On the Creep Behavior of Dual-Scale Particle Strengthened NickelBasedAlloy

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Abstract

A novel dual-scale particle strengthened Ni-20Cr based alloy was developed with addition of 1.2 wt.% nano-Y₂O₃ and 5 wt.% sub-micron Al₂O₃ via mechanical alloying. The Ni-20Cr-1.2Y₂O₃-5Al₂O₃ alloy exhibited significant reduction in the minimum creep rate (in the order of 10⁻⁹s⁻¹) at 800 °C and 100 MPa.

Keywords: Oxide dispersion strengthened alloy; Creep; Transmission electron microscopy; Three-dimensional atom probe; Nano-indentation

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1. Introduction

Development of high temperature structural materials is essential to fulfill the growing need for advanced coal-power plants, gas turbine inlets, and other high temperature applications. These materials should possess a combination of high creep resistance, stiffness and corrosion resistance in the highly pressurized oxygen- and hydrogen-rich environments. Nickel-based oxide dispersion strengthened (ODS) alloys are considered as promising candidate materials for such applications[1]. Dispersion strengthening has emerged as the most promising mechanism to provide high strength and stability at elevated temperatures[2]. This is in large part because of the attractive dislocation– particle interaction at high temperature via “departure side pinning” phenomenon, first reported by Nardone and Tien[3].

ODS alloys are typically processed via mechanical alloying of elemental or pre-alloyed powders in combination with nano-sized Y_2O_3 powder (or other rare earth oxides such as Ce_2O_3 , La_2O_3) followed by hot extrusion or hot isostatic pressing (HIP) [4]. Homogeneous and uniform distributions of nano-sized and stable Y_2O_3 particles in the matrix of Ni-20Cr are potential obstacles to impede the dislocation motion, which would lead to improved creep resistance and other high temperature mechanical properties[5-9]. The dislocation creep model and threshold stress model based on the concept of attractive dislocation-particle interaction have been studied by several researchers[7,10-14]. High temperature strength of ODS alloys can be further improved by providing an additional composite strengthening mechanism or so-called “load transfer” mechanism to the matrix[15]. In this mechanism, the introduction of reinforcement particles would result in improved high temperature mechanical properties based on the concept of dual-scale particle strengthening. Rosler and Baker[16] have proposed a theoretical concept to obtain a synergetic effect through dual-scale particle strengthening by interaction of small dispersoids (in nanometer size range) and larger reinforcement particles (in micrometer to millimeter range). The addition of submicron Al_2O_3 particles to the Ni-20Cr matrix would generate composite strengthening due to a higher elastic modulus of Al_2O_3 [17,18]. In this work, creep tests were performed on three different alloys systems: (i) Ni-20Cr, (ii) Ni-20Cr-1.2 Y_2O_3 , and (iii) Ni-20Cr-1.2 Y_2O_3 -5 Al_2O_3 (all compositions in wt.%) at 100 MPa and 800°C. Steady state creep stage was monitored and analyzed in three alloy systems.

2. Experimental

Gas atomized Ni-20Cr powder with nominal composition Ni-19.6Cr-0.2Fe-0.8Mn-0.9Si (wt.%) and mean particle size of 23.6 ± 1.1 μm , Y_2O_3 powder with high purity (99.99%) and mean particle size of 30-40 nm, and Al_2O_3 powder with 99.99% purity and mean particle size of 300-400 nm were all acquired from the American Elements Inc. The nominal compositions of Ni-20Cr, Ni-20Cr-1.2 Y_2O_3 and Ni-20Cr-1.2 Y_2O_3 -5 Al_2O_3 alloys were prepared via high energy ball milling using Spex 8000M shaker mixer/mill. Details of ball milling and SPS can be found in an earlier report [19]. The compressive creep experiments were performed at a temperature of 800°C and a stress of 100 MPa using a 2320 model Applied Test Systems lever arm creep tester (Butler, PA, USA). For Transmission electron microscopy (TEM), a FEI Tecnai TF30-FEG STwin STEM operated at an accelerating voltage of 200kV was used. The 3D atom probe tomography (APT) experiments were performed using a LEAP (local electrode atom probe) 3000XTM system from Cameca Instruments Inc. Sample preparation for both TEM and APT

studies were done in a dual-beam focused ion-beam (FIB) instrument(Nova Nanolab 200 system)with Ga ion beam. For APT data analysis IVAS 3.6.2TM software was used.Nano-indentation experiments were carried outat room temperature using a Hysitron Triboindenter (Hysitron, Inc., Minneapolis, MN) with a Berkovich diamond tip. The indentation was performed at a load of 10000 μN with loading rate of 2000 $\mu\text{N/s}$, holding time of 2 s and unloading rate of 2000 $\mu\text{N/s}$.

