



The microstructure and tensile properties of Fe–Cr alloys after neutron irradiation at 400°C to 5.5–7.1 dpa

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Abstract

Room temperature tensile properties, void swelling and microstructural changes in Fe–Cr alloys (0, 2, 6, 12 and 18 at.% Cr) have been studied following irradiation at 400°C in the BR-10 fast reactor to doses in the range 5.5–7.1 dpa. Swelling of 3%, 0.03% and 0.1% was found in Fe, Fe–2Cr and Fe–12Cr alloys, respectively. No voids were observed in Fe–6Cr and Fe–18Cr alloys. In Fe, Fe–2Cr and Fe–6Cr dislocation loops lie in 1 0 0 habit planes and have a $\langle 1 0 0 \rangle$ Burgers vector, while in Fe–12Cr and Fe–18Cr alloys a mixed population of loops with a $\langle 1 0 0 \rangle$ and $a/2 \langle 1 1 1 \rangle$ Burgers vectors have been observed. The strength of irradiated alloys increases and ductility decreases with increasing Cr content. Fe–18Cr alloy revealed a severe embrittlement after irradiation. © 1998 Elsevier Science B.V. All rights reserved.

1. Introduction

The Fe–Cr binary alloys are a base for low activated ferritic–martensitic steels used widely as structural materials for fission reactors and being now considered as candidate materials for future fusion reactors. Ferritic–martensitic steels provide an attractive alternative to austenitic stainless steels because they demonstrate a very high resistance to swelling up to damage doses of 200 dpa [1]. Unfortunately, the most of these steels exhibit a low temperature irradiation-induced embrittlement that imposes a severe restriction on their application at temperatures below 400°C. A study of irradiation behavior of Fe–Cr alloys is being undertaken in an attempt to elucidate the basic mechanisms of void swelling and low temperature irradiation-induced embrittlement in ferritic–martensitic steels.

The present work has been conducted with the objective of determining the effect of chromium additions on the microstructure, void swelling and tensile properties in Fe–Cr alloys after neutron irradiation at 400°C to relatively low dose of 5.5–7.1 dpa.

2. Experimental details

After prior electron beam melting the starting materials, four Fe–Cr alloys have been prepared by melting in the arc furnace. The chemical composition of iron used was (wt%): C – <0.015, P – <0.005, S – <0.05, Si – 0.012, Al – 0.001, Cr – 0.0015, Cu – 0.0015, Ni – (0.007–0.03), Mg – <0.0015, Mo – 0.001, O – 0.18, N – (0.002–0.004). The purity of electrolytic chromium was no less than 99.9%. The furnace was refined by first melting a titanium iodide getter button. The alloys have been melted on a water-cooling copper mold in helium atmosphere (133–200 Pa) in order to eliminate the chromium evaporation. Ingots of ≈ 0.05 kg in weight were remelted six times. The composition of alloys was checked by weighing the ingots after final melting. The difference in weights of the starting materials and final ingots did not exceed 0.1%. In addition, the composition of alloys was checked-up by the chemical and spectral analyses. The final chromium content in specimens investigated was equal to 0, 2, 6, 12 and 18 at.%.

After melting, the ingots were hot rolled into approximately 0.6 mm thick strips and then heat treated as follows: 800°C/0.5 h/AC + 1000°C/0.1 h/WQ + 630°C/15 h/WC. Following this treatment all the specimens were cold rolled into 0.5 mm thick strips with cold-work level

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varying from 5% to 15%. Miniature tensile specimens with gauge dimensions of 7.5 mm in length and 2 mm in thickness were punched from the strips. Then all the specimens were inserted into sodium ventilated stainless steel capsules and irradiated in the BR-10 reactor at the temperature of 400°C at dose rate of 1×10^{-7} dpa/s to damage doses in the range from 5.5 to 7.1 dpa (NRT). The tensile tests were conducted at room temperature at the strain rate of $2.2 \times 10^{-3} \text{ s}^{-1}$ using a shielded tensile testing machine. Three tests have been conducted for each alloy. From the test data ultimate and yield strengths (US and YS), uniform and total elongations (UE and TE) have been determined. The scatter of measured YS values did not exceed ± 30 MPa. Disks of 3 mm in diameter have been punched from heads of specimens tested mechanically and after a preliminary mechanical thinning up to 0.2 mm, specimens suitable for transmission electron microscopy (TEM) have been prepared. The TEM specimens of pure Fe and Fe–2Cr, Fe–6Cr alloys were polished using the standard A-8 ('Struers') electrolyte at voltage of 110–115 V, current of 0.3–0.35 A and temperature of +18°C. Oxide films formed on the surfaces of Fe and Fe–2Cr alloy specimens were removed by flash electropolishing at 25–30 V in the 4 wt% solution of chromium anhydride (CrO_3) in the

