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# Thermal fatigue crack nucleation in ferritic–martensitic steels before and after neutron irradiation

L.A. Belyaeva<sup>a</sup>, A.A. Zisman<sup>a</sup>, C. Petersen<sup>b,\*</sup>, V.A. Potapova<sup>a</sup>, V.V. Rybin<sup>a</sup>

<sup>a</sup> Central Research Institute of Structural Materials "Prometey", Shpalernaya 49, 193015 St. Petersburg, Russian Federation <sup>b</sup> Forschungszentrum Karlsruhe Technik und Umwelt, Institut für Materialforschung II, Postfach 3640, D 76021, Karlsruhe, Germany

# Abstract

Thermal fatigue behaviour of the ferritic–martensitic steels MANET-II, 12Cr–1.5NiMo and F82H-mod. have been investigated in the temperature range from 50°C to 350°C and total strain range  $\leq 0.33\%$ . Crack appearance has been checked after  $3 \times 10^3$ ,  $6 \times 10^3$  and  $10^4$  cycles and has been successively detected in these steels. The thermal fatigue cracks have a transgranular character; sometimes, intergranular cracks are observed in the F82H-mod. steel. A certain correlation of grain size and ferrite content with the thermal fatigue crack peculiarities has been noted. Specimens of MANET-II and 12Cr–1.5NiMo have been irradiated in a WWR-M reactor with a fluence of  $1 \times 10^{25}$  n m<sup>-2</sup> at a temperature of 300°C and then subjected to thermocyclic loading. It has been established that the neutron irradiation does not significantly affect fatigue crack nucleation in both materials. © 2000 Elsevier Science B.V. All rights reserved.

### 1. Introduction

Ferritic-martensitic steels containing 8–12% Cr are candidate structural materials for the blanket and first wall of DEMO fusion reactors. The cyclic character of thermal, mechanical and neutronic loading that these steels will be subjected to poses the problem of thermal fatigue, as well as the problem of influence of irradiation on fatigue behaviour. The objective of the present research is the investigation of thermal fatigue crack nucleation in the ferritic-martensitic steels MANET-II, 12Cr–1.5NiMo and F82H-mod. in the unirradiated state and of MANET-II and 12Cr–1.5NiMo after neutron irradiation.

# 2. Experimental

The former European ferritic-martensitic reference steel MANET-II, the Russian ferritic-martensitic steel

 $12Cr{-}1.5NiMo$  and the Japanese reduced-activation ferritic-martensitic steel F82H-mod. have been investigated. Chemical compositions (in wt%) are:

- MANET-II: 0.1 C, 10.3 Cr, 0.65 Ni, 0.57 Mo, 0.19 V, 0.14 Nb, 0.14 Si, 0.75 Mn, 0.08 B, 0.03 N;
- F82H-mod.: 0.09 C, 7.68 Cr, 0.15 V, 0.01 Nb, 0.16 Mn, 0.03 Ta, 2.2 W, 0.007 N;
- 12Cr-1.5NiMo: 0.04 C, 11.4 Cr, 1.5 Ni, 0.9 Mo, 0.13
   Si, 0.45 Mn, 0.02 W, 0.001 N, 0.09 S, 0.03 Ti, 0.008 P, 0.04 Cu.

The heat treatments are:

- MANET-II: homogenization 2 h at 960°C (air cooled), austenitization 30 min at 1075°C (air cooled), tempering 2 h at 750°C (air cooled);
- F82H-mod.: austenitization 38 min at 1040°C (air cooled), tempering 1 h at 750°C (air cooled);
- 12Cr-1.5NiMo: normalization 2h at 1000°C (air cooled), tempering 2h at 700°C (air cooled).

Cylindrical specimens with a diameter of 8 mm and a length of 30 mm with four longitudinal notches having a depth of 0.5 mm with different radii of 0.1, 0.25, 0.5 and 1.0 mm were used. Thermocyclic loading of the specimens up to  $10^4$  cycles was performed by heating these in an electric furnace under air atmosphere up to  $350^{\circ}$ C for 10 min, followed by cooling in distilled water down to  $50^{\circ}$ C for 1.5 min.

