ELSEVIER
Journal of Nuclear Materials 271&272 (1999) 92–96
journal of nuclear materials

Properties of precipitation hardened steel irradiated at 323 K in the Japan materials testing reactor

M. Niimi [a,*], Y. Matsui [a], S. Jitsukawa [b], T. Hoshiya [a], T. Tsukada [b], M. Ohmi [a], H. Mimura [a], N. Ooka [a], K. Hide [c]

[a] *Department of JMTR, Japan Atomic Energy Research Institute, 3607 Narita-cho, Oarai-machi, Higashi Ibaraki-gun, Ibaraki-ken, 311-1394, Japan*

[b] *Department of Materials Science and Engineering, Japan Atomic Energy Research Institute, Tokai-mura, Naka-gun, Ibaraki 319-1195, Japan*

[c] *Central Research Institute of Electric Power Industry (CRIEPI), Iwatokita, Komae city, Tokyo 201-0004, Japan*

Abstract

A precipitation hardening type 630 stainless steel was irradiated in the Japan Materials Testing Reactor (JMTR) in contact with the reactor primary coolant. The temperature of the irradiated specimens was about 330 K. The fast neutron ($E > 1$ MeV) fluence for the specimens ranged from 10^{24} to 10^{26} m^{-2}. Tension tests and fracture toughness tests were carried out at room temperature, while Charpy impact tests were done at temperatures of 273–453 K. Tensile strength data showed a peak of 1600 MPa at around 7×10^{24} m^{-2}, then gradually decreased to about 1500 MPa at 1.2×10^{26} m^{-2}. The elongation decreased with irradiation from 12% for unirradiated material to 6% at 1.2×10^{26} m^{-2}. The fractography after the tension test revealed that the fracture was ductile. Fracture toughness decreased to about a half of the value for unirradiated material with irradiation. The cleavage fracture was dominant on the fractured surface. Charpy impact tests showed an increase of ductile–brittle transition temperature (DBTT) by 60 K with irradiation.

1. Introduction

The precipitation hardening type 630 stainless steel (630PH) is considered to be a candidate material for joints of future fusion reactor such as the International Thermonuclear Experimental Reactor (ITER). However, there have been limited number of data on the mechanical properties of irradiated 630PH. Due to its intergranular structure, 630PH steel has rather high fracture strength but its toughness is not very high. Therefore, it is important to evaluate the irradiation effect on the ductile–brittle transition temperature (DBTT) and the fracture toughness. In order to collect such data, test specimens of the 630PH were irradiated in the Japan Materials Testing Reactor (JMTR; 50 MW t) in which the reactor coolant temperature was 323 K. As for post-irradiation examinations, tension tests, fracture toughness tests and Charpy impact tests were curried out.

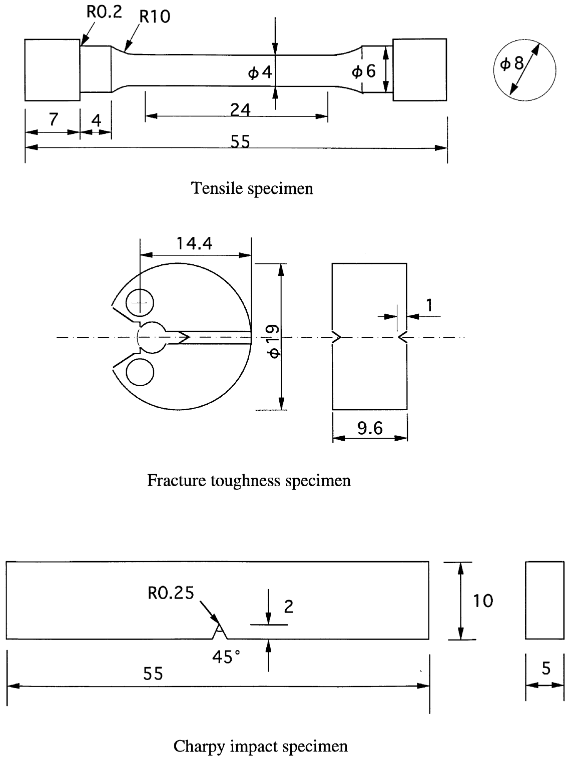
2. Experimental procedure

2.1. Irradiation tests

The shapes and the dimensions of tension test specimens, fracture toughness test specimens (Disk-Shaped Compact Specimen, DCT) and Charpy impact test specimens are shown in Fig. 1. The chemical composition of unirradiated material is shown in Table 1. Neutron irradiation was performed in the JMTR, which has 50 MW of thermal output and uses light water as the reactor coolant. The highest neutron flux is 2×10^{18} m^{-2} s^{-1} ($E > 1$ MeV) and 4.4×10^{18} m^{-2} s^{-1} ($E > 0.1$ MeV) in the reactor fuel region. The ($E > 1$ MeV) neutron fluence for each irradiated specimen ranged from

* Corresponding author. Tel.: +81-29 264 8345; fax: +81-29 264 8480; e-mail: nii@oarai.jaeri.go.jp.