phosphoric acid (H_3PO_4). The Fe–12Cr and Fe–18Cr alloy specimens were electropolished in the standard A-2 ('Struers') electrolyte at 35–40 V and temperature of +12°C. The thin-foil specimens were examined using a JEM-100CX electron microscope equipped with a side entry goniometer stage. For a quantitative TEM analysis, those places of a foil were chosen where extinction fringes were parallel to the foil edge or not observed at all. The foil thickness (varying from 100 to 150 nm) was determined by measuring the widths of grain or subgrain boundary projections. The accuracy of thickness determinations is believed to be $\pm 20\%$.

3. Results

3.1. TEM microstructure

3.1.1. Unirradiated specimens

Typical microstructures of unirradiated Fe and Fe–Cr alloys are shown in Fig. 1(a)–(d). Final cold working to levels of 5–15% has resulted in the formation of a cellular dislocation structure in ferritic grains of all materials. The dislocation density in the materials varied from 2.2×10^{14} to $5.0 \times 10^{14} \text{ m}^{-2}$ depending on cold worked

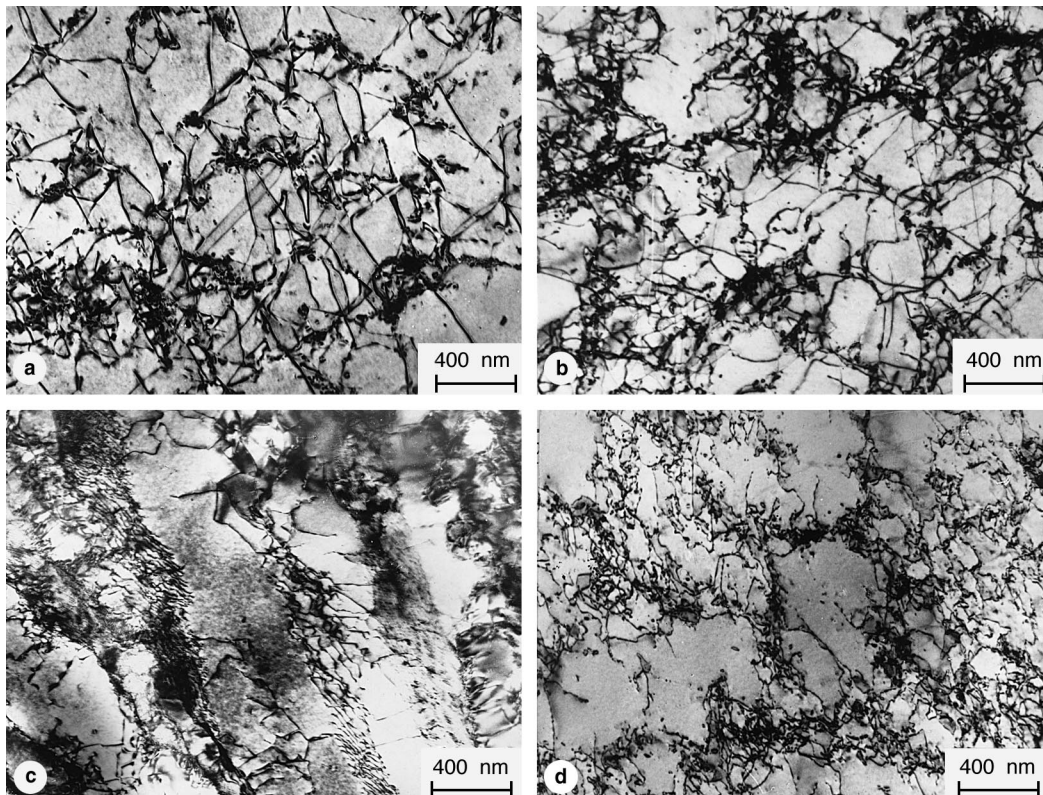


Fig. 1. Dislocation structures in unirradiated Fe (a) and Fe–2Cr (b), Fe–6Cr (c), Fe–18Cr (d) alloys.