<sup>\*</sup>Corresponding author. Tel.: +49-7247 823 267; fax: +49-7247 823 826.

E-mail address: claus.petersen@imf.fzk.de (C. Petersen).

The microstructure of the specimens and the crack appearance near the notch tip were investigated by optical microscopy after about  $3 \times 10^3$ ,  $6 \times 10^3$  and  $10^4$ cycles. Thermal stress varied with notch radius. Thermal stresses and strains have been evaluated as follows. To obtain the elastic strain range ( $\Delta \varepsilon_{el}$ ), the thermo-elastic problem was first solved for a notch-free cylinder, and the notch was then included as a stress concentrator [1]. To estimate plastic strains ( $\Delta \varepsilon_{pl}$ ) and, consequently, total strains ( $\Delta \varepsilon_t$ ), the peak stress ( $\sigma^*$ ) in the cycle at the notch was compared with the yield stress values  $\sigma_{0.2}^{(0)}$  and  $\sigma_{0.2}^{(s)}$ , corresponding to the stress-free temperature  $T_0$  and the local temperature  $T^*$  at the moment the thermal stress reaches  $\sigma^*$ , respectively; that is,

for 
$$\sigma^* > \sigma_{0.2}^{(0)} + \sigma_{0.2}^{(*)} \Rightarrow \Delta \varepsilon_{\text{pl}} = (1 - \nu)(\sigma^* - \sigma_{0.2}^{(0)} - \sigma_{0.2}^{(*)})/G;$$
(1a)

for 
$$\sigma^* \leqslant \sigma_{0,2}^{(0)} + \sigma_{0,2}^{(*)} \Rightarrow \Delta \varepsilon_{\text{pl}} = 0;$$
 (1b)

with shear modulus G and Poisson ratio v.

Similar experiments have been carried out with specimens after neutron irradiation in the WWR-M reactor of the Petersburg Nuclear Physics Institute (PNPI), at a temperature of about 300°C with a fluence of  $1 \times 10^{25}$  n m<sup>-2</sup>. The specimens were irradiated in hermetic aluminum ampoules at a flux of  $3 \times 10^{13}$  n cm<sup>-2</sup> s<sup>-1</sup>. Thermocyclic tests of irradiated specimens as well as grinding, polishing and light microscopy have been performed by remote-controlled machines installed in a 'hot' cell [2].

#### 3. Results and discussion

Figs. 1 and 2 present microstructures of unirradiated and irradiated MANET-II and 12Cr–1.5NiMo specimens before and after thermocycling. MANET-II has a fine-grained structure with a grain size of 30–40  $\mu$ m, while 12Cr–1.5NiMo has larger variation in grain size. F82H-mod. (Fig. 3) is the most coarse-grained (80–100  $\mu$ m) among the three materials. It can be seen from Figs. 1–3 that thermal cycling generated cracks of 0.01–0.02 mm length at the stress concentrators. Within 10<sup>4</sup> cycles, the cracks in these steels grew slowly, increasing from 0.01 to 0.017–0.02 mm, and along with these first cracks, additional cracks were generated. The cracks commonly had a transgranular character, but, sometimes, intergranular cracks were detected in the steel F82H-mod.

Cracks 0.01–0.02 mm in length appeared in specimens of F82H-mod. after  $3 \times 10^3$  thermal cycles and in specimens of 12Cr–1.5NiMo and MANET-II after  $6 \times 10^3$  and  $10^4$  cycles, respectively.

The appearance of thermal fatigue cracks more than 0.01 mm in length at different total strain ranges are presented in Table 1. The data indicate that the MA-NET-II steel has the best thermal fatigue endurance and that 12Cr–1.5NiMo behaves better than the F82H-mod. steel. A plausible explanation of this result is that the F82H-mod. steel is the most coarse-grained of the three (Fig. 3), while the 12Cr–1.5NiMo steel, in relation to MANET-II, has a larger variation in grain size, facilitating plastic flow heterogeneity. Moreover, crack nucleation in all three steels was sometimes detected within



Fig. 1. Microstructure of MANET-II steel in unirradiated (a) and (b) and irradiated (c) states: (a) N=0; (b) and (c)  $\Delta \varepsilon_t = 0.32\%$ ,  $N_c = 10158$ .



