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Hydrogen–irradiated steel interaction during alternating hydrogenation and annealing

E.A. Krasikov [*], A.D. Amajev

Russian Research Centre, Kurchatov Institute, Kurchatov sq. 1, Moscow 123182, Russian Federation

Abstract

At present when notable progress in plasma physics has been obtained, practical energy utilization from fusion reactors is determined by the state of material science problems. The last includes not only the routine problems of nuclear engineering but also a number of entirely new problems connected with extreme conditions of materials operation – irradiation environment, hydrogenation, thermocycling, etc. Limited data suggest that the combined effect of these factors is more severe than any one of them alone. To study the possible significance of in-service synergistic phenomena, we studied hydrogen–irradiated steel interaction during alternating hydrogenation and heat treatment (annealing). These studies suggest the existence of residual (unrecovered by annealing) degradation effects caused by hydrogen–irradiated steel interaction during alternating hydrogenation and annealing. We propose that this phenomenon must be taken into account when in-service properties of the candidate structural materials are analyzed. © 2000 Elsevier Science B.V. All rights reserved.

1. Introduction

Controlled thermonuclear reactor structural materials will be subjected to neutron irradiation, hydrogenation, thermocycling, etc. For the in-vessel components, hydrogen (protium, deuterium, tritium)–radiation defect interaction during thermonuclear reactor operation has been recognized to be one of the most complex future problems of their safety [1,2].

In the case of intensive interaction, the problem of hydrogen embrittlement of irradiated materials arises because of the exposure to the plasma. However, available information [3] indicates that the life of the first wall could be expanded if periodic post-irradiation in-place annealing was utilized. In this respect, more work must be done in the irradiated/annealed metal–hydrogen interaction.

2. Material and experimental procedures

The commercially fabricated Grade 15Cr–2Mo–V steel was used for this study. The chemical composition of this steel is shown in Table 1.

The heat treatment of the 15Cr–2Mo–V samples consisted of austenization at 980–1000°C, oil quenching, tempering at 680°C, and cooling in the furnace to 350°C. After fabrication, the specimens were annealed in a vacuum furnace at 680°C.

Three-point bend prismatic specimens $3.3 \times 3.3 \times 27$ mm^3 with a sharp notch ($\rho = 0.1$ mm) were placed into capsules for irradiation.

Electrolytical hydrogenation of the specimens was performed in 4% sulphuric acid. The current density was 1000 A m^{-2}, electrolyte temperature 15°C, hydrogenation times 4–24 h.

Capsules were irradiated in the Russian Research Center 'Kurchatov Institute' materials testing MR reactor at a range of temperatures from 50°C (specimens in contact with the water of the reactor pool) to 340°C (irradiation out of contact with water). The maximum fast ($E > 0.5$ MeV) neutron fluence of 5×10^{20} cm^{-2} was attained.

The amounts of electrolytically introduced hydrogen were determined by vacuum thermal (up to 500°C) extraction and subsequent gas chromatography.

Three-point bend tests were performed on a tensile testing machine with a capacity of 10 kN at a load rate of 1 mm/min.

To prevent hydrogen release prior to testing, hydrogenated specimens were held in liquid nitrogen.

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[*] Corresponding author. Tel.: +7-095 196 9233; fax: +7-095 196 1701.

E-mail address: ekrasikov@hotmail.com (E.A. Krasikov).

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Table 1
Chemical composition of 15Cr–2Mo–V steel (wt%)

C	Si	Mn	S	P	Cr	Ni	Mo	V	Cu
0.16	0.30	0.43	0.011	0.014	2.75	0.16	0.67	0.26	0.11

3. Experimental results and discussion

Fig. 1 shows the examples of load–strain diagrams for three-point bend tests of the irradiated, irradiated/annealed, and hydrogenated specimens.

Table 2 represents the detailed information on hydrogen–irradiated steel interaction during alternating hydrogenation and annealing.

It follows from these results that hydrogenation of the neutron irradiated specimens can modify strength parameters (reduces the strength of the material from 1690 MPa down to 1310 MPa), but it leads to a drastic decrease in plasticity (Table 2, step no. 2). The efficiency of recovery after irradiation depends on the temperature of annealing (step nos. 3,4).

Post-hydrogenation heat treatment leads to a recovery of the mechanical properties; in this case the higher the heat treatment temperature the greater the recovery efficiency (step nos. 5,6). In turn, post-irradiation annealing leads to a drop in hydrogen solubility and hydrogen embrittlement relief (step nos. 7,8).

In this connection, it should be noted that from the scientific and practical point of view, it is extremely interesting to clarify the metal's behavior under hydrogenation after the irradiated metal is heat-treated (annealed).