level. However, the distribution of dislocations was different in the materials. So, more or less uniform dislocation network with weakly pronounced cells has been observed in pure iron (Fig. 1(a)). An irregular cell structure with varying cell sizes and shapes was observed in the Fe–2Cr and Fe–12Cr alloys. The cellular dislocation structure was strongly pronounced in the Fe–18Cr alloy (Fig. 1(d)). The Fe–6Cr alloy has a tempered lath martensitic structure. In this alloy dislocation walls form boundaries of thin elongated subgrains with a nonuniform dislocation network inside (Fig. 1(c)). When measuring the dislocation density in the Fe–6Cr alloy, the contribution of subgrain boundaries has been taken into account. The Fe–12Cr alloy has a duplex structure with 9:1 ferrite-to-martensite ratio. In the Fe–18Cr alloy needle like intragranular precipitates up to 2 μm in length were detected. The precipitates were randomly distributed in the matrix, their concentration was less than $1 \times 10^{18} \text{ m}^{-3}$. These precipitates have orthorhombic structure with lattice parameters $a = 0.46 \text{ nm}$, $b = 0.51 \text{ nm}$, $c = 0.67 \text{ nm}$ and most likely are cementite (M_3C) precipitates. No precipitation was found in the starting microstructure of other materials investigated.

The grain size in all materials investigated was in the range from 20 to 50 μm .

3.1.2. Irradiated specimens

In all specimens irradiated the void formation and dislocation structure changes have been observed. In addition, in Fe–12Cr and Fe–18Cr alloys radiation-induced α' -precipitates have been detected. The results of TEM measurements are given in Table 1. Typical microstructures of irradiated specimens are shown in Figs. 2 and 3.

voids. Void swelling in Fe–Cr alloys versus the chromium content is shown in Fig. 4 together with the experimental data of Little and Stow [2] and Gelles [1,3,4] obtained for Fe–Cr alloys with the pre-irradiation heat treatment different from used in the present work. As it is seen from Fig. 4, the swelling in Fe–Cr alloys inves-

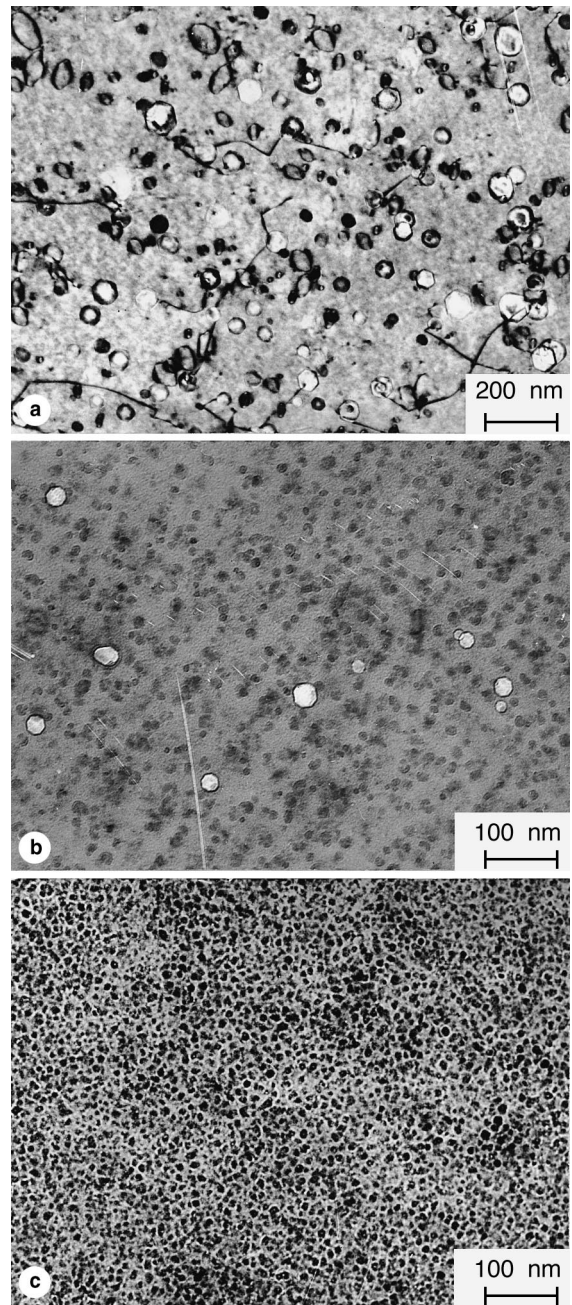


Fig. 2. Microstructures in Fe (a) and Fe–12Cr (b), Fe–18Cr (c) alloys after neutron irradiation at 400°C to 5.5–6.5 dpa. The imaging conditions: (a) (1 1 1)-orientation, $g = [1 \bar{1} 0]$, (b), (c) – kinematic condition, void contrast.

