§2. Fabrication and Characterization of Reference 9Cr and 12Cr-ODS Low Activation Ferritic/martensitic Steels

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One of the key parameters of ODS steels is the level of Cr, being generally categorized into those with ~9%Cr tempered martensitic matrix phase and 12-16%Cr ferritic matrix phase. High Cr ODS steels are potential for higher temperature operation and enhanced corrosion resistance but have drawbacks such as anisotropic properties originated from extrusion and forging direction and as radiation-induced undesirable phases such as alpha prime. The NIFS effort launched in 2008 for fabrication of ODS steels with 9%Cr (9Cr-ODS) followed by those with 12% Cr (12Cr-ODS) in 2013 by similar fabrication processes. Basic characterization of 9Cr-ODS has been carried out on tensile and creep properties [1], and compatibility with liquid breeders [2]. This paper reports initial efforts of comparing the two alloys.

The manufacturing process of the ODS steel plates is shown in Fig. 1. The argon gas atomized alloy particles with a size of less than 150 µm were mechanically alloyed with Y_2O_3 powders of 20 nm in mean diameter. The 10 kg mass of powders was ball-milled for 48 h in an argon gas atmosphere at a rotational speed of 220 rpm. The mechanically mixed powder was then canned and degassed at 673 K for 3 h. Hot-extrusion was carried out to the ratio of 6.4 at 1423 K into three bars of ~30 mm in diameter, followed by hot forging at 1473K. Final heat treatments were carried out for 9Cr-ODS at 1323 K for 1 h (Air cooling) followed by 1073 K for 1 h (Air cooling), and for 12Cr-ODS at 1473 K for 1 h. After final shaping, 6 plates each of ~40 mm × ~200 mm × 5~6 mm were produced.

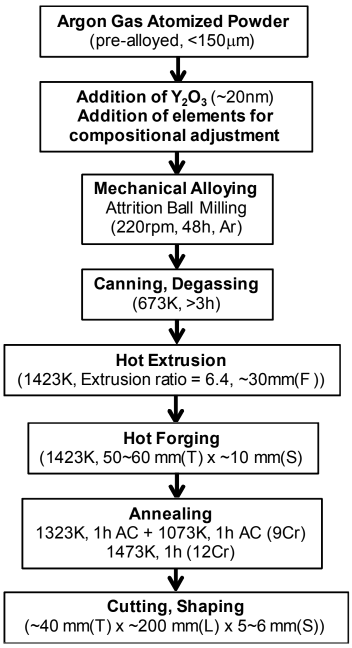

Fig. 1. Manufacturing process of 9Cr and 12Cr-ODS steel plates.

The compositions of the alloys were analyzed by combustion infrared absorptiometry for C and S, absorption spectrophotometry for Si and P, inductively coupled plasma atomic emission spectroscopy for Mn, Ni, Cr, W, Ti and Y, and inert gas fusion method for O, N and Ar, and are shown in Table 1 with estimated Y_2O_3 and excess O levels assuming all Y form Y_2O_3.

Fig. 2 shows microstructure of the alloys by OM and SEM. There is a remarkable difference in grain size, grain orientation and carbide precipitates. 9Cr-ODS has smaller and homogeneous grains and higher density of carbide precipitates than 12Cr-ODS. In 12Cr-ODS the grains are coarsened and elongated towards the longitudinal direction.

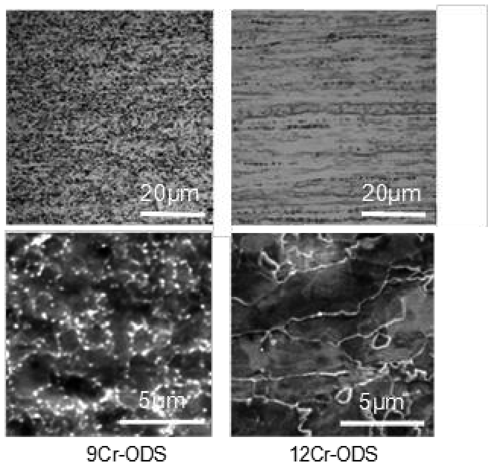

Fig. 2. OM and SEM microstructures of the ODS steels.

Fig. 3 is low and high magnification TEM images of the alloys. High density of nano-particles was observed in the both alloys. 9Cr-ODS has a tempered martensitic phase with carbides and dislocations. In contrast, 12Cr-ODS has coarse structure with weak internal strain, low carbide density and medium density of residual dislocations in the matrix. These results suggest that 12Cr-ODS was not recrystallized during the thermal and mechanical treatments.

Mechanical property tests including thermal creep deformation are in progress.

[1] Y. Li et al., *Fusion Eng. and Des.* **86,** 2495 (2011).
[2] Y. Li et al., *J. Nucl. Mat.* **443,** 200 (2013)

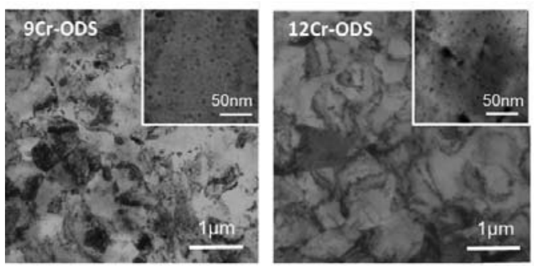

Fig. 3. TEM microstructures of the ODS steels.

Table 1. Chemical composition of the alloys produced.

Alloy	Composition (wt%)												Estimation	
	C	Si	Mn	P	S	Ni	Cr	W	Ti	Y	O	N	Ar	Y_2O_3	Ex.O
9Cr-ODS	0.14	0.06	0.09	<0.005	0.004	0.03	9.08	1.97	0.23	0.29	0.16	0.013	0.005	0.37	0.082
12Cr-ODS	0.035	0.03	0.02	<0.005	0.003	0.04	11.65	1.90	0.29	0.18	0.083	0.005	0.005	0.23	0.03