Important information can be gained from Table 2, where the influence of repeated cycles of 'annealing/hydrogenation/annealing' on neutron–irradiated steel is presented (step nos. 9–12). It is obviously seen that a cycling treatment with an 'annealing' episode as a final

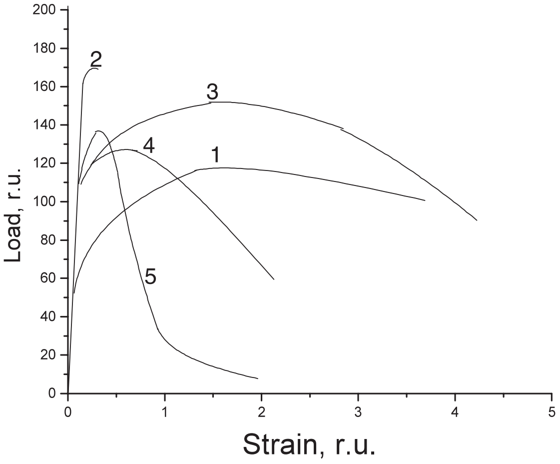

Fig. 1. Load–strain diagrams for three-point bend tests of the prismatic irradiated/annealed, hydrogenated specimens. Curve (1) represents the initial (unirradiated) condition; (2) irradiation up to 2.7×10^{20} cm^{-2} at 125°C; (3) irradiation up to 2.7×10^{20} cm^{-2} at 125°C and annealing; (4) irradiation up to 2.7×10^{20} cm^{-2} at 125°C, annealing, hydrogenation and re-annealing; and (5) irradiation up to 2.7×10^{20} cm^{-2} at 125°C, annealing, hydrogenation. Specimens were annealed at 300°C for 1 h.

Table 2
Influence of neutron irradiation (2.7×10^{20} cm^{-2}) at 125°C and repeated cycles of hydrogenation and annealing (at 250°C and 300°C for 1 h) on mechanical properties of 15Cr–2Mo–V steel[a]

Step of cycling	H (h/ppm)	A (°C)	RH (h/ppm)	RA (°C)	UTS (MPa)	YS (MPa)	f_{Pmax} (mm)	f_{tot} (mm)	A_{Pmax} (ru)	A_{tot} (ru)
1	N/A	N/A	N/A	N/A	1690	1670	0.06	0.12	1.6	2.5
2	24/25	N/A	N/A	N/A	1310	1310	0	0	0	0
3	N/A	250	N/A	N/A	1530	1260	0.9	2.3	12.3	33
4	N/A	300	N/A	N/A	1380	1030	1.5	3.7	15.3	48
5	24/25	250	N/A	N/A	1520	1330	0.4	1.6	5.8	21.5
6	24/25	300	N/A	N/A	1600	1300	1.3	3.3	19	49
7	N/A	250	24/13	N/A	1290	1130	0.15	0.2	1.9	0.5
8	N/A	300	24/6	N/A	1200	1000	0.3	0.8	3.1	8.6
9	N/A	250	4/17	N/A	1410	1330	0.1	0.1	1.3	1.5
10	N/A	300	4/8	N/A	1300	1070	0.4	0.9	4.9	10.7
11	N/A	250	4/17	250	1320	1230	0.2	0.9	1.8	10.4
12	N/A	300	4/8	300	1290	1030	0.4	1.9	4.9	21.5

[a] *Legend*: H (h/ppm) is hydrogenation (time in hours/hydrogen content in weight ppm); RH (h/ppm) is repeat hydrogenation (time in hours/hydrogen content in weight ppm); A is annealing; RA is repeat annealing; UTS is ultimate tensile strength; YS is yield strength; f_{Pmax} is magnitude of deformation up to maximum load; f_{tot} is magnitude of total deformation; A_{Pmax} is work required to deform the specimen up to maximum load in relative units; A_{tot} is work required to fracture the specimen in relative units.

condition (step nos. 11,12) leads to the degradation of the material in comparison with the consequences of a single ‘annealing’ (step nos. 3,4) or ‘hydrogenation + annealing’ (step nos. 5,6) episodes.

4. Conclusions

These studies suggest the existence of residual (unrecovered by annealing) degradation effects caused by hydrogen–irradiated steel interaction during alternating hydrogenation and annealing.

On one hand it may be worthwhile to treat periodically the structural components of the thermonuclear reactor subject to hydrogenation and irradiation at temperatures up to 200°C by annealing at higher temperatures. On the other hand, the repetition of annealing cycles may result in severe degradation of the irradiated hydrogenated–annealed steel.

We propose that this phenomenon must be taken into account when in-service properties of the candidate structural materials are analyzed.

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