Table 1

Microstructural data for Fe–Cr alloys irradiated in the BR-10 reactor at the temperature of 400°C and dose rate of 1×10^{-7} dpa/s

Cr content, at.%	0	2	6	12	18
dpa	6.2	6.2	7.1	6.5	5.5
$\langle d_v \rangle$, nm	44	7	–	23	–
N_v , 10^{20} m^{-3}	4	4.1	–	1.1	–
$\Delta V/V$, %	3	0.03	–	0.1	–
$\langle d_l \rangle$, nm	28	7.2	8.5	8.4	7.0
N_l , 10^{22} m^{-3}	0.15	7.2	5.0	2.0	3.0
$\rho_d^{\text{unirr.}}$, 10^{14} m^{-2}	2.2	3.6	5.0	2.2	4.0
$\rho_d^{\text{irr.}}$, 10^{14} m^{-2}	1.2	2.5	3.5	0.6	2.0
$\langle d_p \rangle$, nm	–	–	–	9	6
N_p , 10^{22} m^{-3}	–	–	–	2.8	5.0

tigated in the present work is a complicated function of chromium content, reaching 3% in pure iron. At 400°C and 6.2–7.1 dpa the swelling in Fe–2Cr and Fe–6Cr alloys is strongly suppressed as compared with Fe–12Cr alloy. From other data shown in Fig. 4 it is seen that

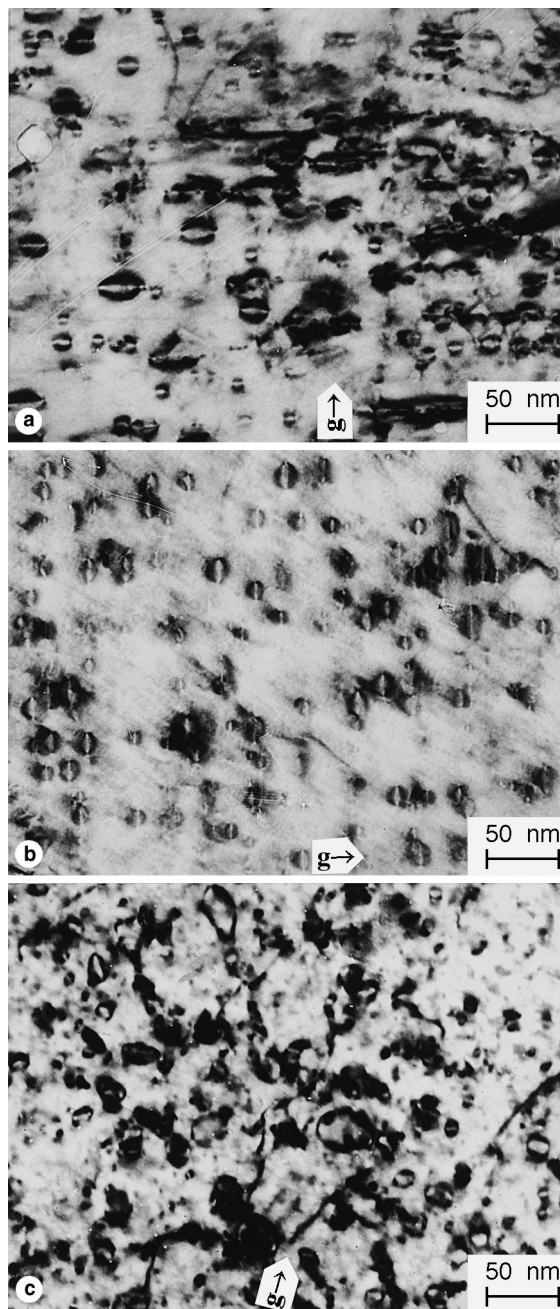


Fig. 3. Dislocation structure of Fe-2Cr (a), Fe-6Cr (b) and Fe-18Cr (c) alloys after neutron irradiation at 400°C to 5.5–7.1 dpa. The imaging conditions: (a) (0 0 1)-orientation, $g = [2\ 0\ 0]$, (b) (0 0 1)-orientation, $g = [2\ 0\ 0]$, (c) (1 1 2)-orientation, $g = [1\ \bar{1}\ 0]$.

low swelling in neutron irradiated Fe–Cr alloys with chromium content of 2–3% seems to be a general feature, independent of differences in starting microstructures.

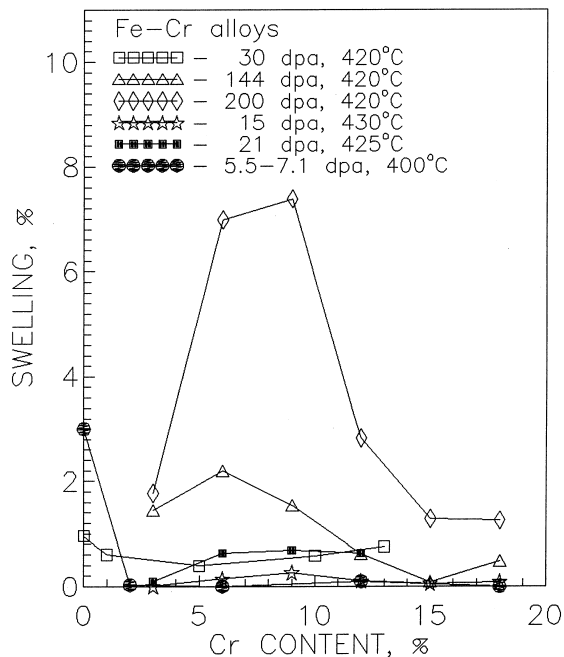


Fig. 4. Swelling in Fe–Cr alloys as a function of chromium content.