0022-3115/99/$ – see front matter
PII: S0022-3115(98)00699-0



(unit : mm)

Fig. 1. Dimensions of specimens.

5.9×10^{24} to 1.2×10^{26} m^{-2}, while the neutron fluence ranged from 1.3×10^{25} to 2.6×10^{26} m^{-2}. Each specimen was irradiated with direct cooling by reactor primary cooling water at 323 K. Typical surface temperature and center temperature of the specimens were evaluated by calculation at ~330 and ~335 K respectively.

2.2. Post-irradiation tests

Tension tests were carried out at 293 K in air. The cross-head speed was 0.1 mm/min. Measured data were yield strength, tensile strength, fracture strength and elongation. Reduction and organization of fracture surface of the specimen were observed by optical microscope.

Fracture toughness tests were carried out using DCT (outer diameter 19 mm) with pre-cracking by fatigue according to the ASTM Standards E813-89 and E399. The loading speed was 0.5 mm/min, and the tests were conducted in air at 293 K. A hydraulic type test machine generating a load of 6.2×10^4 N was used, and a clip gauge was used for measuring a crack opening displacement (2–10mm). The relationship between displacement obtained by the clip gauge and load applied was examined. The crack length of the initial fatigue and pre-crack was measured by fractography after the test.

Charpy impact tests were carried out using a remote-controlling machine capable of 30 kg m, at the temperatures ranging from 273 to 453 K. The test temperature was controlled using silicone oil as medium in a bath in which the irradiated specimens were held for 15 min. Data of absorbed energy, percent shear fracture and lateral expansion were obtained. An optical microscope and a scanning electron microscope (SEM) were used in the fractography.

3. Results and discussion

3.1. Tensile properties

The results of tension tests are shown in Fig. 2. It was indicated that the tensile strength and the yield strength are increasing with neutron fluence until 7×10^{24} m^{-2}, then gradually decreasing with further irradiation. The tensile strength changed from 1400 MPa for unirradiated specimens to a peak of 1600 MPa at $\sim 7 \times 10^{24}$ m^{-2}, and then gradually decreased to about 1500 MPa at $\sim 1.2 \times 10^{26}$ m^{-2}. These are different from the data obtained for the austenitic stainless steels (SUS304 and SUS316) irradiated under the same condition. The austenitic stainless steels did not show a lowering of the yield strength with irradiation [1]. A possible cause of such lowering of the yield strength of 630PH with irradiation is considered to be a change in the precipitate shape by irradiation.

The fracture strength was measured at 1050 MPa for unirradiated specimen, and ~1100 MPa for the irradiated specimen. The fracture did not occur at the central part of parallel area of specimen. The total elongation of the unirradiated specimen was measured at 12%. The elongation was lowered with the increase of the yield strength [1–5]. It decreased to ~7% at a fluence of 7×10^{24} m^{-2} and to ~6% at $>6 \times 10^{25}$ m^{-2}. As shown in Fig. 3, there is ~45% reduction at 2×10^{25} m^{-2} and a 25% reduction at 1.2×10^{26} m^{-2}.

A SEM photograph of one fracture surface is shown in Fig. 4(a). Dimples were observed on the fracture surface regardless of the neutron fluence. In the optical

Table 1
Chemical composition (wt%)

Elements	C	Si	Mn	P	S	Ni	Cr	Cu	Nb	Co
630PH	0.043	0.57	0.31	0.020	0.012	4.17	15.87	3.39	0.26	<0.01

