these two alloys is respectively 1.2×10^9 and 5.8×10^9 cm^{-2}. High Cr alloys with 9% and 12%Cr content, have very small grains of the order of 1 μm. Their microstructure consists of bainitic ferrite which is obtained by rapid cooling. Grains are small with many planar boundary segments. The density of dislocations for Fe–9%Cr is 6.3×10^9 cm^{-2} and for Fe–12%Cr a value of 5.5×10^9 cm^{-2} is obtained. Dislocations are predominately of type $a_0/2\langle 1\,1\,1\rangle$ Burgers vectors, where a_0 is the lattice parameter. Thus, the microstructure of high Cr alloys is considered to be very similar to the usual ferritic–martensitic steels [21], as the grains are pretty similar to the martensitic laths, but in these model alloys the laths consist of ferrite grains with a well structured density of dislocations.

2.4. Tensile tests of as-received model alloys

The tensile specimens with nominal dimensions (overall length = 27 mm, gage length = 12 mm and diameter = 2.4 mm) were prepared using an EDM cutting machine followed by a fine mechanical polishing of the surface. Tensile tests were performed according to ASTM E8M-01 and E21-92 (1998) standards with an electro-mechanical test frame (INSTRON 8500, model 1362), and a crosshead displacement rate of 0.2 mm/min corresponding to a strain rate of approximately 2.8×10^{-4} s^{-1} in the temperature range from −160 °C to 300 °C. Engineering stress–strain curves at room temperature (RT) are presented in Fig. 3. Both the yield stress (YS) and the ultimate tensile stress (UTS) for model alloys increase linearly with Cr content. In terms of ductility, low Cr alloys are more ductile than high Cr alloys. It was found that the ductility of the investigated materials is also related to the grain size. Thus, low Cr alloys that have larger grain sizes tend to have a lower

Table 1
Chemical composition of investigated Fe–Cr model alloys measured in wt% Fe balance the alloys of reference 251, 259, 252, 253 are named Fe–2.5Cr, Fe–5Cr, Fe–9Cr, Fe–12Cr, respectively in the text

Alloy	Mn	Si	P	S	Al	Ti	Cr	Ni	O	C	N	V	Total amount of impurity (wt ppm)
251	0.009	0.02	0.013	0.0020	0.003	0.004	2.4	0.044	0.035	0.008	0.0117	0.001	~149
259	0.02	0.04	0.011	0.006	0.0033	0.0028	4.6	0.06	0.065	0.02	0.0127	0.001	242
252	0.03	0.09	0.012	0.00066	0.0069	0.0034	8.4	0.07	0.066	0.02	0.0148	0.002	333
253	0.03	0.11	0.05	0.006	0.003	0.0037	11.6	0.09	0.03	0.027	0.0237	0.002	~375