Dislocation structure. All the irradiated specimens contain dislocation loops of high number density and a rather dense dislocation network. The mean loop diameter is larger and the loop number density is lower in pure iron as compared with alloys. The Cr additions result in a decrease of loop size and in an increase of loop number density (Table 1). The dislocation structure in alloys with Cr content less than 6% consists of loops lying in {1 0 0} habit planes and having predominately a $\langle 1\ 0\ 0 \rangle$ type Burgers vector. In other alloys a mixed population of dislocation loops with a $\langle 1\ 0\ 0 \rangle$ and $a/2\ \langle 1\ 1\ 1 \rangle$ Burgers vectors is observed. It should be noted that subgrain boundary configurations in the Fe-6Cr alloy seem to be not affected by neutron irradiation. One can see from Table 1 that the mean loop diameter is practically independent of chromium content in the range from 2% to 18% Cr, that is in agreement with TEM measurements for a $\langle 1\ 0\ 0 \rangle$ dislocation loops in Fe–Cr alloys irradiated in the FFTF [3].

Precipitates. In Fe-12Cr and Fe-18Cr alloys α' -precipitates formed under irradiation (Fig. 2(b) and (c)). Particles of α' -phase were clearly visible in thin parts of foils (50–70 nm) at a wide incident electron beam under slightly underfocused conditions.

3.2. Tensile mechanical properties

Averaged tensile test data for unirradiated and irradiated specimens are given in Table 2. Figs. 5–7 show

Table 2

Tensile mechanical properties of Fe–Cr alloys irradiated in the BR-10 reactor at 400°C and dose rate of 1×10^{-7} dpa/s

Cr content, at.%	0	2	6	12	18
dpa	6.2	6.2	7.1	6.5	5.5
YS ^{unirr.} , MPa	200 ± 10	250 ± 20	445 ± 10	370 ± 10	440 ± 30
YS ^{irr.} , MPa	265 ± 5	495 ± 5	710 ± 20	740 ± 30	–
US ^{unirr.} , MPa	250 ± 20	290 ± 10	500 ± 10	400 ± 10	455 ± 20
US ^{irr.} , MPa	275 ± 5	505 ± 5	715 ± 20	760 ± 30	760 ± 50
TE ^{unirr.} , %	20 ± 2	13.5 ± 0.5	7.5 ± 0.5	14 ± 1	6 ± 1
TE ^{irr.} , %	13.5 ± 0.5	10.0 ± 0.5	2.5 ± 0.5	4.5 ± 1	0
UE ^{unirr.} , %	12 ± 1	6.0 ± 0.5	2.0 ± 0.5	5.5 ± 0.5	1.0 ± 0.5
UE ^{irr.} , %	1.5 ± 0.5	0.5 ± 0.1	0.5 ± 0.1	0.5 ± 0.1	0

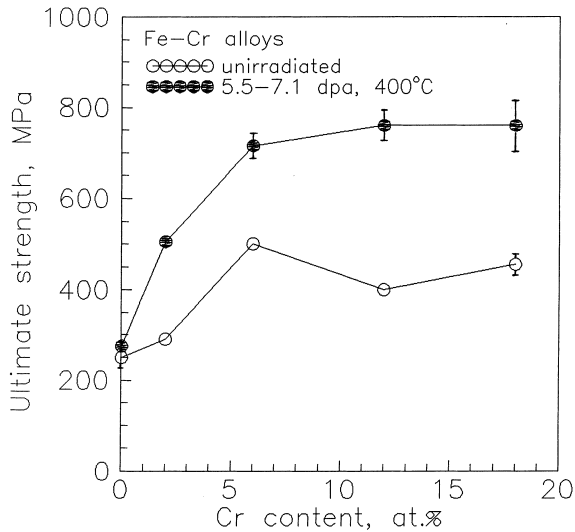


Fig. 5. Ultimate strength in Fe–Cr alloys versus chromium content.