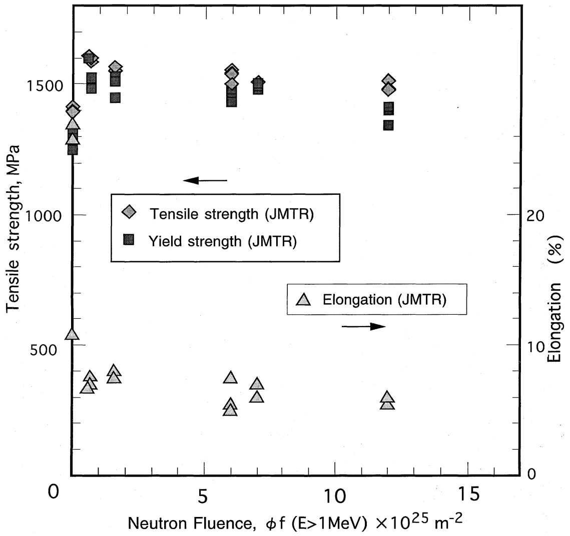


Fig. 2. Dose dependence of the tensile properties of 630PH. Yield strength and tensile strength increased with dose to about 1×10^{25} m^{-2} and remained almost constant for higher doses to 1.2×10^{26} m^{-2}. Elongation decreased with neutron dose. Specimens were irradiated in JMTR.

microscope, there was no clear difference of fracture surfaces between irradiated and unirradiated specimens.

3.2. Fracture toughness

The relationship between applied load and crack opening displacements was almost linear with increasing crack opening displacement until fracture occurred. The *J*-values were not measured since stable crack growth

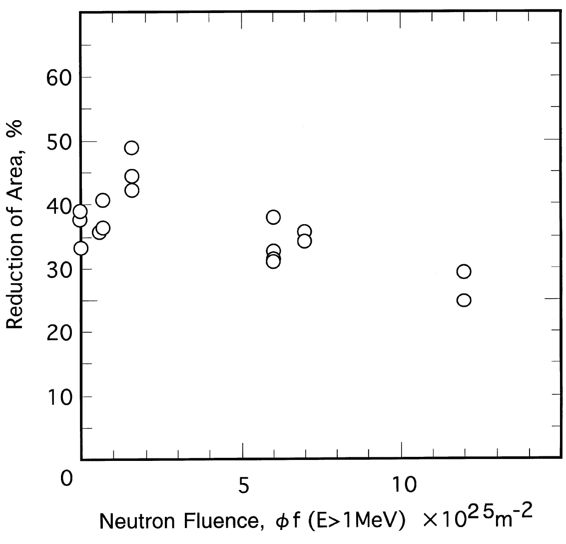


Fig. 3. Reduction of area of 630PH steel tested at 298 K.

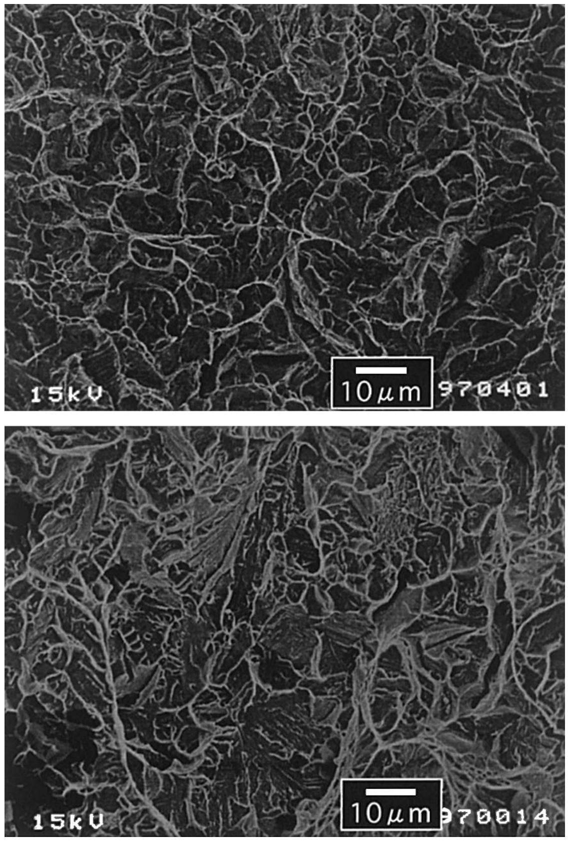


Fig. 4. (a) Fracture surface of an irradiated tensile specimen tested at 293 K. Dimples were seen on the surface. (b) Cleavage fracture was seen in the Charpy impact specimen tested at 353 K. Fractographs of tensile and Charpy impact specimens irradiated to 1.2×10^{26} m^{-2} ($E > 1$ MeV).

was not observed. The *K*-value was evaluated from the applied load, the specimen form and the crack length at fracture. As shown in Fig. 5, the *K*-value was 46 MPa $m^{1/2}$ for the unirradiated specimen, and was decreasing with irradiation to 25 MPa $m^{1/2}$ at 3×10^{25} m^{-2}. The lowering of fracture toughness saturated with neutron fluence. The fracture surface was observed to be almost flat. The SEM observation shows that the cleavage fracture covered the entire surface.

