Contents lists available at ScienceDirect

## Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat



# Fracture toughness of irradiated modified 9Cr-1Mo steel

Sung Ho Kim, Ji-Hyun Yoon\*, Woo Seog Ryu, Chan Bock Lee, Jun Hwa Hong

Korea Atomic Energy Research Institute, Daejeon, Republic of Korea

## ABSTRACT

The effects of irradiation on fracture toughness of modified 9Cr–1Mo steel in the transition region were investigated. Half size precracked Charpy specimens were irradiated up to  $1.2 \times 10^{21}$ n/cm<sup>2</sup> (E > 0.1 MeV) at 340 °C and 400 °C in the Korean research reactor. The irradiation induced transition temperature shift for a modified 9Cr–1Mo was evaluated by using the Master Curve methodology. The  $T_0$  temperature for the unirradiated specimens were measured as -67.7 °C and -72.4 °C from the tests with standard PCVN (precracked charpy V-notch) and half sized PCVN specimens, respectively. The  $T_0$  shifts of specimens after irradiation at 340 °C and 400 °C were 70.7 °C and 66.1 °C, respectively. The Weibull slopes for the fracture toughness data obtained from the unirradiated and irradiated modified 9Cr–1Mo steels were determined to confirm the applicability of master curve methodology to modified 9Cr–1Mo steel.

© 2008 Elsevier B.V. All rights reserved.

#### 1. Introduction

9-12%Cr ferritic/martensitic steels have been used most extensively in the power-generation industry throughout the world due to their many advantages such as low thermal expansion coefficient, high thermal conductivity, high elevated temperature strength, and adequate creep resistance [1]. These steels are the proposed candidates for the crosscutting materials of the advanced nuclear power system. It is important to understand the effects of neutron irradiation on material properties for application these materials to nuclear power system. The irradiation effect on the fracture toughness of structural materials is one of the main concerns for the designing of the fusion devices. Nuclear materials are required to have an adequate fracture resistance so that they can withstand a high pressure, high-temperature and a neutron radiation environment. The current fracture toughness requirements in the surveillance programs for the light water reactor are based on the drop-weight and Charpy impact properties which had their origin in the old test methods that were not directly fracture mechanics related [2]. Therefore, the T<sub>0</sub> reference temperature and the J-R fracture resistance were introduced recently to the integrity assessment of the nuclear materials adding to the conventional fracture properties [3]. It is expected that the fracture toughness parameters such as the  $T_0$  reference temperature will be applied to the radiation embrittlement analysis for the nuclear materials of future nuclear systems. While it has been shown that

E-mail address: jhyoon4@kaeri.re.kr (J.-H. Yoon).

modified 9Cr-1Mo has excellent thermal properties and a high-temperature strength, the data on the effects of radiation on fracture toughness of modified 9Cr-1Mo are limited [4].

In this study,  $T_0$  reference temperature was evaluated for an irradiated modified 9Cr–1Mo by using the Master Curve method. This study also focused on confirming the applicability of Master Curve method to modified 9Cr–1Mo.

#### 2. Experimental procedures

The test material was a 16 mm thick commercial modified 9Cr– 1Mo plate which was normalized at 1050 °C and tempered at 770 °C. The chemical composition of material is listed in Table 1. The half sized precracked Charpy specimens with dimensions of *W*: 5 mm, *T*: 5 mm, and *L*: 27.5 mm were fabricated in *T*–*L* orientation. These specimens were irradiated at the Korean research reactor, HANARO. The specimens were irradiated up to  $1.2 \times 10^{21}$  n/ cm<sup>2</sup> (*E* > 0.1 MeV) which produced 0.85 dpa in the instrumented capsules filled with helium gas and designed to maintain temperatures of 340 °C and 400 °C. The measured irradiation flux was  $2.5 \times 10^{13}$ n/cm<sup>2</sup> (*E* > 0.1 MeV) with ±10% uncertainty. The *K*<sub>Jc</sub> and *T*<sub>0</sub> reference temperature data for unirradiated specimens were generated by using precracked Charpy specimens both of a standard and half size to investigate size effect.