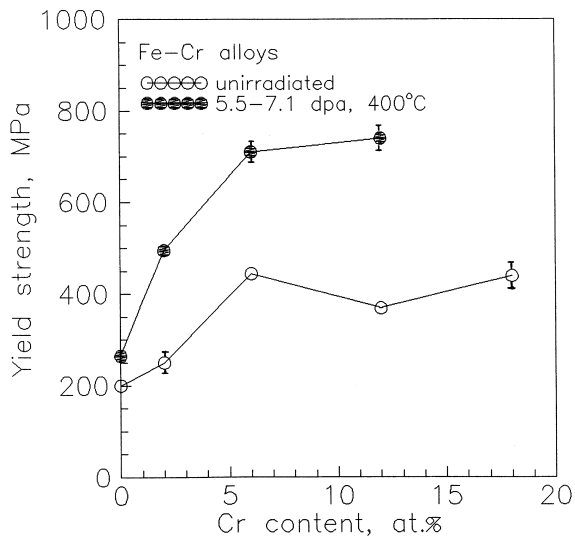


Fig. 6. Yield strength in Fe–Cr alloys as a function of chromium content.

measured US, YS and TE versus chromium content. Neutron irradiation at 400°C to doses from 5.5 to 7.1 dpa results in strengthening of Fe–Cr alloys accompanied by a simultaneous reduction of their ductility. An extent of irradiation strengthening and ductility loss increases with increasing the chromium content. The Fe–18Cr alloy exhibits a complete embrittlement after irradiation.

4. Discussion

One of the striking results of the present study is a very high swelling in pure iron at 6.2 dpa and 400°C. At dose rate of 1×10^{-7} dpa/s the mean swelling rate of pure iron is approximately 0.5%/dpa that is much larger than the swelling rate of 0.03%/dpa found by Little et al. [1] in pure Fe irradiated in DFR at 420°C to damage dose of 30 dpa (N/2) at a dose rate, which is probably higher approximately one order of value.

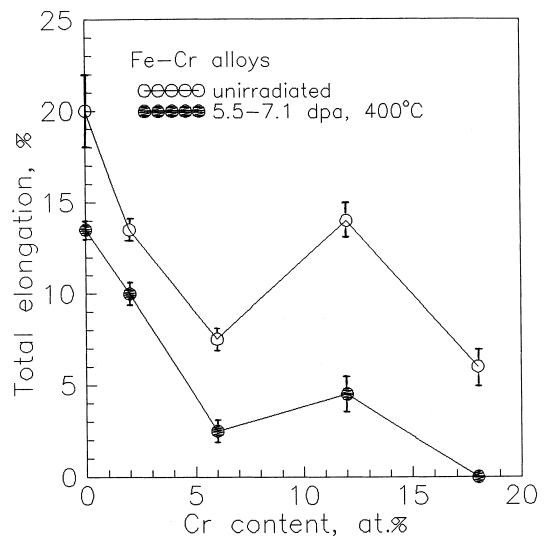


Fig. 7. Total elongation in Fe–Cr alloys versus chromium content.

To authors knowledge, the swelling level as large as 3% was never observed in pure Fe after neutron, ion or electron irradiation at 400°C and 6.2 dpa. For a plausible explanation, one can assume that the cold-work level in our experiment turned out to be most optimal for swelling promotion in pure Fe. The existence of some optimal dislocation density has been demonstrated in the HVEM experiment on 1 MeV electron irradiation of pure Fe at 350°C to dose of 17 dpa [5]. But, for the present it is not known whether such an optimal dislocation density exists in the case of neutron irradiation.

A second striking result is the number of point defects containing in dislocation loops in the Fe–2Cr, Fe–6Cr and Fe–18Cr alloys. Estimating this number as $(\pi/4)\langle d_l \rangle^2 b N_l$ where $\langle d_l \rangle$ is the mean loop diameter, N_l the loop number density, b the Burgers vector, one finds that in Fe–2Cr, Fe–6Cr and Fe–18Cr alloys it exceeds significantly the total number of vacancies stored in voids (i.e. swelling). It should be noted that according TEM data of Ref. [3], only in Fe–3Cr alloy irradiated in the FFTF at 430°C to 15 dpa the total number of point defects in $a(100)$ loops also exceeds the swelling value. If observed loops are interstitial in nature, then on the basis of a common concept of network dislocations being a preferential sink for interstitials one should expect that except voids vacancies are stored in some small unresolvable clusters, most likely in small gas bubbles. It is possible also that the initial cold work structure could play a role. But, in the last case it is necessary to propose that in contrast to pure Fe and Fe–12Cr alloy some components of the starting dislocation structure in Fe–2Cr, Fe–6Cr and Fe–18Cr alloys are biased for vacancies more strongly than both the rest of the components and interstitial loops are biased for interstitials. If the loops are vacancy in nature, one has to assume that they are appropriately biased and can grow despite of network dislocations are being preferential sinks for interstitials.

