THE RECENT DEVELOPMENT OF FABRICATION OF ODS FERRITIC STEELS FOR SUPERCRITICAL WATER-COOLED REACTORS CORE APPLICATION

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Abstract

Development of cladding materials which can work at high temperature is crucial to realize highly efficient and high-burnup operation of Generation IV nuclear energy systems. Oxide dispersion strengthened (ODS) ferritic steel is one of the most promising cladding materials for advanced nuclear reactors, such as superciritical water-cooled reactor. ODS ferritic steels with Cr content of 12, 14 and 18% were designed and fabricated in China through the mechanical alloying (MA) route. The process parameters were discussed and optimized. Mechanical properties were measured at room temperature and high temperature.

1. Introduction

Oxide Dispersion Strengthened (ODS) ferritic steels are an emerging class of nanostructured alloys, which contain Y-Ti-O enriched dispersion strengthening nano particles (clusters) with very high number density. These Y-Ti-O nano particles are stable at high temperature (even close to the melting point of the steel) or under irradiation up to 800^{o} [1-3]. Due to these nano structural features, ODS ferritic steels show excellent high temperature mechanical properties and neutron irradiation resistance ability. ODS ferritic steels have been considered as one promising high temperature structural material used in advanced nuclear reactors, such as fusion reactor and fast breeder reactor. The temperature window of ODS ferritic steels is expected to be higher than 600^{o} (compared to $350\text{-}550^{o}$ for application of reduced activation ferritic/martensitic steel) [4-6]. Recent studies indicate that the ODS ferritic steels also show excellent corrosion-resistance properties, and thus are promising for in-core application in supercritical water-cooled reactors (SCWR) [7,8].

The general fabrication method for ODS ferritic steels is a mechanical alloying (MA) method: first, using high-energy milling to deform mixtures of elemental or alloyed metal powders, then hot compacted or extruded, finally, hot-rolled or forged and heat treated to produce a fully dense material in the required shape. The service properties of ODS ferritic steels are decided by its chemical composition design and processing history. In this work, the effects of MA parameters on the microstructural properties and element distribution of the as-milled powders for ODS ferritic steels were investigated. The microstructures and the mechanical properties of the HIPed ODS ferritic steels were observed and tested.

2. Materials and experiment

The nominal compositions of the ODS ferritic steels are listed in Table 1. The prealloyed powders with an average particle size of 45 μm were mechanical alloyed with Y_2O_3 powders (the average powder size is 30 nm) in a high-energy planetary ball mill under a pure argon atmosphere. The ball-to-powder weight ratio was 10:1. The rotation speed was between 260 rpm to 500 rpm, and the milling time was between 10 h to 90 h.

Table 1 The nominal compositions of the ODS steels

Samples name	Composition, %
12 Cr-ODS	Fe-12Cr-2W-0.2Si-0.5Ti-0.35 Y_2O_3
14 Cr-ODS	Fe-14Cr-2W-0.2Si-0.5Ti-0.35 Y_2O_3
18 Cr-ODS	Fe-18Cr-2W-4Al-0.2Si-0.5Ti-0.35 Y_2O_3

The MA ferritic steel powders were degassed and sealed in a stainless steel container under a vacuum of 10^{-2} Pa at 450^{o}. The canister was then consolidated by hot isostatic pressing (HIP) at 1150^{o} for 3 h under a pressure of 200 MPa (for 12 Cr and 18 Cr ODS) or 70 MPa (for 14 Cr ODS). After HIPing, the 70 MPa HIPed materials were forged at 850^{o} -1150^{o} with 70% deformation and then air cooled to room temperature. The as-HIPed and as-forged samples were annealed at 1100^{o} and 1300^{o} for 2 h in vacuum quartz tubes.

The crystal structure of the milled powder was examined using an X-ray diffractometer (XRD; D/MAX—RB). The particle size distribution was measured by means of a laser diffraction scattering method using a particle size analyzer (LMS-30). SEM and TEM equipped with EDS were used for microstructure observation. The SEM samples were mirror polished using standard metallographic methods and etched with 4% nital. The TEM samples were thinned in a twin-jet electro-polisher with 10% $HClO_4$ in glacial acetic acid at temperature of 10℃ and potential of 40V.