3.3. Charpy impact property

The relationship obtained by the Charpy impact tests between the percent shear fracture and the test temperature is shown in Fig. 6. Because of the high DBTT of the 630PH steel, insufficient were obtained for impact energy region. Therefore, DBTT was determined by using the temperature of the 50% ductile fracture.

The DBTT, which was 373 K for unirradiated material, increased to 400 K at 8×10^{24} m^{-2} and 433 K at $>3 \times 10^{25}$ m^{-2}. The relationship between DBTT and the

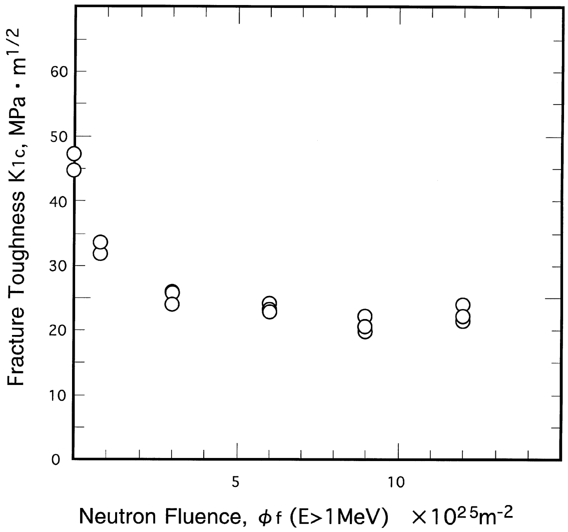


Fig. 5. Dose dependence of the fracture toughness (K_{IC}) of 630PH at 293 K. K_{IC} becomes ~25 MPa $m^{1/2}$ above a dose of 3×10^{25} m^{-2}.

neutron fluence for 50% ductile fracture is shown in Fig. 7. The increase of DBTT with neutron irradiation showed a tendency to saturate. It is well known that DBTT of the ferrite/martensitic steel is increased by neutron irradiation. DBTT for the material HT-9, a typical martensitic steel and used for ducts in Fast Breeder Reactor (FBR), is known to increase more than 200 K with neutron irradiation. The increase of DBTT of the 630PH was only 60 K.

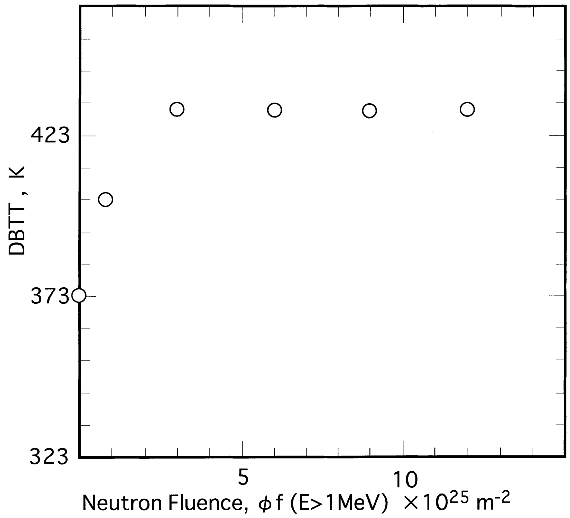


Fig. 7. Dose dependence of DBTT at 50% shear fracture. The DBTT shift saturates with dose.