Under neutron irradiation conditions the equation for the void volume fraction $\Delta V/V$ dose rate can be written in the following form:

$$\frac{d}{dKt} \frac{\Delta V}{V} = B \frac{S_v S_d}{(S_v + S_d)(S_v + S_d + S_n)}, \quad (1)$$

where K is the dose rate, B is the bias factor, S_v and S_d are the sink strengths for voids and dislocations, respectively, S_n is the strength of ‘neutral’ sinks defined here as sinks for which the difference of two rates, the rate of vacancy capture and the rate of vacancy evaporation, is equal to the rate of capture of freely migrating interstitials. As such neutral sinks one can consider precipitates, quasi-stable gas bubbles as well as vacancy dislocation loops grown to some limiting size beginning from which a vacancy loop becomes to be a preferential sink for interstitials. Solute atoms (either interstitial or substitutional) which trap radiation-induced monovacancies to

form saturable point defect-solute complexes can be discounted as neutral sinks because at 400°C the binding energies of such complexes in Fe–Cr alloys must be as large as ≈ 1 eV or even more, that seems unlikely.

It is seen from Eq. (1), that the dependence of voidage rate on alloy composition is determined by both the bias factor B and the combination of sink strengths. From data shown in Table 1, the swelling rate can be crudely estimated as ratio of a final swelling value and the damage dose accumulated. Ignoring the sink efficiencies, assuming the precipitates as neutral sinks and using the calculated sink strengths given in Table 3 (for voids $S_v = 2\pi\langle d_v \rangle N_v$, for loops $S_l = \pi\langle d_l \rangle N_l$, for precipitates $S_p = 2\pi\langle d_p \rangle N_p$ and for network dislocations $S_d = \rho_d$, where $\langle d \rangle$ is the mean diameter, N the number density and ρ_d the dislocation density), and considering dislocation loops as interstitial ones, one finds that the bias factor B is equal to 2.3%, 0.5% and 2.2% in Fe, Fe–2Cr and Fe–12Cr, respectively. In a systematic study of the swelling dependence on nickel content in experimental fcc Fe–17Cr–Ni alloys under 1 MeV-electron irradiation, Walters [6] has found that the bias factor magnitude decreases from 2.8–3.0% to 0.5% with increasing nickel content from 15 to 60 wt%. Surprisingly, the estimated magnitudes of bias factor in Fe–Cr alloys fall in the same range. By authors opinion, the bias factor decrease from 2.3% to 0.5% with increasing chromium content from 0 to 2% seems to be hardly probable for Fe–Cr alloys. However, when assuming the bias factor B being independent of Cr content and its value is close to 2.2% in the Fe–Cr alloys, one has to propose an existence of some neutral sinks with the extremely high sink strength of $6.5 \times 10^{15} \text{ m}^{-2}$ in Fe–2Cr alloy. For gas bubbles as such neutral sinks that means that their mean diameter should be less than 0.7 nm at the bubble number density of order of $1 \times 10^{24} \text{ m}^{-3}$. When considering the vacancy dislocation loops as such a neutral sink for point defects, one finds that the bias factor is 3.0%, 0.5% and 2.7% in Fe, Fe–2Cr and Fe–12Cr alloys, respectively. It should be noted that the calculated value of B factor in Fe–2Cr alloy, in fact, is insensitive to dislocation loop character because the void sink strength is very low in this alloy. Therefore, in the case of compositional independence of the bias factor the assumption on

Table 3
Sink strengths in Fe–Cr alloys irradiated in the BR-10 reactor at 400°C and dose rate of 1×10^{-7} dpa/s

Cr content, at.%	0	2	6	12	18
dpa	6.2	6.2	7.1	6.5	5.5
$S_v, 10^{14} \text{ m}^{-2}$	1.10	0.18	–	0.16	–
$S_l, 10^{14} \text{ m}^{-2}$	1.3	16.3	13.6	5.3	6.6
$S_d^{\text{unirr.}}, 10^{14} \text{ m}^{-2}$	2.2	3.6	5.0	2.2	4.0
$S_d^{\text{irr.}}, 10^{14} \text{ m}^{-2}$	1.2	2.5	3.5	0.6	2.0
$S_p, 10^{14} \text{ m}^{-2}$	–	–	–	15.8	18.8

the presence of unresolvable gas bubbles in matrix alloys, at least partially, to answer the question why a minimum in swelling versus chromium concentration curves is observed at Cr concentrations near 3 at.% (see Fig. 4).