The  $K_{\rm Jc}$  tests were conducted in a servohydraulic test system MTS 810 TestStar in conjunction with an environmental chamber which is facilitated in a hot cell. The loading rate was 0.1 mm/min. The multi-temperature method was used to determine  $T_0$ . The detailed test procedures followed ASTM Standard E 1921-05.

<sup>\*</sup> Corresponding author. Tel.: +82 42 868 8554.

#### Table 1

Chemical composition of material (modified 9Cr-1Mo) in wt%.

С	Mn	Р	S	Si	Cr	Мо	Ni	V	Nb	Ν	Al
0.085	0.379	0.019	0.0008	0.336	9.376	0.911	0.097	0.189	0.080	0.0420	0.032

#### 3. Results and discussion

#### 3.1. $T_0$ reference temperature

The variation of  $K_{\rm lc}$  static fracture toughness with the temperature for an unirradiated modified 9Cr-1Mo plate is shown in Fig. 1. The Master Curves corresponding to  $K_{\rm Ic}$  data for the standard and half size PCVN specimens were generated as shown in Fig. 1. The tension test data for the test material were obtained at the temperatures above room temperature. Therefore, the  $K_{\rm lc}$  censoring limits presented in Fig. 1 were generated from the yield stresses extrapolated by polynomial fitting from available yield stress data measured above room temperature. Master Curve expresses the trend of the fracture toughness change with the temperature for unirradiated modified 9Cr-1Mo steel successfully. However, some  $K_{\rm lc}$ data was located below 5% tolerance bound curve and above 95% tolerance bound curve. To measured by using the standard PCVN and a half size PCVN were -67.7 °C and -72.4 °C respectively. It means that the data obtained from the smaller specimen is less conservative. It is a generally observed phenomenon which is attributed to the loss of a constraint in the smaller specimen [5].

The change of  $K_{\rm Jc}$  static fracture toughness with the temperature for modified 9Cr–1Mo steel after irradiated at 340 °C and 400 °C is plotted in Fig. 2. Master Curve corresponding to the  $K_{\rm Jc}$ data was generated as shown in Fig. 2.  $T_0$ , reference temperatures measured for the half size charpy specimens irradiated at 340 °C and 400 °C were –1.7 °C and –6.3 °C, respectively. The difference in reference temperance shift between two irradiation temperatures was not significant. The  $T_0$  temperature shifts for the modified 9Cr–1Mo steel after irradiation were 70.7 °C and 66.1 °C, respectively, at 340 °C and 400 °C as shown in Fig. 3.

Klueh et al. reported that the shift in transition temperature of a modified 9Cr–1Mo was 52 °C for irradiation to 13 dpa at 390 °C [6]. Sokolov et al. reported the shift in reference temperature for F82H steel irradiated up to 5 dpa at 300 °C was about 110 °C [7]. The irradiation induced increase in yield strength at 400 °C was about just 10% in the authors' study. However, embrittlement was observed in F82H steel at 500 °C without any change in strength in recent



Fig. 1. Master Curve determined for unirradiated modified 9Cr-1Mo steel obtained for PCVN and 0.5PCVN specimens.



Fig. 2. Master Curves determined for modified 9Cr–1Mo steels obtained for 0.5PCVN specimens irradiated at 340  $^\circ$ C and 400  $^\circ$ C.



**Fig. 3.**  $T_0$  transition temperature shifts for modified 9Cr-1Mo steel irradiated at 340 °C and 400 °C.

study [7]. The embrittlement without significant hardening could be attributed to irradiation-enhanced precipitation.