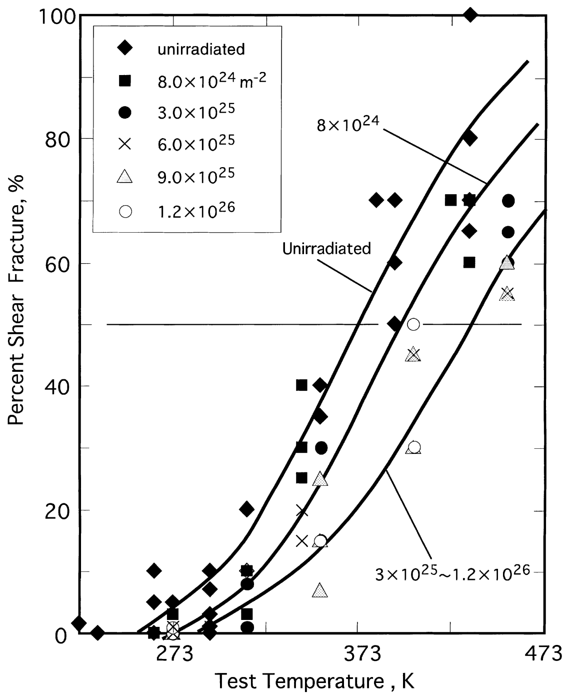


Fig. 6. Temperature dependence of the percent shear fracture before and after the irradiation. Irradiation caused a decrease in the percent shear fracture.

The central part of the fracture surface was observed by SEM. Cleavage was observed on fracture surface in specimens with ductile fracture of 0–30%. The effect of neutron irradiation cannot be observed from Fig. 4(b) and Fig. 8.

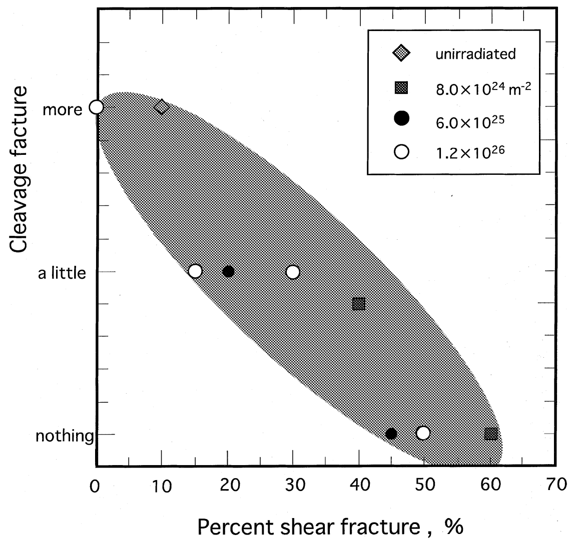


Fig. 8. Dose dependence of cleavage and percent shear fracture. There is no clear influence of dose.

4. Conclusions

To evaluate the mechanical properties of 630PH, the data of irradiated 630PH were measured as by their dependence on neutron fluence ranging from 5.9×10^{24} to 1.2×10^{26} m^{-2} in the JMTR. Tension tests (at 293 K), fracture toughness tests (at 293 K) and Charpy impact tests (at 273–453 K) were carried out.

1. The tensile strength increased from 1400 MPa for unirradiated specimen to a peak of 1600 MPa for irradiated specimen.
2. The total elongation decreased from 12% for unirradiated specimen to 7% at 7×10^{24} m^{-2}, and decreased about 6% at $>6 \times 10^{25}$ m^{-2}.
3. There was a 45% reduction of area at 2×10^{25} m^{-2} and a 25% reduction of area at 1.2×10^{26} m^{-2}.
4. The *K*-value decreased from 46 MPa $m^{1/2}$ for unirradiated specimen to 25 MPa $m^{1/2}$ at $>3 \times 10^{25}$ m^{-2}. The lowering of fracture toughness seems to saturate with increasing neutron fluence.
5. The DBTT was 373 K for unirradiated specimens, increased to 400 K at 8×10^{24} m^{-2} and 433 K at $>3 \times 10^{25}$ m^{-2}. The increase of DBTT with neutron irradiation seems to saturate. Cleavage fracture was observed on fracture surface in specimens of ductile fracture 0–30%.

It can be conclusively said that mechanical properties of the 630PH steel changed extensively within a small range of neutron fluence, and then the properties showed a tendency toward saturation with increasing of neutron fluence.

Acknowledgements

This research was performed under CRIEPI/JMTR project collaboration.

References

[1] Y. Matsui, M. Niimi, T. Hoshiya, F. Sakurai, S. Jitsukawa, T. Tsukada, M. Ohmi, H. Sakai, R. Oyamada, T. Onchi, J. Nucl. Mater. 233–237 (1996) 188.
[2] Division of JMTR Project, Surveillance Test of the JMTR Core Components – The results of post-irradiation examination – JAERI-M 5936, 1974.
[3] T. Takeda, H. Amezawa, K. Tobita, JAERI-M 86-007, 1986.
[4] H.D. Gronbeck, ETR Radiation Damage Surveillance Programs Progress Report II, IN-1036.
[5] H.J. Rack, SAND 80-2302, TTC-0161, 1981, p. 63.