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SOME EVIDENCE ON THE NON-EQUILIBRIUM CARBON CONCENTRATION PRODUCED BY FAST-NEUTRON IRRADIATION OF IRON-CARBON ALLOYS

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Introduction

Recently, the radiation anneal hardening further referred to as RAH in α -iron containing different quantities of carbon was observed at 0.21 T_m (1); the results indicated that the RAH peak is closely related to the presence of interstitial impurity. In the present work, an analysis of the possible carbon behaviour during irradiation and after post-irradiation annealing was made. The idea to be followed is that at least a part of precipitated carbon is transferred during irradiation into solid solution, i.e. some kind of supersaturation occurs. This carbon on annealing migrates towards radiation-induced defects, giving a rise to hardening observed at 0.21 T_m.

Experimental

Chemical composition of the iron (wt.%) was: C-0.0270; Mn--0.0050; N-0.0005; O-0.0035. Specimens 10x6x1 mm for both, microhardness and transmission electron microscopy were cut from as received plate. Annealing treatment: 24 hr at 700°C and 27 hr at 300°C in vacuum (10^{-7} mm Hg), slowly cooled to room temperature. After this treatment all carbon was assumed to be precipitated in the form of Fe_3C. The grain structure was rather homogeneous with an average grain diameter of 0.021 mm.

The isochronal anneals of irradiated specimens were carried out in the hot-cell under the inert gas atmosphere.

The irradiations were carried out in CEN Saclay-OSIRIS reactor. The test assembly was made of two welded Al plates, one of which was 1 mm grooved to contain the specimens. The assembly was filled with He and placed into experimental channel cooled by the water. The irradiation temperature was measured by 3 thermocouples welded to the specimens and did not exceed 33°C.

The integrated flux as determined by Cu and Ni detectors was 3.5×10^{19} $n.cm^{-2}$ ($E > 1$ MeV).

The remotely controlled TUCKON machine was used for microhardness tests, at the charge of 100 gr. Each microhardness value referred to correspond to an average of 40 measurements.

The mechanical polishing was used to reduce the plate thickness down to 0.1 mm for electron microscopy. Final chemical thinning in the 2.5-3% solution of HF in H_2O_2 gave satisfactory thin foils.

Results

The microhardness as a function of annealing temperature is presented in FIG. 1, curve (a), together with the tensile tests data for 30 and 70 ppm C iron, curve (b) and (c), taken from the earlier work (1).

The data in FIG. 1 show:

- an increase in the microhardness as a result of irradiation;
- the microhardness peak after post-irradiation annealing at about 125°C;
- the yield stress peak at about 125°C; the amplitude of the peak depends on carbon content.

After 4 months storage at R.T., the yield stress of 70 ppm C iron obtained in an isothermal test at 105°C was approximately constant and close to the maximum yield stress in the isochronal test (FIG. 2). These data suggest that the process responsible for RAH could take place even at room temperatu-

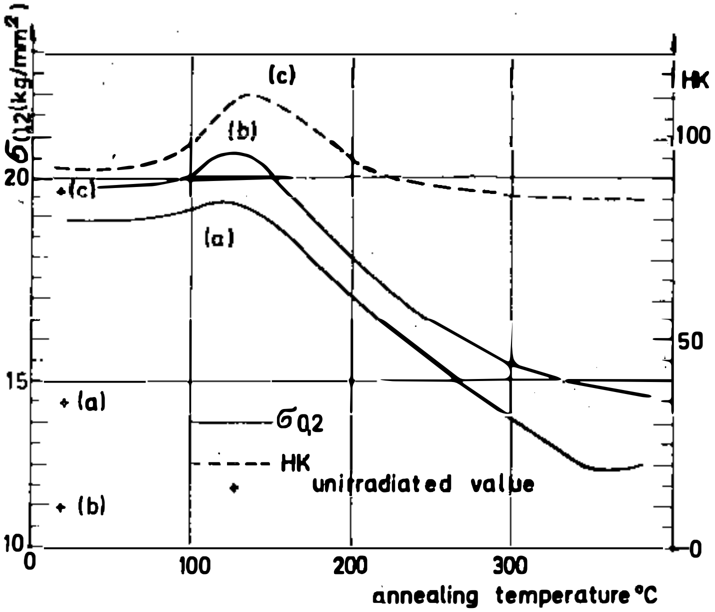

FIG. 1.

Microhardness and yield stress of irradiated iron as a function of annealing temperature
(a) 30 ppm C, 9.4×10^{18} n/cm^2;
(b) 70 ppm C, 5.5×10^{18} n/cm^2;
(c) 275 ppm C, 3.5×10^{19} n/cm^2.

re, providing the time interval between the end of irradiation and the beginning of tensile tests is long enough.

No "black spots" could be detected by TEM in the "as irradiated" specimens. Those annealed at 150°C show essentially the same structure. However, some of them, being deformed during manipulation, have shown the "bowed-out" dislocations between invisible defects. The distance between defects was estimated at about 800 Å.

