Post-extrusion heat treatment effects on microstructure and mechanical properties of 9Cr nanostructured ferritic alloy

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1. Introduction

Nanostructured ferritic alloys (NFAs) show enhanced high temperature strength by adding nanoscale oxide particles into ferritic-martensitic (FM) steel [1]. High Cr FM steels are under intense research as candidate materials for the components of nextgeneration nuclear systems [2-4]. The adequate mechanical and fractural properties are prerequisites for core materials that are subjected to a rigorous environment at a high temperature of up to 650°C and neutron irradiation of 200~400 dpa [5]. A few recent researches have reported that the fracture toughness of NFAs is very low at above 300°C [5, 6]. To overcome this drawback of NFAs, post extrusion heat treatments that can evolve the partial phase transformations in a 9Cr-NFA were applied in this study.

The objectives of this study are to improve the fracture toughness and ductility of NFA through a simple post-extrusion thermo-mechanical process, which can lead to changes in the microstructure of nanostructured 9Cr alloy into a dual phase structure.

2. Experimental

The pre-alloyed Fe-9Cr base metallic powder and 0.3 wt.% Y2O³ oxide particles were mixed and mechanically alloyed by ball milling. The mixed powder was sealed in 3 inch diameter mild steel cans, degassed, and extruded. The chemical composition of the as-extruded base material (9YWTV-PM1 in this study) is listed in Table 1.

Table 1. Chemical compositions of NFAs (wt.%).

Material C Cr W Ti V Fe			
9YWTV-PM1 $\frac{0.15}{5}$ 8.92 2.19 0.36 0.21 bal.			

The tensile and fracture toughness tests were conducted for 9YWTV-PM1 up to 700°C using MTS 810 servo-hydraulic test machine in conjunction with high vacuum furnace. The microstructural examinations were carried out on 9YWTV-PM1 using field emission-transmission electron microscopy (FE-TEM), electron backscatter diffraction (EBSD) and Xray diffraction (XRD). Differential scanning calorimetry (DSC) was used for monitoring phase transformation during heating and cooling samples.

The DSC samples were heated and cooled at the rate of 5 K/min.

3. Results and Discussion

Onset temperature of $\alpha \rightarrow \gamma$ transformation in 9YWTV-PM1 was found to be 950°C as the results of in-situ high temperature XRD and DSC analyses as shown in Fig. 1.

Fig. 1. DSC spectrum of as-extruded Fe-9Cr base NFA (9YWTV-PM1) heated and cooled at 5 K/min. The numbers above or below the features indicate the peak temperatures.

Phase fractions of γ as a function of temperature and time were calculated from XRD spectrums using peak area integration method as plotted in Fig. 2. Volume fractions of γ phase in samples those were exposed to various heat treatment temperatures were saturated after 50 min.

Fig. 2. Phase fraction of FCC (γ) in Fe-9Cr base NFA (9YWTV-PM1) samples exposed at various heat treatment temperatures as a function of time.

The ultimate tensile strength of the base material at room temperature is 1.77 GPa. The alloy retained relatively high strength up to 700°C, though there was a sudden drop in strength above 500°C as shown in Fig. 3.

The uniform elongations are more or less than $\sim 1\%$ over the test temperature range.

The effects of partial phase transformation heat treatment on as-extruded 9YWTV-PM1 were significant over a testing temperature range. Strength of 9YWTV-PM1 increased after heat treatment except data obtained from testing at 700° C as shown in Fig 3.

Fig. 3. Stress-strain curves for 9YWTV-PM1 obtained from tests at various temperatures.

Uniform elongation increased after the heat treatment also. The data obtained from tension tests for as-extruded and heat treated alloys are listed in Table 2.

Table 2. Tension testing results for 9YWTV-PM1 obtained at at various temperatures.

	Y.S.	U.T.S.	U.E.				
Temp.	(MPa)	(MPa)	$\%$				
R T	1705/1675	1766/1842	1.2/1.9				
300° C	1386/1492	1521/1581	3.4/3.6				
500° C	1182/1259	1301/1428	6.0/6.2				
700° C	396/302	424/359	9.7/16.1				

Fig. 4. Fracture toughness, KJQ, of 9YWTV-PM1 measured at various temperatures.

Fracture toughness, K_{JO} , were measured as-extruded and heat treated 9YWTV-PM1 through 3-point bending testing. K_{JQ} were calculated from J_Q values measured at each testing condition.

Fracture toughness of the 9YWTV-PM1 has increased by more than 40 % at every testing temperature after heat treatment as shown in Fig. 4.

In conclusion, it is found that the mechanical property and fracture toughness of 9Cr-NFA has been improved through the heat treatment that leads partial phase transformation in nano grains.

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