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MICROSTRUCTURAL AND TENSILE PROPERTY DIFFERENCES BETWEEN Ni-16Mo AND ODS ALLOYS FABRICATED BY THE POWDER METALLURGY PROCESSES

Ni-16Mo and ODS alloys were fabricated by the powder metallurgical processes, and their microstructures and tensile properties were investigated. Ni-16Mo-7Cr and Ni-16Mo-7Cr-0.3Ti-0.35Y_2O_3 (in wt.%) alloys were prepared by mechanical alloying, uniaxial hot pressing, and heat treatment processes. Microstructural observations of these alloys revealed that the Ti and Y_2O_3 additions to a Ni-16Mo alloy were significantly effective to refine the grain size and form nano-sized Y-Ti-O oxide particles. Consequently, the tensile strengths at room temperature and 700°C were considerably enhanced. This improvement of tensile properties can be mainly attributed to the formation of nano-sized oxide particles, as well as the refined grain size. It is thus concluded that Ni-16Mo alloy with Ti and Y_2O_3 additions would be very effective in improving the mechanical properties especially at elevated temperatures.

Keywords: Ni-16Mo alloy; oxide dispersion strengthened (ODS); grain refinement; nano-oxide particle; tensile property

1. Introduction

Molten salt reactor (MSR) is one of the most prospective next-generation nuclear systems due to its intrinsic safety, positive cooling, reduced nuclear waste production, and high economic feasibility [1-3]. The first application of the MSR technology was launched as the Aircraft Nuclear Propulsion (ANP) program in the 1950s [4]. The Molten Salt Reactor Experiment (MSRE) program in 1960s subsequently demonstrate the high feasibility of MSR systems as a promising energy generation source [2]. To realize this system, it is necessary to develop the advanced structural material having high mechanical property, corrosion and irradiation resistance at high temperatures [2,5-6]. However, commercial structural materials such as 2¼ low alloy steel, high Cr ferritic/martensitic steel, austenitic stainless steel and even Ni alloys revealed severe corrosivity in fluoride and chloride molten salts. At present, a Ni-16Mo-7Cr superalloy, named as 'Hastelloy N' had been developed by Oak Ridge National Laboratory for a structural material of the MSR. Hastelloy N could be recognized as one of the promising candidates because of moderate strength and corrosion resistance at high temperatures. However, the transmutation reaction between Ni and neutron under MSR operating conditions results in the He generations at grain boundaries and subsequently, formation of He bubble. This eventually leads to a significant decrease of the mechanical properties and dimensional stability like an irradiation swelling [7]. Hence, there has been much effort to develop the new structural materials possessing both He swelling- and molten salt corrosion-resistant alloy systems. The oxide dispersion strengthening (ODS) alloy is the most promising structural material because of excellent creep and irradiation resistance by uniformly distributed nano-oxide particles with a high density, which is extremely stable at high temperature [5,8]. The interface between nano-scaled oxide particle and Ni matrix could be effective inhabitations of He bubble at high temperature. This has inspired the development of new Ni-based superalloy as promising structural materials for MSRs. In this study, to investigate the effect of Ti and Y_2O_3 additions on microstructures and tensile properties, Ni-16Mo and ODS alloys were fabricated by mechanical alloying, uniaxial hot pressing, and heat treatment processes. The grain morphology, precipitate distribution, and tensile properties were characterized.