Discussion

The above experiments, in addition to some literature data (2) suggest a possible "strengthening" of radiation-induced defects by carbon atoms migrating towards them during post-

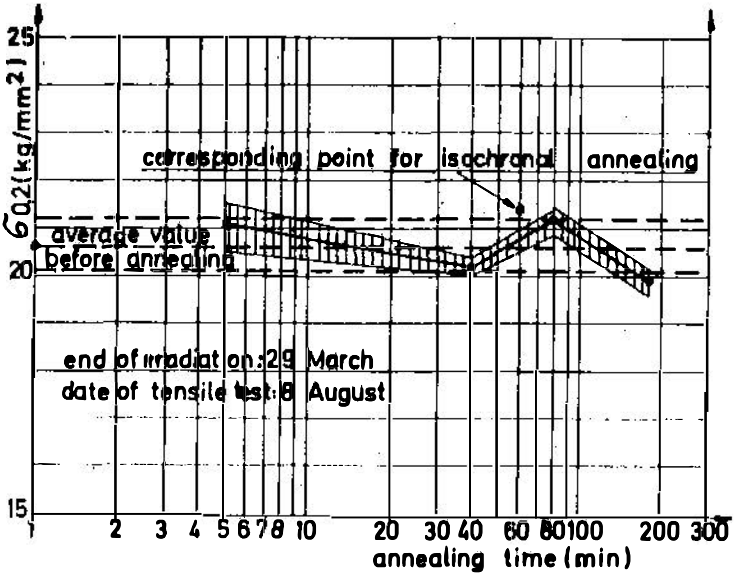

FIG. 2.

The yield stress of irradiated iron as a function of annealing time at 105°C

-irradiation annealing. However, the question arises whether the equilibrium carbon concentration, which is supposed to be 1 ppm (3) at T_{irr} is sufficient to explain this strengthening. Therefore a rough estimation of both these concentrations and their comparison will be made.

Concentration of radiation-induced defects

With the probability of 0.5 for the formation of "zones" effective in hardening (4) and for our experimental conditions we obtain the number of hardening defects as:

$$N_{zef} = \frac{1}{2}\, Ø\, n_{Fe}\, \bar{\sigma}_{el} = \frac{3.5x10^{19} x 0.846x10^{23} x 2.10^{-24}}{2} = 3x10^{18} (cm^{-3}) \quad (1)$$

where: Ø - neutron dose; n_{Fe} - density of target atoms;
$\bar{\sigma}_{el}$ - average elastic scattering cross section of iron.
These defects were assumed to be vacancy loops, ~ 20 Å in diameter (1).

Concentration of carbon in a solid solution

If the equilibrium quantity of carbon in a solid solution at T_{irr}(35°C) corresponds to max 0.0001 wt.% (0.00046 at%), then its concentration will be 3.9×10^{17} cm^{-3}.

Comparison of this figure with the concentration of hardening defects gives a difference of one order of magnitude and makes hard to explain the RAH peak by the strengthening of defects by C atoms.

The idea of C going from meta-stable carbides into solution was suggested by Mc Rickard and Chow (3), and supported by the recent experiments of Seidel (5) with policrystalline Fe. Actually, no experimental data exist allowing to estimate the quantity of C in a supersaturated solution during irradiation. If all C in the present experiment was in solid solution, this would give a carbon concentration of 10^{20}(cm^{-3}), while 10% in solution would give 10^{19} atoms C per cm^3. Even in the latter case carbon concentration would be one order of magnitude higher than defect concentration.

It seems possible from these simple considerations to attribute radiation anneal hardening of α-iron to the strengthening of defects by carbon atoms from a supersaturated solution created during irradiation.

The position of RAH peak (0.21 T_m) in our experiments corresponds to a region of carbon migration (6), and agrees well with Tucker's results (2) for RAH in irradiated Nb.

Distance between defects

The distance between defects was estimated by using the relationship (7):

$$\frac{\mu b}{4l'} = \frac{\mu b d N_{zeff}^{\frac{2}{3}}}{8} \; ; \; l' = \frac{2}{d N_{zeff}^{\frac{2}{3}}} = \frac{2}{20 \times 10^{-8} (3 \times 10^{18})^{\frac{2}{3}}} = 480 \text{Å} \qquad (2)$$

where: μ-shear modulus; b-Burger's vector; l'-distance between defects; d-loop diameter; N_{zeff}-number of effective "zones" (loops).

Distance between visible obstacles as measured from the TEM micrographs gives approx. 800 Å, i.e. close to the calculated value. The fact that the calculated distance was obtained with "as irradiated" specimens and that the experimental value comes from the specimen annealed near RAH temperature supports the idea that the distance between defects does not change considerably during annealing. In other words there is no formation of new defects which could explain RAH, but the defects induced by irradiation are strengthened as obstacles for mobile dislocations.

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References

(1) N. Milašin, J.Nucl.Mat. 34 (1970) 34

(2) R.P. Tucker, Thesis, ORNL-TM-2480

(3) S.B. Mc Rickard and J.G.Y. Chow, Acta Met. 14 (1966)1195

(4) Refered in: F.A. Nichols, Phil. Mag. 14 (1966) 335

(5) G.P. Seidel, Radiation effects 1 (1969) 1

(6) L.J. Cuddy, Phil. Mag. 12 (1965) 855

(7) S.M. Ohr, R.P. Tucker and M.S. Wechsler, ORNL-4195