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# Low cycle fatigue properties of ODS ferritic-martensitic steels at high temperature

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#### Abstract

Strain-controlled low cycle fatigue tests were conduced for 9Cr–ODS and 12Cr–ODS steels at 873, 923, 973 and 1023 K. The total strain ranges were controlled from 0.5% to 1.5% with strain rate of 0.1%/s. Corresponding plastic strain ranged from 0.01% to 1%. The ODS steels exhibit relatively low level of plastic strain and thus have longer fatigue life in the low total strain region compared to conventional ferritic steels such as Mod. 9Cr–1Mo steel. Neither noticeable cyclic hardening nor softening was observed in ODS steels, whilst Mod. 9Cr–1Mo steel shows an apparent cyclic softening at 873 and 923 K. This is attributable to the excellent microstructure stability due to the presence of nano-sized oxide particles dispersed in ODS steels.

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## 1. Introduction

The oxide dispersion strengthened (ODS) ferritic-martensitic steels with superior creep strength above 973 K are prospective materials not only for the long-life cladding tubes of the advanced fast reactor fuel elements but also for structural materials of blanket systems of the DEMO fusion reactor. In JAEA (Japan Atomic Energy Agency), extensive efforts have been made to develop 9Cr–ODS and 12Cr–ODS steels [1–5], and the data base for structural design has steadily grown. High temperature properties including tensile and creep data are avail-

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able [6]. However, knowledge of low cycle fatigue properties on ODS steels is very limited. The main purpose of the present study is to evaluate the fatigue life and cyclic stress response of 9Cr–ODS and 12Cr–ODS steels at elevated temperatures.

### 2. Experimental procedure

Two types of materials were used for this test: Mm14 heat of 9Cr–ODS martensite and F14 heat of 12Cr–ODS ferrite. Both were manufactured by hot-extrusion of the mechanically alloyed powders at 1423 K, and consolidated bars were normalizedand-tempered for 9Cr–ODS martensite and annealed for 12Cr–ODS ferrite. Basic chemical compositions are 9Cr–0.14C–2W–0.2Ti–0.35Y<sub>2</sub>O<sub>3</sub>– 0.09Ex.O and 12Cr–0.04C–2W–0.25Ti–0.23Y<sub>2</sub>O<sub>3</sub>– 0.05Ex.O, where Ex.O means excess oxygen defined

Table 1 Chemical composition and heat treatment condition of ODS steels

	С	Si	Mn	Р	S	Ni	Cr	W	Ti	$Y_2O_3$	Ex.O*	Ν	Ar
9Cr–ODS martensite (Mm14)	0.14	0.008	< 0.01	< 0.005	< 0.003	0.01	8.88	1.97	0.2	0.35	0.091	0.012	0.005
12Cr-ODS ferrite (F14)	0.044	0.051	0.08	0.005	0.003	0.08	11.45	1.91	0.25	0.229	0.056	0.01	0.004

Heat treatments.

9Cr–ODS martensite (Mm14): 1323 K for 1 h and air cooling => 1073 K for 1 h and air cooling.

12Cr-ODS ferrite (F14): 1523 K for 1 h and air cooling.

Ex.O: The amount of excess oxygen estimated as total oxygen content minus oxygen contained in  $Y_2O_3$  (total oxygen content – yttrium mass%  $\times 0.27$ ).

as the amount of oxygen in the material exceeding that contained in  $Y_2O_3$ . Table 1 shows the detailed chemical composition and heat treatment conditions. Round bar type specimens were machined from the extruded bars with dimension of 5 mm diameter and 10 mm gauge length. The longitudinal direction of the specimen is parallel to the hotextruded direction.

The strain-controlled low cycle fatigue tests were conduced at 873, 923, 973 and 1023 K. Strain control was completely reversed using a triangular wave form. The total strain range was controlled from 0.5% to 1.5% with strain rate of 0.1%/s. The corresponding plastic strain was 0.01% to 1%.

## 3. Results and discussion

Fig. 1 shows the total, plastic and elastic strains as a function of number of fatigue cycle to failure at 973 K for 9Cr-ODS and 12Cr-ODS steels under the condition of total strain range control. Both 9Cr-ODS and 12Cr-ODS steels have similar fatigue behavior. The total strain is composed of the elastic and plastic deformation. The amount of plastic deformation attains about 1% at total strain of 1.5% and 0.06% at total strain of 0.5%, while the elastic strain range remains almost constant at around 0.4%. The plastic strain behavior of 9Cr-ODS and 12Cr–ODS steels during fatigue test at temperature of 873, 923, 973 and 1023 K is shown in Fig. 2. In 9Cr-ODS steel, the plastic strain is relatively larger at the longer fatigue cycles, compared to that of 12Cr-ODS steel. This result suggests that the plastic deformation can be easily initiated in 9Cr–ODS steel. The effect of test temperature is that more plastic strain mode can be induced with increasing temperature in both ODS steels. The plastic strain-fatigue life curves at each test temperature for both ODS steels are represented by the following equations [7], based on the Manson-



Fig. 1. Total, plastic and elastic strains at 973 K under straincontrolled fatigue tests.