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2. Experimental

The nominal composition of ODS alloy used in this study was Ni(bal.)-16Mo-7.5Cr-0.35Fe-0.3Ti-0.35 Y_2O_3 in weight ratio. The ODS alloy was fabricated by mechanical alloying, uniaxial hot pressing (UHP) and heat treatment processes. Metallic raw powders and Y_2O_3 powder were mechanically alloyed by a laboratory scaled-planetary ball-mill apparatus (model: FRITSCH P6PL) with high carbon chromium bearing steel balls (ASTM A295 Gr.52100). Mechanical alloying atmosphere is thoroughly controlled in ultra-high purity argon (99.9999%) gas. The MA was performed at a rotation speed of 180 rpm for 20 h with a ball-to-powder weight ratio of 10:1. All powder handling processes for the weighing, collecting, and sieving were conducted in a completely controlled high purity argon atmosphere to prevent the oxygen contamination during the process. To investigate the oxide dispersion strengthening effect, an Ni-16Mo alloy without Ti and Y_2O_3 was also fabricated with the same manufacturing processes. Chemical compositions of Ni-16Mo alloy powder after the MA process were summarized in TABLE 1. The chemical compositions were experimentally analyzed by ICP-AES and inert gas fusion method with the CSON analyzer. The MA powder was then consolidated using UHP method at 1100°C for 2h at a heating rate of 10°C/min. The process was carried out in a high vacuum ($<5\times10^{-4}$ Pa) under a hydrostatic pressure of 75 MPa in uniaxial compressive loading mode. After the consolidation process, the hydrostatic pressure was relieved and the ODS alloys were cooled in the furnace. After the UHP process, heat treatments at 1180°C for 60 min were subsequently carried out. To compare the microstructures and tensile properties, a Ni-16Mo alloy without Ti and Y_2O_3 additions was also fabricated with the same fabrication processes. Alloy samples were mechanically wet ground and electrolytically polished in a 5% $HClO_4$ + 95% methanol solution in volume % at 18 V with 0.5 mA. Grain morphologies were observed by a field emission scanning electron microscopy (FE-SEM, model: JEOL JXA-8530F) and the grain size was determined by the linear intercept methods using FE-SEM micrographs with back-scattered electron imaging mode. To observe nano-scaled oxide particles, a field emission transmission electron microscopy (FE-TEM, model: JEOL JEM-F200) was used. Thin foil specimens for the FE-TEM were fabricated by a focused ion beam (FIB) method. Mean particles size and number density were evaluated using a commercial image analysis software. To evaluate the tensile properties, miniaturized tensile test were carried out using sheet-type tensile specimens whose gauge length was 5 mm, width was 1.2 mm, and thickness was 0.5 mm. Tensile tests were carried out at room temperature and 700°C in air at a strain rate of 6.7×10^{-3} s^{-1}.

TABLE 2

Chemical composition of Ni-16Mo and ODS alloys (in wt.%)

Materials	Ni	C	Cr	Mo	Ti	Fe	Y_2O_3	Ex.O
Ni-16Mo alloy	bal.	0.02	7.27	16.2	—	3.65	—	—
ODS alloy	bal.	0.04	7.36	15.8	0.27	3.75	0.28	0.11

3. Results and discussion

3.1. Microstructures of Ni-16Mo and ODS alloys

The grain morphologies on the ODS alloy and Ni-16Mo alloy without Ti and Y_2O_3 additions are shown in Fig. 1. The effect of Ti and Y_2O_3 additions in the Ni-16Mo alloy gave a dramatic evolution in the microstructures. Homogeneous grain distributions were observed and microstructure of the Ni-16Mo alloy showed a typical austenite grains in 4.5 μm with twin structures. In contrast to this, ODS alloy with Ti, Y_2O_3 additions had quite inhomogeneous grain distribution, which consisted of the co-existed microstructure with fine (<1 μm) and coarse (1 ~ 3 μm) grains. It is estimated that addition of Ti, Y_2O_3 in Ni-16Mo alloy leads to the significant decrease of the grain size. Fig. 2 shows the analysis results of oxide particle morphology in the ODS alloy. It was observed that extremely fine oxide particles were homogeneously distributed on the Ni-16Mo alloy matrix and grain boundaries, as shown in Fig. 2(a). A TEM-EDS analysis revealed that fine oxide particles were mainly consisted of Y, Ti and O, as shown in Fig. 2(b). Some researchers reported that the Y-Ti-O complex oxides are formed by a combination of Y_2O_3, Ti and residual O in Ti added ODS alloys. These were