The relative density of the specimens after HIPping was measured by means of the Archimedes method. The tensile strength test was conducted on the samples with a gauge dimension of 25 mm in length and 5 mm in diameter at room temperature (RT) and temperature up to 700^{o}. Annealing testing was conducted on the as-HIPed and forged steels. The samples were sealed in a vacuum quartz tube and then annealed in a muffle furnace at 1100℃ and 1300℃ for 5 h, respectively.

3. Results and discussion

3.1 The effect of MA parameters

Rotational speed and milling time are the most important MA parameters. According to the XRD and SEM results, the optimal rotational speed and milling time for ferritic steels are 380 rpm for 30 h. Fig. 1 shows the powder morphologies and cross-section morphology of the as received MA powders. The powder morphology of the MA powders show that the mixed powder was more closely spherical in shape with a more uniform average size (Fig.1 a, c, e). The average particle size is 13.4 μm for 12 Cr-ODS ferritic steel after 30 h milling. The cross-section morphology show that the typical MA lamellar structures are destroyed and the composition distribution is very homogeneous (Fig.1 b,d, f).

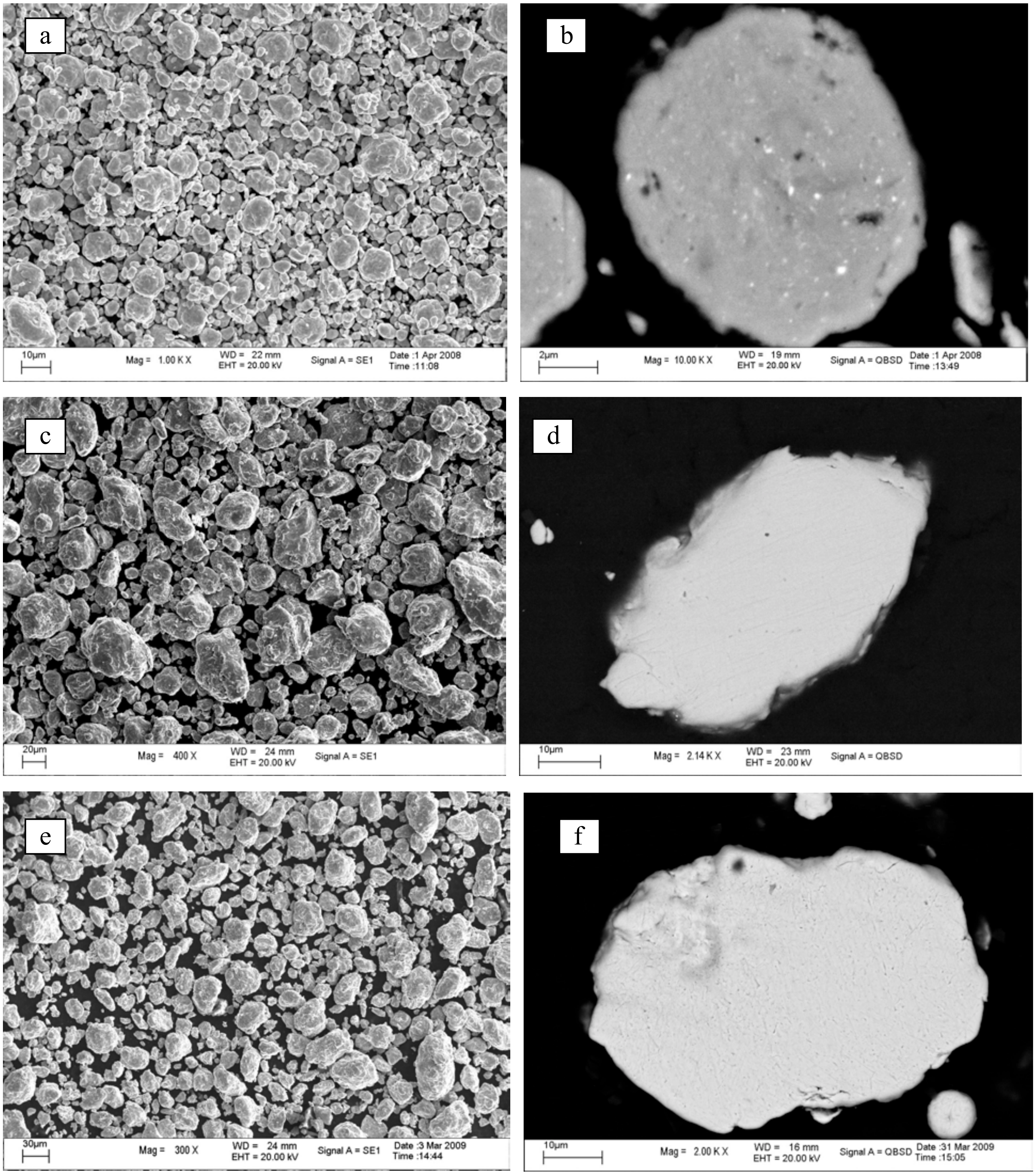

Figure 1 SEM images of the as received MA powders: (a), (b) 12 Cr-ODS; (c), (d) 14 Cr-ODS; (e), (f) 18 Cr-ODS