Fig. 2. Plastic strain behavior of 9Cr–ODS and 12Cr–ODS steels.

Coffin law, using plastic strain range  $\Delta \varepsilon_{pl}$  (%) and number of cycle to failure  $N_f$ . It is clearly shown that power law exponent is high in 12Cr–ODS steel and decreases with increasing test temperature in both steels.

$\Delta \varepsilon_{\rm pl} = 39.15 N_{\rm f}^{-0.66}$
$\Delta \varepsilon_{\rm pl} = 8.68 N_{\rm f}^{-0.45}$
$\Delta \varepsilon_{\rm pl} = 8.70 N_{\rm f}^{-0.44}$
$\Delta \varepsilon_{\rm pl} = 6.63 N_{\rm f}^{-0.38}$
$\Delta \varepsilon_{\rm pl} = 108.52 N_{\rm f}^{-0.85}$
$\Delta \varepsilon_{\rm pl} = 22.06 N_{\rm f}^{-0.65}$
$\Delta \varepsilon_{\rm pl} = 36.29 N_{\rm f}^{-0.66}$
$\Delta \varepsilon_{\rm pl} = 19.94 N_{\rm f}^{-0.58}$

The stress amplitude,  $2\Delta\sigma$ , vs. total strain range,  $\Delta\varepsilon_t$ , is shown in Fig. 3. The stress inducing the same level of total strain is lower in 9Cr–ODS steel, owing to the stress relaxation by plastic deformation. This behavior is demonstrated in the hysteresis loops at 973 K for 9Cr–ODS and 12Cr–ODS steels shown in Figs. 4 and 5. It reveals that 9Cr–ODS and 12-ODS steels ideally deform in an elastic–plastic manner. The lower yield strength in 9Cr–ODS steel leads to the low level of induced stress at the restricted total strain condition.

Fig. 6 compares the stress amplitude for 9Cr– ODS, 12Cr–ODS and Mod. 9Cr–1Mo steels [8] as a function of number of cycles for strain range control of 0.5% at 923 K. These are plotted until failure. Neither cyclic softening nor hardening is observed in 9Cr–ODS and 12Cr–ODS steels, while Mod. 9Cr–1Mo steel shows an apparent cyclic softening



Fig. 3. The stress amplitude  $2\Delta\sigma$  vs. total strain range for the ODS steels.



Fig. 4. The hysteresis loop of 9Cr-ODS steel at 973 K.



Fig. 5. The hysteresis loop of 12Cr-ODS steel at 973 K.

behavior. The fatigue softening is a typical phenomenon for ferritic-martensitic steels hardened by precipitates and lath structure [9]. It is reported that the stress amplitude decreases markedly in the initial stage, and then approaches the saturated region in F82H [10]. The structure of ODS steels is significantly more stable than the conventional steels during the entire period of fatigue test.

Fig. 7 shows a comparison of the total and plastic strains at 923 K for 9Cr–ODS, 12Cr–ODS and Mod. 9Cr–1Mo steels, where literature data [8] were quoted for Mod. 9Cr–1Mo steel. Based on these data, a schematic diagram is given in Fig. 8. If the total strain range exceeds around 1.0%, the Mod. 9Cr–1Mo steel shows a superior fatigue life to the



Fig. 6. Stress amplitude as a function of number of cycles for 9Cr–ODS and 12Cr–ODS steels and conventional Mod. 9Cr–1Mo steel at 0.5% total strain at 923 K.



Fig. 7. Comparison of total and plastic strains between the ODS steels and Mod. 9Cr–1Mo steel at 923 K under condition of strain-controlled fatigue.

ODS steels. This is attributable to the higher ductility of the Mod. 9Cr–1Mo steel than the ODS steels. The fatigue life of the ODS steels is markedly improved with decreasing total strain range. For a total strain range below around 1.0%, the fatigue life becomes superior to that of the Mod. 9Cr– 1Mo steel. The plastic strain is much smaller in the ODS steels than in the Mod. 9Cr–1Mo steel, because the ODS steels have higher yield strength than Mod. 9Cr–1Mo steel. The suppression of plastic deformation in the ODS steels could result in



Fig. 8. Schematic diagram of fatigue properties at lower and higher number of cycle to failure for ODS and Mod. 9Cr–1Mo steels.

their fatigue life to be markedly improved in low strain condition compared to the Mod. 9Cr-1Mo steel.

#### 4. Summary

The results of this study are summarized as follows:

- (1) The 9Cr–ODS and 12Cr–ODS steels have similar fatigue properties, and they show superior fatigue life under the high cycle conditions compared to conventional ferritic steels such as Mod. 9Cr–1Mo steel. This superiority results from the higher yield strength in the ODS steel than that of Mod. 9Cr–1Mo steel, which leads to lower fraction of plastic strain under strain-controlled fatigue condition.
- (2) No appreciable cyclic softening is observed in the 9Cr–ODS and 12Cr–ODS steels even at 1023 K, while Mod. 9Cr–1Mo steel shows an apparent cyclic softening at 873 and 923 K. This is attributable to the excellent microstructure stability produced by the presence of nano-size oxide particles dispersed in ODS steels.

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