3. Results and discussion

The analysis of creep strain and creep rate as a function of time at 800°C and 100 MPa for the Ni-20Cr, Ni-20Cr-1.2Y₂O₃ and Ni-20Cr-1.2Y₂O₃-5Al₂O₃alloys are plotted in Fig. 1. The minimum creep rate in order of 10^{-4} , 10^{-8} and 10^{-9}s^{-1} were observed for Ni-20Cr, Ni-20Cr-1.2Y₂O₃and Ni-20Cr-1.2Y₂O₃-5Al₂O₃ alloys, respectively. Based on these experiments, the minimum creep rate of Ni-20Cr was significantly higher than the other alloys. The creep tests were performed for long duration to minimize the primary creep effect. Figure 1(c) illustrates the minimum creep rate in all three alloy systems. The addition of Y₂O₃ in Ni-20Cr matrix improved the creep resistance significantly. Creep resistance wasfurther enhanced by introducing Al₂O₃ particles into Ni-20Cr-1.2Y₂O₃matrix. A low minimum creep rate of $\sim 2.3 \times 10^{-9}\text{s}^{-1}$ was observed for this relatively longer time creep test of Ni-20Cr-1.2Y₂O₃-5Al₂O₃alloy. Further characterization experiments were performed to analyze the exceptional creep strength on this alloy.

Fig. 1.(a) Creep strain versus creep test time plot for Ni-20Cr, Ni-20Cr-1.2Y₂O₃ and Ni-20Cr-1.2Y₂O₃-5Al₂O₃ alloys (b) Creep rate versus creep test time plot for Ni-20Cr, Ni-20Cr-1.2Y₂O₃ and Ni-20Cr-1.2Y₂O₃-5Al₂O₃ alloys showing the enhanced creep strength due to addition of nano-Y₂O₃ and sub-micron Al₂O₃ to Ni-20Cr matrix and (c) minimum creep rate for different Ni-20Cr based ODS alloys.

Nano-indentation experiments were performed on Ni-20Cr-1.2Y₂O₃-5Al₂O₃ alloy in 5×5 matrix points as shown in Fig. 2(a) with 5 μ m separation distance between two consecutive points in X and Y directions. In nano-indentation technique, the penetration depth of the indenter on the surface was measured as the load was applied to the indenter. An illustrative load vs. displacement curve obtained from this measurement is illustrated in Fig. 2(b). The known geometry of indenter allowed us to measure the area of contact from the indentation depth. Figure

2(c) and (d) show the elastic modulus and hardness plots in Ni-20Cr-1.2Y₂O₃-5Al₂O₃ alloy, respectively. In this alloy, the hardness and elastic modulus varied between 4 to 7 GPa and 180 to 230 GPa, respectively. The average elastic modulus and hardness were measured as approximately 200 GPa and 5.5 GPa, respectively. As reported earlier, Ni-20Cr-1.2Y₂O₃-5Al₂O₃ alloy has better mechanical properties than Ni-20Cr[19]. It can be noted from Fig. 2(d), the hardness distribution is not uniform which can be attributed to the heterogeneous microstructure of the material because of multiple phases. Various researchers have reported the influence of heterogeneous microstructure on mechanical properties[20-22]. In this composite, heterogeneity of microstructure occurred due to the distribution of nano-scale Y₂O₃ and micro-scale Al₂O₃ in the matrix. The nano indentation technique is able to map the hardness fluctuations at this length scale due to its nanoscale probe size. The smaller size Y₂O₃ particles are expected to enhance the creep strength through dislocation-particle interaction, whereas larger size Al₂O₃ particles would improve creep strength via load transfer.

Fig.2. Nano-indentation of Ni-20Cr-1.2Y₂O₃-5Al₂O₃ alloy (a) location of the indent points, (b) load-displacement curve, (c) elastic modulus and (d) correlated hardness plot.

TEM bright field (BF) and scanning transmission electron microscopy high angle annular dark field (STEM HAADF) images obtained from Ni-20Cr-1.2Y₂O₃-5Al₂O₃ alloy are shown in Fig. 3(a-b), respectively. Figure 3(a) reveals that ultrafine grains developed in this alloy with clear and distinct boundaries. As evident in TEM micrographs, grains were developed without

any significant grain coarsening. The micrograph shown in Fig. 3(b) exhibits the distribution of dispersoids and reinforcement particles. The average particle diameter and mean particle separation distance of dispersoids were 22 nm and 72 nm, respectively, as reported by Pasebani *et al.*[19](coarser particles enriched in Al were not considered).

Fig. 3. Ni-20Cr-1.2Y₂O₃-5Al₂O₃ alloys: (a) TEM BF micrograph showing nanograins and (b) STEM HAADF micrograph showing the distribution of dual-scale strengthening particles

The atomic distribution of Ni, Cr, Al, Y and O atoms obtained from the LEAP experiments are displayed in Fig. 4(a-e), respectively. The reconstructed maps show that Ni and Cr atoms were distributed uniformly throughout the matrix. Al particles were distributed mainly top and right bottom corner, whereas Y atoms were evident at the center of the map. The O atoms map confirmed the existence of Y₂O₃ at the middle of the map and Al₂O₃ particles at the top and bottom corner of the map. The APT was performed on randomly lifted sample to confirm the existence Y₂O₃ and Al₂O₃ oxide particles. APT analysis revealed the nearly spherical Y₂O₃ particles (Fig. 4(d,e)) with size of 20nm, which is consistent with the TEM results. The shape and size of Y₂O₃ is a key element to the exceptional creep strength. Additionally, much bigger Al₂O₃ particles are also evident (Fig. 4(c,e)).