#### 3.2. Weibull slope

ASTM E 1921 standard test method for determination of reference temperature,  $T_0$  is based on the weakest-link-theory used to describe the brittle fracture of a polycrystalline material and the following three-parameter Weibull model [8]:

$$P_{\rm f} = 1 - \exp\{-[(K_{\rm Jc} - K_{\rm min})/(K_0 - K_{\rm min})]^{\rm p}\}.$$
(1)

 $P_{\rm f}$  is the probability at  $K_{\rm I} \leq K_{\rm Jc}$  for an arbitrarily chosen specimen from a specimen set. The Weibull slope in this case is b for a cleavage fracture of low alloy steel was set equal to 4, and  $K_{\rm min}$  was set equal to 20 MPa $_{\rm V}$ m for a ferritic steel.

Unfortunately the data obtained at each single temperature were not enough to measure the Weibull slope in this study, the Weibull slope indicated in Fig. 4 was measured for the gathered data from all the test temperatures in an engineering sense. It



Fig. 4. 3-Prameter Weibull plot for fracture toughness data of modified 9Cr-1Mo steel.

was assumed that the Weibull slopes didn't depend on temperature even though,  $K_0$  depended on temperature.

The experimental Weibull slopes determined for the unirradiated and irradiated modified 9Cr-1Mo are 3.1 and 2.3, respectively, as shown Fig. 4. They are much lower than the slope for typical low alloy steels. The lower is Weibull slope, the higher is the data scatter. It might be attributed to various possible reasons. It was reported that the lower Weibull slopes for ferritic-martensitic steels were attributed to constraint loss in small specimens in the previous research [8]. The small number of test specimens used to generate the Weibull plot can be another reason. It is known that the Weibull slope can only be evaluated accurately when a sample size is sufficiently large (of the order of 100 specimens) [9]. The intrinsic factors such as the microstructural inhomogeneity and anisotropy in a rolled plate could be a possible reason also.

## 4. Summary

The effects of irradiation on the fracture toughness of modified 9Cr-1Mo steel in the transition region were investigated by using the Master Curve methodology.

- 1. T<sub>0</sub> reference temperatures of unirradiated modified 9Cr-1Mo measured by using a standard PCVN and a half size PCVN were -67.7 °C and -72.4 °C respectively.
- 2. T<sub>0</sub> reference temperatures of modified 9Cr-1Mo irradiated at 340 °C and 400 °C were -1.7 °C and -6.3 °C, respectively. The  $\Delta T_0$  of specimens irradiated at 340 °C and 400 °C were 70.7 °C and 66.1 °C, respectively.
- 3. Master Curve expressed the temperature dependence of static fracture toughness data for the unirradiated and irradiated modified 9Cr-1Mo steel successfully. But the scattering of data was a little larger when compared to typical low alloy steels.

### Acknowledgements

This work has been carried out as a part of the development of basic key technologies for Gen IV SFR fuel under the mid- and longterm nuclear R&D plans by the Korean Ministry of Science and Technology.

#### References

- [1] R.L. Klueh, Elevated-Temperature Ferritic and Martensitic Steels and their Application to Future Nuclear Reactors, ORNL/TM-2004/ 176, Oak Ridge National Laboratory, Oak Ridge, USA, 2004.
- Vessels Materials, Washington DC, USA, 1988.
- Pressure Vessel Integrity in Nuclear Power Plants, Technical Report Series No. 429, IAEA, Vienna, Austria, 2005.
- nuclear application, Mornograph 3 in ASTM'S Monograph Series, ASTM, PA, USA, 2001.
- [6] R.L. Klueh et al., J. Nucl. Mater. 377 (2008) 427.

- USNRC, Regulatory Guide 1.99, Rev. 2, Radiation Embrittlement of Reactor
- IAEA, Guidelines for Application of the Master Curve Approach to Reactor
- [4] R.L. Klueh, D.R. Harries, High chromium ferritic and martensitic steels for
- G.R. Odette et al., J. Nucl. Mater. 329-333 (2004) 1243. [5]
- [7] M.A. Sokolov, H. Tanigawa, J. Nucl. Mater. 367-370 (2007) 587.
- [8] E. Locon, J. Nucl. Mater. 367-370 (2007) 575.
- [9] K. Wallin, Eng. Fract. Mech. 19 (6) (1984) 1085.