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Microstructural investigation of oxide dispersion strengthened alloy 617 after creep rupture at high temperature

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ABSTRACT

Oxide dispersion strengthened (ODS) alloys have been widely considered as candidate structural materials for advanced nuclear reactors because of their good high temperature properties and irradiation resistance. In this study, the creep properties of oxide dispersion strengthened Alloy 617 (ODS-617) was examined at 950 °C under 25 MPa in the air. Conventional Alloy 617 tested under the same conditions was included for comparison. The ODS-617 exhibited superior creep resistance, including a much longer rupture life of 8533 h and a steady-state creep rate of 8.9×10^{-8} (1/s), which was two orders of magnitude lower than that of the conventional alloy. The lower steady-state creep rate was related to the dispersion of nano-sized Y-Al-O particles, which may impede grain boundary diffusion. It seems that cracks preferentially developed near some large rod-like AlN particles, resulting in the brittle creep fracture mode.

1. Introduction

The key components of the very high temperature reactor (VHTR) system, such as intermediate heat exchanger (HIX) and hot gas ducts (HGD), are expected to serve at 950 °C under 3–8 MPa over a design life of 60 years [1–3]. Candidate materials for these parts should possess good creep property, oxidation resistance and long-term microstructure stability at high temperature, which leaves material selection a major technical challenge.

Alloy 617 is an important structural material for applications at high temperature because of its good creep properties and formability [4,5]. These desirable properties are attributed to the precipitation strengthening of ordered gamma prime phase ($Ni_3(Al,Ti)$) and solid solution strengthening owing to Co and Mo [6]. Also $M_{23}C_6$, which is primarily distributed on grain boundaries, could contribute to improve the creep resistance of alloys by pinning of grain boundaries [7]. However, the gamma prime phase tends to dissolve at temperatures above 900 °C [8–11]. In addition, coarsening of $M_{23}C_6$ is reported during long term aging even at 750 °C [12–14], resulting in performance degradation of Alloy 617. The high temperature mechanical properties of such alloys can be improved by oxide dispersion strengthening (ODS) as the thermally stable oxide particles dispersed in the matrix would pin dislocation motion and also could stabilize microstructure [15], which have been demonstrated in iron-based and nickel-based ODS alloys [16–21]. J. Rösler et al. [22] has reported a concept of dual scale particle strengthening that combines dispersion and reinforcement hardening, and this has been demonstrated to be effective to improve the creep properties of nickel-based ODS alloys further [23,24]. The creep behaviors of nickel-based ODS alloys, including TD alloy [25–28], MA754 [29] and DY alloy [30], have been studied. Compared with the conventional alloys, these nickel-based ODS alloys exhibited different characteristics, such as anomalously high stress exponent values and the existence of threshold stress. It is suggested that interaction between dispersed particles and dislocations may contribute to the unique characteristics of ODS alloys [31–34]. Therefore it is necessary to investigate the evolution of the dispersed particles during creep rupture testing.

In our previous study, the rupture life of Alloy 617 tested at 950 °C under 25 MPa was less than 900 h [35], which could not satisfy the requirements for the HIX and HGD in VHTR. Therefore, the oxide dispersion strengthened Alloy 617 (ODS-617) was developed in our laboratory. In this study, the creep property of ODS-617 was also tested at 950 °C under 25 MPa in the air, and the creep ruptured microstructure was investigated. The potential creep mechanism was also discussed.

2. Experimental

A mixture of Alloy 617 powders with particle sizes of around 40 μm and 0.6 wt% Y_2O_3 powders with particle sizes of around 30 nm were mechanically alloyed (MA) using a horizontal ball mill (ZOZ CM08) for

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Table 1
Chemical composition of the ODS-617 specimen (wt%).

Ni	Cr	Co	Mo	Fe	Al	Ti	Cu	Si	Y	C	O	N
Bal.	22.50	12.80	10.00	1.27	1.19	0.39	0.01	0.11	0.37	0.77	0.69	0.01

40 h at 200 rpm with a ball to powder ratio of 15:1 (in weight) under an argon atmosphere. The MA powders were hot extruded at 1100 °C with a reduction ratio of 6.25:1. The chemical composition of the ODS-617 specimen is listed in Table 1.

Cylindrical creep sample with a gauge diameter and length of 6 mm and 30 mm, respectively, was prepared from the ODS-617 alloy. The creep rupture testing was performed at 950 °C under a stress of 25 MPa in the air, and one creep sample was tested in this study. The specimen elongation was measured using linear variable differential transformer (LVDT), which was directly connected to the specimen gage. The steady-state creep rate ($\dot{\varepsilon}_{SS}$) was obtained by linearly fitting the secondary stage of the strain-time curve.

Physical density of gauge and grip portions of the creep ruptured sample was measured using Mettler Toledo balance (XS205) equipped with density accessories based on the Archimedes' principle. The average density was obtained from three measurements. And the relative density was calculated as the ratio of the measured physical density and the theoretical density. Hardness was measured using a Vickers microhardness tester under 0.2 kgf, and the average value was obtained from eight measurements. Fracture morphology was observed using scanning electron microscope (SEM, JEOL FE6500F) equipped with energy dispersive spectrometry (EDS). Microstructures of both the gauge and grip portions were investigated using electron back scattered diffraction (EBSD) and transmission electron microscope (TEM, JEOL FE2100F). Carbon replica specimens were primarily used for the particle identification and quantitative analyses, and thin specimens prepared by a dual focused ion beam (FIB, Nova 200) were also examined.