Fig. 4. APT maps of (a) Ni, (b) Cr, (c) Al, (d) Y and (e) O atoms in Ni-20Cr-1.2Y₂O₃-5Al₂O₃ alloy

The major contribution towards high creep strength is generated from dispersion strengthening. Distribution of nano-size oxide dispersion particles in the Ni-20Cr matrix was expected to act as an effective barrier towards dislocation movement and to enhance creep strength through the pinning effect[5-9]. Mishra *et al.*[14] proposed an empirical relationship based on the concept of dislocation-particle interaction and suggested that the dislocation-particle interaction leads to both threshold stress and changes in the deformation kinetics, expressed as:

$$\dot{\epsilon} = 8.3 \times 10^8 \frac{DGb}{k_B T} \left[\exp \left(-104 \sqrt{\frac{b}{\lambda}} \right) \right] \left(\frac{\sigma - \sigma_0}{E} \right)^5 \quad (1)$$

where $\dot{\epsilon}$ is the steady state creep rate, D the diffusivity, k_B the Boltzmann constant, G the shear modulus of the matrix, b the Burgers vector, λ the inter-particle distance, E the Young's modulus, σ the applied stress and σ_0 the threshold stress. The threshold stress proposed by Mishra *et al.* [10] is based on reduction in dislocation energy by dissociation of lattice dislocations in the matrix-particle interface. This transformation of lattice dislocation to interfacial dislocation can be expressed as:

$$\sigma_0 = 0.002 G \left(\frac{b}{r} \right) \exp \left(20 \frac{r}{\lambda} \right) \quad (2)$$

With the help of TEM micrographs, particle radius r and λ were measured to be 11 nm and 72 nm, respectively. Values of 76 GPa and 0.25 nm were assumed for G and b , respectively [23,24]. Therefore, threshold stress of 73 MPa was calculated using Equation (2).

The other reason for such high creep strength in Ni-20Cr-1.2Y₂O₃-5Al₂O₃ alloy was anticipated to be the load transfer mechanism due to addition of Al₂O₃ particles, with higher modulus of elasticity. Roseler *et al.* [16] proposed that combination of distributed nano-dispersoids and reinforcement particles could offer both dispersion strengthening and composite strengthening in dual-scale particle strengthened (DSPS) alloy. DSPS alloy showed synergistic effect through the interaction of small dispersoids and large reinforcements and resulted in significantly improved creep strength compared to mono-scale particle strengthened alloy. Here the strengthening coefficient due to load transfer can be expressed as [16,25,26]

$$\Lambda \approx 1 + 2 \left(2 + \frac{l}{R} \right) f_r^{\frac{3}{2}} \quad (3)$$

where l is fibers length and diameter and volume fraction of reinforcement particles are $2R$ and f_r , respectively. Assuming spherical reinforcements distribution, Λ can be determined as 1.039 for this Ni-20Cr-1.2Y₂O₃-5Al₂O₃ alloy. The effective stress causing dislocation motion is now termed as $\sigma' = \sigma / \Lambda$ and therefore Equation (1) can be re-written as:

$$\dot{\epsilon} = 8.3 \times 10^8 \frac{DGb}{k_B T} \left[\exp \left(-104 \sqrt{\frac{b}{\lambda}} \right) \right] \left(\frac{\sigma' - \sigma_0}{E} \right)^5 \quad (4)$$

Assuming $D \sim 1.898 \times 10^{-16}$ m²/s in Ni-20Cr matrix and measured value of 200 GPa for E through nano-indentation experiments, the minimum creep rate was predicted to be 8×10^{-9} s⁻¹, which is higher than the experimentally observed value. This difference is most likely due to the assumption of spherical reinforcement distribution in the theoretical minimum creep rate calculation. Another source of discrepancy can be the estimate of threshold stress.

4. Conclusions

Three alloy systems were prepared via ball milling and SPS for steady state creep study, (i) Ni-20Cr base alloy for solid solution strengthening, (ii) Ni-20Cr-1.2Y₂O₃ alloy for dispersion strengthening through nano-Y₂O₃ dispersion and (iii) Ni-20Cr-1.2Y₂O₃-5.0Al₂O₃ alloy for synergistic strengthening of dispersion and load transfer mechanism via introduction of sub-micron Al₂O₃. Among three alloy systems produced in this study, Ni-20Cr-1.2Y₂O₃-5.0Al₂O₃ exhibited the highest creep resistance with minimum creep rate of $\sim 2.3 \times 10^{-9} \text{ s}^{-1}$ under conditions of 100 MPa and 800°C, implying effectiveness of the dual-scale particle strengthening mechanism.

Acknowledgment

The support of the US Department of Energy (DOE) via a grant (DE-FE0008648) managed by the National Energy Technology Laboratory (NETL) is gratefully acknowledged. The authors would also like to thank S. Mridha for helping with nano-indentation experiment and Dr. Sundeep Mukherjee for providing access to nano-indentation equipment located at the University of North Texas.

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