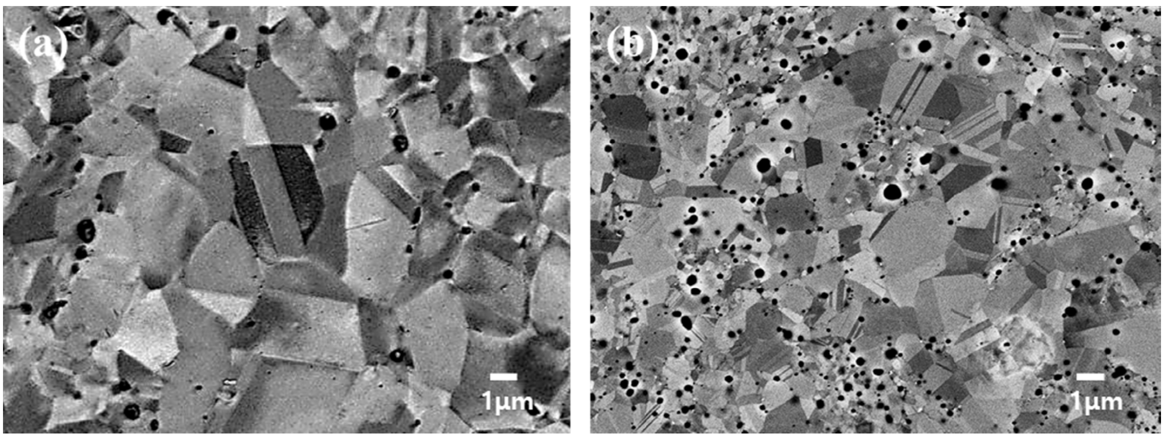

Fig. 1. Grain morphologies of (a) Ni-16Mo alloy, (b) ODS alloy

complex oxides such as $Y_2Ti_2O_7$, Y_2TiO_5 and they were formed in the beginning stage of the hot consolidation process at above 900°C in the ODS alloys [9]. During a hot consolidation process, grain growth and grain boundary migration were suppressed by uniformly distributed the nano-oxide particles which resulted dramatic grain refinement. Based on TEM observations in this study, mean particle size and number density of the nano-oxide particles in the ODS alloy were estimated to 12.5 nm and 6.0×10^{21} m^{-3}, respectively.

(a)

(b)

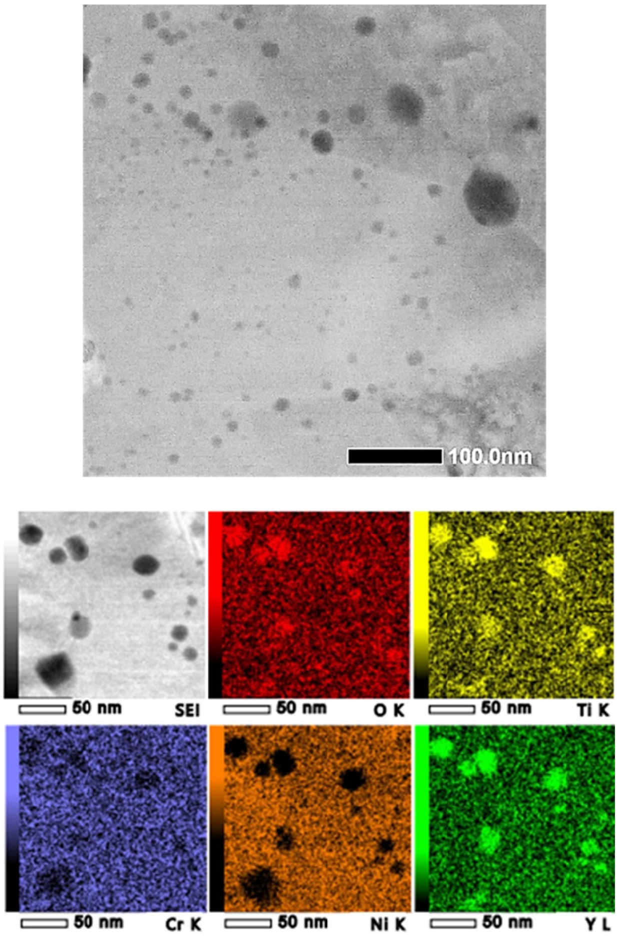

Fig. 2. (a) TEM bright field image and (b) TEM-EDS analysis of the oxide particles in ODS alloy