3.2 Microstructure observation

The relative densities of the as-HIPed ODS ferritic steels are higher than 98.5%. Fig. 2 shows the SEM and TEM morphology of the as-HIPed materials and samples after annealing at 1300 ^{o} for 2h. The grain size of the as-HIPed materials is very fine, around 2-5 μm. A high number density of precipitates with a size less than 20 nm could be observed in TEM image for the 12 Cr-ODS. EDS analysis indicated that these very fine precipitates were Y-, Ti- oxide particles. These nano complex oxide particles were very stable after annealing at 1100℃ for 2 h. This is the reason why ODS alloys have good mechanical properties at high temperature. A high number density of nano-sized particles exerts a pinning force on grain boundaries to retard boundary migration. When the annealing temperature increased to 1300℃, Cr-enriched segregation phases with island shapes with sizes of several microns, were formed for 12 Cr-ODS.

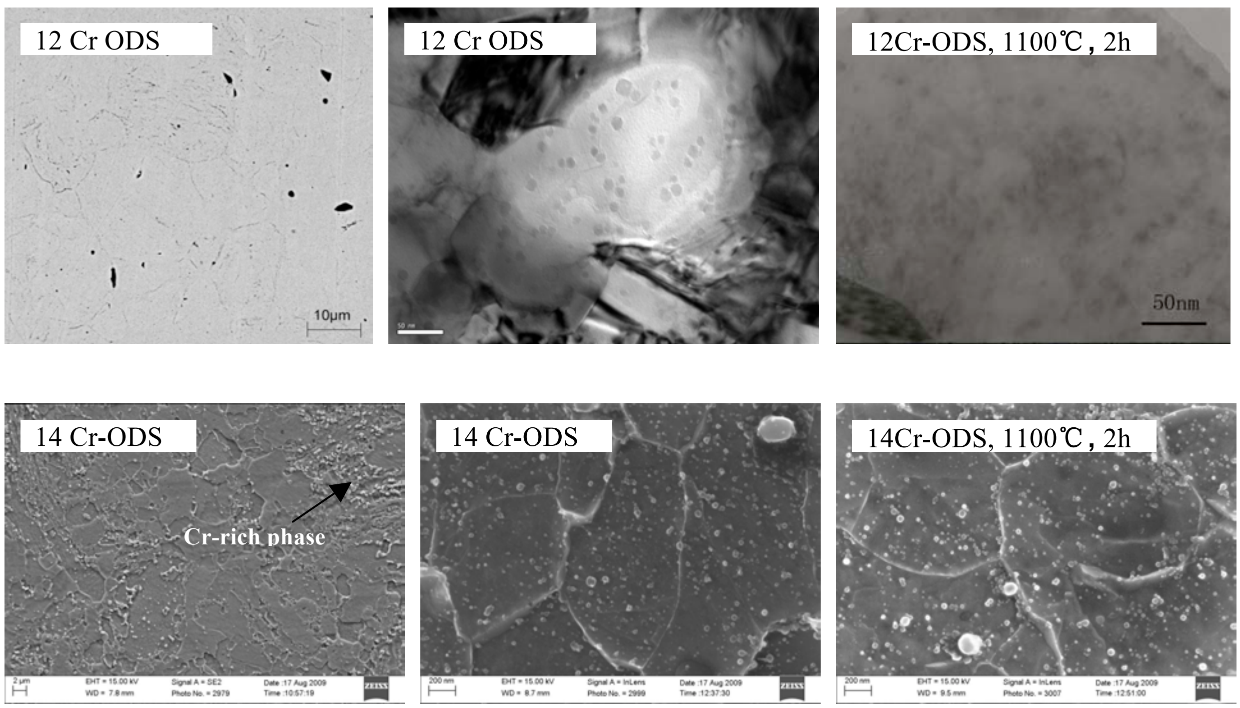

Figure 2 SEM and TEM micrographs of as-HIPed and annealed materials

3.3 Mechanical properties

The tensile strength was measured at RT and temperature of 400, 600 and 700℃ for the as-HIPed ODS steels. Table 2 shows the results of ultimate tensile strength (UTS), critical tensile strength and total elongation. It should be noted that a very high UTS is achieved at RT for the as-HIPed 12 Cr-ODS (without additional deformation work). However, the fabricated steel shows a very low total elongation value at RT and 700℃. It is also obvious that the higher the Cr content of the ODS steels, the lower the tensile strength will be. However, the total elongation showed the opposite effect. The high Cr content (14 and 18 %) ODS steels showed very high total elongation, especially at high test temperature. After aging the 18 Cr-ODS steel at 500℃ for 100h, the tensile strength increased with a small amount at room temperature, but the total elongation decreased.