Fig. 2. Microstructure of 12Cr–1.5NiMo steel in unirradiated (a) and (b) and irradiated (c) states: (a) N = 0; (b) and (c)  $\Delta \varepsilon_t = 0.21\%$ ,  $N_c = 6089$ .



Fig. 3. Microstructure of F82H-mod. steel in unirradiated state: (a) N = 0, (b)  $\Delta \varepsilon_t = 0.23\%$ ,  $N_c = 6089$ , (c)  $\Delta \varepsilon_t = 0.33\%$ ,  $N_c = 10158$ .

Table 1 Appearance of the cracks of more than 0.01 mm length ('+' – present, '-' – absent)

Material, state	$\Delta \varepsilon$											
	0.13-0.14% $N_{\rm c}$ (×10 <sup>3</sup> )			0.16-0.18% $N_{\rm c}~( imes 10^3)$			0.21-0.23% $N_{\rm c}$ (×10 <sup>3</sup> )			0.31–0.33% N <sub>c</sub> (×10 <sup>3</sup> )		
	3	6	10	3	6	10	3	6	10	3	6	10
MANET-II, unirradiated	-	-	-	-	-	-	-	-	+	-	-	+
MANET-II, irradiated	-	-	-	-	-	-	-	-	+	-	-	+
12Cr-1.5NiMo, unirradiated	-	-	-	-	-	-	-	+	+	-	+	+
12Cr-1.5NiMo, irradiated	-	-	-	-	-	-	-	+	+	-	+	+
F82H-mod., unirradiated	-	-	-	-	+	+	+	+	+	+	+	+

a ferrite phase, which is softer and results in plastic strain localization, Figs. 1 and 2.

Neutron damage of MANET-II and 12Cr-1.5NiMo up to 0.1 dpa does not influence the thermal fatigue crack nucleation of these materials (Table 1). This behaviour is similar to that of isothermal fatigue, which has been attributed to the rapid 'sweeping out' of radiation defects by the cyclic plastic strain [3]. However, this does not mean that the influence of irradiation on the degradation of 'in-pile' fatigue can be neglected. The interaction of deformation and irradiation, produced simultaneously 'in-pile' but not in postirradiation examination, may result in accelerated damage [4]. Thus, 'in-pile' creep of austenitic steels has shown an order of magnitude lower lifetime with respect to that of pre-irradiated materials [5]. The study of both mechanical and thermal fatigue under irradiation, however difficult this test may be, is, therefore, an urgent materials science problem in designing fusion reactors.

## 4. Conclusion

Thermal fatigue crack nucleation of MANET-II, 12Cr–1.5NiMo and F82H-mod. steels has been investigated during thermocycling from 50°C to 350°C in the unirradiated state.

Thermal fatigue cracks of 0.01–0.02 mm length are first formed in specimens of MANET-II, 12Cr–1.5NiMo and F82H-mod. after  $3 \times 10^3$ ,  $6 \times 10^3$  and  $10^4$  cycles,

respectively. The ranking order given above correlates with microstructure: F82H-mod. is the most coarsegrained, while 12Cr-1.5NiMo differs from MANET-II by a larger variation in grain size and apparent ferrite content.

Neutron damage of 0.1 dpa of MANET-II and 12Cr-1.5NiMo steels at a temperature of about 300°C does not have any significant effect on their thermal fatigue behaviour.

Thermal fatigue cracks in specimens of these ferriticmartensitic steels commonly have a transgranular character, but the cracks of F82-mod. steel are sometimes intergranular.

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## References

 G. Neiber, Stress Concentration, OGIZ, Gostekhizdat, Moscow, 1947.

- [2] L.A. Belyaeva, V.A. Gosudarencova, V.D. Yaroshevich, Shipconstr. Ind. Ser. Metall. 3 (1986) 38.
- [3] M.I. De Vries, ASTM STP 782 (1982) 665.
- [4] J. Bertsch, R. Lindau, A. Möslang, J. Nucl. Mater. 233–237 (1996) 276.
- [5] H. Ullmaier, J. Nucl. Mater. 133&134 (1985) 100.