3.2. Tensile properties of Ni-16Mo and ODS alloys

Miniaturized tensile tests were carried out to evaluate the mechanical properties of Ni-16Mo and ODS alloys at room and elevated temperatures. Typical stress-strain curves for two alloys were presented in Fig. 3. By an addition of Ti and Y_2O_3 in Ni-16Mo alloy, tensile strengths of ODS alloys were extraordinarily higher than Ni-16Mo alloy in both room and elevated temperatures. Ultimate tensile strengths (UTS) of ODS alloy were 1669, 424 MPa with a sufficient total elongation (TE) of 8.6, 14.2% at 25, 700°C, respectively. Furthermore, Ni-16Mo alloy exhibited relatively insufficient UTS and TE at an elevated temperature, which corresponded to less than 65% for UTS and 35% for TE of the ODS alloy. Total elongations in tensile test were significantly different between Ni-16Mo and ODS alloys. It could be observed that fine nm-scaled oxide particles were uniformly distributed with a high number density of 6.0×10^{21} m^{-3} in Ni-16Mo ODS alloys as shown in Fig. 2. The presence of oxide particles is detrimental to the ability of plastic deformation at a room temperature. Low plasticity was attributed to the dispersion of oxide particles and extremely fine grains presented in Fig. 1 and 2. A Ni-16Mo alloy showed significantly poor tensile strength and TE at 700°C, whereas the ODS alloy had a favorable tensile strength and elongation due to an interaction between nano-oxide particle and dislocation. This is attributed to the effects of dispersion strengthening by uniformly distributed nano-oxide particles in the grain and on the grain boundaries at high temperatures. This result coincided with microstructural observation and analysis in 3.1. Extremely fine grains of the ODS alloy contributed to an improvement in the tensile strength by the grain boundary strengthening at a room temperature. Moreover, the nano-oxide particle is extremely stable at a high temperature of up to 1300°C. These especially plays an important role as 'Orowan strengthening', which is a strong obstacle for the dislocation gliding and grain boundary sliding when the material deforms at elevated temperatures [10,11]. In contrast with ODS alloy, conventional Ni-16Mo alloy had solid strengthening elements such as Mo, Cr, Fe. However, they were ineffective to enhance the tensile strength and elongation at 700°C.

(a)

(b)

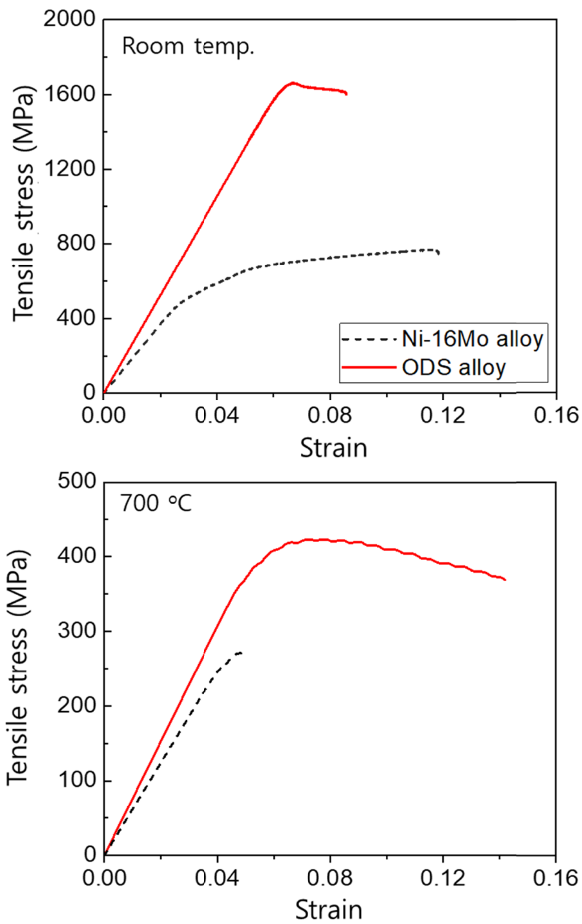

Fig. 3. Stress-strain curves for Ni-16Mo and ODS alloys at (a) room and (b) elevated temperatures

4. Conclusions

The Ni-16Mo and ODS alloys were instantly fabricated by the MA and UHP processes, and evaluated the differences between two alloys on the microstructures and tensile properties. The microstructure of Ni-16Mo alloy showed a typical coarsened austenite structure without any precipitates. Due to the addition of Ti and Y_2O_3, grain refinement significantly occurred in the Ni-16Mo ODS alloy owing to a uniform distribution of nano-sized complex oxide particles. This led to the tensile strength and total elongation of the ODS alloy being more superior than those of the conventional Ni-16Mo alloy at room and elevated temperatures. It is considered that nano-oxide particles in ODS alloy play important roles for the suppression of the grain growth during the hot consolidation process, and strong obstacle for the dislocation gliding or grain boundary sliding when the material deforms at elevated temperatures.

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