Fig. 3 shows the fracture surface morphology of the three as-HIPed ODS steels after tensile strength tests at RT. Typical quasi-cleavage fracture and shallow dimples on the surface can be observed for 12 Cr-ODS steel, while typical dimple fracture was observed for 14 Cr-ODS and 18 Cr-ODS steels.

Table 2 Ultimate tensile strength σ_u, critical tensile strength $\sigma_{0.2}$, uniform elongation ε_u and elongation at failure ε_f of the ODS ferritic steels

materials	T / ^{o}	σ_u / MPa	$\sigma_{0.2}$ / MPa	ε_u / %	ε_f / %
12 Cr-ODS	25	1210	1170	0.5	1.5
	700	325	290	7.0	12.5
14 Cr-ODS	25	940	900	10	41
	700	290	205	19.5	36.5
18 Cr-ODS	25	895	660	15	35
	700	265	225	44.5	68
14 Cr-ODS after forging	25	1260	-	-	-
	700	285	215	24.5	45
18 Cr-ODS after aging	25	1030	815	7.5	4.5

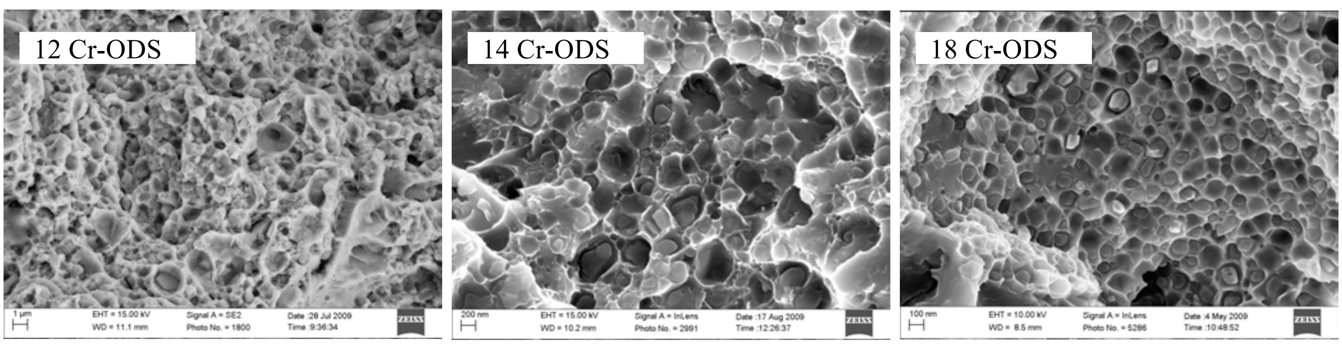

Figure 3 SEM fracture surface morphology after tensile strength testing at RT

4. Conclusions

The morphological and microstructural properties of powder particles were systematically examined to optimize the powder processing of high chromium oxide dispersion strengthened (ODS) ferritic steels. A good milling efficiency could be obtained at a milling speed of 380 rpm; after 30 h milling time, the particle shape became more closely spherical with more uniform size. The average particle size is 13.4 µm for 12 Cr-ODS ferritic steel.

The relative density reached 98.5% for ODS ferritic steels after HIPing at 1150 ^{o} for 3h under a pressure of 200 MPa. The oxide dispersion particle with sizes less than 20 nm were found to be very stable, even after annealing at 1100 ^{o} for 2h. The tensile strength of the fabricated 12 Cr-ODS ferritic steel is very high, but the ductility still need improvement. The higher the Cr content in the ODS steels, the lower the tensile strength will be, but the higher the total elongation will be.

5. Acknowledgements

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