3. Results

3.1. Creep Properties

Fig. 1 shows the creep properties of Alloy 617 and ODS-617 tested at 950 °C under 25 MPa. It should be noted that the X axis of creep time was given in the logarithmic form in order to compare the two samples. Fig. 1(a) shows the creep strain-time curves, and the rupture life of Alloy 617 and ODS-617 was 887 h and 8533 h, respectively. As shown by the creep rate curves (Fig. 1(b)), both samples exhibited three creep stages: the primary stage where the creep rate decreased, the steady stage with a nearly constant creep rate and the tertiary stage where the creep rate increased with creep time. The primary stage constituted a very small portion smaller than 0.5% in both samples. The tertiary stage of Alloy 617 took a portion of 38.2%, while it was 11.4% for the ODS-617. The tertiary stage of ODS-617 began more abruptly compared to that of Alloy 617, as shown in Fig. 1(a) and (b). The steady-state creep rates ($\dot{\varepsilon}_{SS}$) of the Alloy 617 and ODS-617 were 6.1×10^{-6} and 8.9×10^{-8} (1/s), respectively. It is noticeable that the rupture strain of the ODS-617 was only half that of Alloy 617. Low elongations to failure have also been reported for other nickel-based ODS alloys [26,29,36], which is related to brittle fracture mode. It can be concluded that the creep property of the ODS-617 was much superior to that of the conventional Alloy 617 based on the rupture life and creep rate.

3.2. Microstructure Observation

The density and microhardness of the creep ruptured ODS-617 sample are summarized in Table 2. Compared with the grip portion, the gauge portion showed a lower density and lower hardness with large variation, indicating that the microstructure of gauge portion may be not uniform. Fig. 2 is the grain boundary distribution maps of the ODS-617 sample after creep rupture. The gauge and grip portions of the crept sample showed similar microstructures, which consisted of fine grains (~500 nm) and a small fraction of coarse grains. The fractions of low angle grain boundaries (LAGBs, < 15°) in gauge and grip portions were 13.4% and 14.2%, respectively. It is apparent that a sub-grain structure did not develop during the high temperature creep testing.

TEM images of the gauge portion of the ODS-617 sample are shown in Fig. 3. Cr-enriched particles (< 1 μm), (Y)-Al-O (30–300 nm), Mo (~500 nm) and Ti-N (~500 nm) were observed according to the EDS mapping results. It should be noted that the regions enriched in Cr and

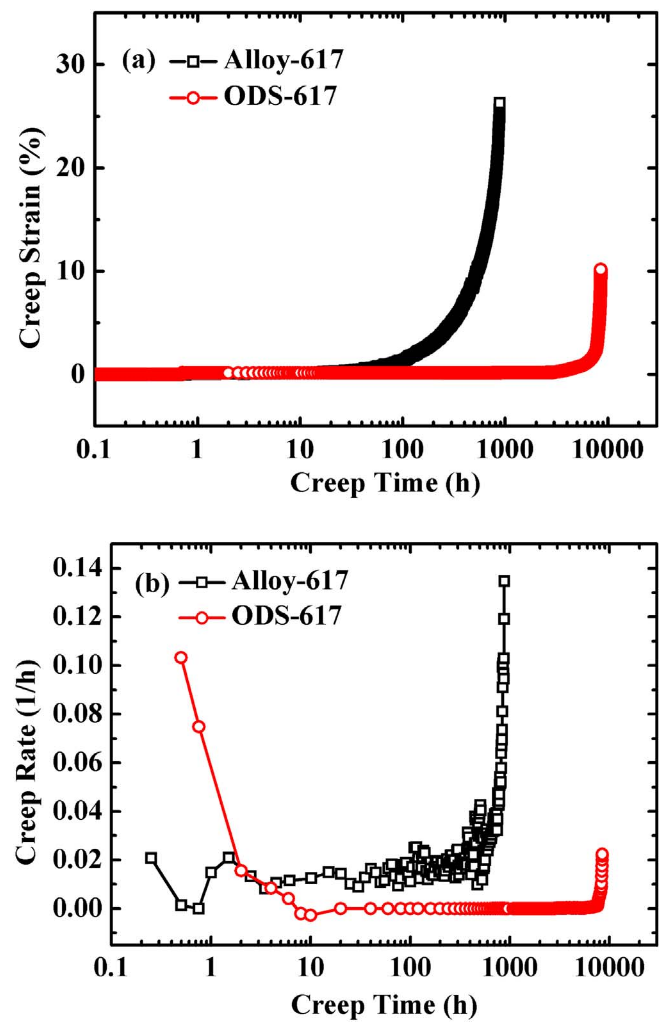

Fig. 1. Creep properties of ODS-617 and conventional Alloy 617 at 950 °C under 25 MPa. (a) Creep strain-time curves; (b) Creep rate-time curves.

Table 2
Density and microhardness of ODS-617 after creep rupture (wt%).

Type	Physical density (g/cm^3)	Relative density	Microhardness
Gauge portion	7.731 ± 0.109	93.1	420 ± 49
Grip portion	8.165 ± 0.049	98.3	511 ± 8