For an analysis of data on the yield strength σ_y in terms of microstructural data the following equation for unirradiated and irradiated materials was used:

$$\sigma_y = \sigma_0 + \alpha_d Gb\sqrt{\rho_d} + \alpha_l Gb\sqrt{\langle d_l \rangle N_l} + \alpha_v Gb\sqrt{\langle d_v \rangle N_v} + \alpha_p Gb\sqrt{\langle d_p \rangle N_p}, \quad (2)$$

where G is the shear modulus, b is the Burgers vector, α_d , α_l , α_v and α_p are the barrier strength constants for network dislocations, dislocation loops, void and precipitates, respectively. It was assumed that values of σ_0 are not affected by irradiation.

To reduce the number of parameters involved in Eq. (2), the experimental 25°C data of Ref. [7] on the dependence of flow stress on average dislocation density in pure iron have been taken into account. According to Ref. [7] the value of σ_0 is close to zero. In the present work $\sigma_0 = 0$ was chosen for the unirradiated Fe–2Cr alloy that resulted in $\alpha_d = 0.64$. It should be noted that this value of α_d differs markedly from 0.9 evaluated in Ref. [7].

Using the work hardening barrier constant $\alpha_d = 0.64$ for all the materials investigated, the values of σ_0 have been calculated for each chromium concentration. For calculations it was taken $G = 83$ GPa, $b = 0.2482$ nm in Fe–Cr alloys. Then considering the values of α_l , α_v and α_p as best fitting parameters, from the experimental data on yield strength in neutron irradiated materials shown in Table 2 it was found that $\alpha_d = 0.64$, $\alpha_l = 0.68$, $\alpha_v = 0.17$ and $\alpha_p = 0.85$. In calculations the measured values of yield and ultimate strengths were taken to be equal for irradiated Fe–18Cr alloy. The calculated values of σ_y deviate from experimental ones no more than 26 MPa which is within error bars. It was proposed that dispersed quasi-equilibrium gas bubbles do not affect the yield strength because one can expect that such bubbles impose low stresses on the matrix due to the compensation of internal gas pressure by the bubble surface tension and, therefore, cannot act as barriers resisting the dislocation glide.

5. Conclusions

1. The swelling in Fe–Cr alloys neutron irradiated at dose rate of 1×10^{-7} dpa/s and temperature of

400°C to 5.5–7.1 dpa is strongly dependent on Cr concentration.

2. The highest swelling of 3% was observed in pure iron at the irradiation conditions. A strong suppression of void formation occurred in Cr content ranges from 3% to 6% Cr and for >12% Cr.
3. Dislocation loops with predominantly $a \langle 100 \rangle$ type Burgers vector have been observed in irradiated Fe and Fe–2Cr, Fe–6Cr alloys. In Fe–Cr alloys containing more than 6% Cr a mixed $a \langle 100 \rangle$ and $a/2 \langle 111 \rangle$ loop populations formed under neutron irradiation at 400°C.
4. Both the ultimate and yield strengths in neutron irradiated Fe–Cr alloys increase monotonically with increasing chromium content. Irradiated Fe–18Cr alloy has zero values of total and uniform elongations.
5. In Fe–2Cr alloy the total number of point defects in visible dislocation loops exceeds by more than three times the total number of vacancies in voids. In the case of interstitial loops one can conclude that during neutron irradiation a significant portion of vacancies has condensed in the form of unresolvable clusters, presumably, in small quasi-equilibrium gas bubbles. Probably, a low swelling in Fe–Cr alloys with chromium concentrations near 3 at.% is due to the presence of unresolvable clusters of a high sink strength for point defects.

References

- [1] D.S. Gelles, J. Nucl. Mater. 225 (1995) 163–174.
- [2] E.A. Little, D.A. Stow, in: Proc. Int. Conf. on Irradiation Behavior of Metallic Materials for Fast Reactor Core Components, Ajaccio, France, 4–8 June 1979, pp. 17–24.
- [3] D.S. Gelles, J. Nucl. Mater. 108&109 (1982) 515.
- [4] D.S. Gelles, in: N.H. Packan, R.E. Stoller, A.S. Kumar (Eds.), Effects of Radiation on Materials: Fourteenth International Symposium, vol. I, ASTM STP 1046, American Society for Testing and Materials, Philadelphia, PA, 1989, p. 73.
- [5] F. Kuramoto, K. Futagami, K. Kitajima, in: Proc. Sixth Int. Conf. on High Voltage Electron Microscopy, Kyoto, 1977, p. 589.
- [6] G.P. Walters, J. Nucl. Mater. 136 (1985) 263.
- [7] D.J. Bailey, W.F. Flanagan, Philos. Mag. 15 (133) (1967) 43.