Fabrication and mechanical properties of a 14Cr-ODS steel

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Abstract: In this paper, the fabrication method and the mechanical properties of a 14Cr-ODS steel are investigated. The pre-alloyed steel powders and oxide powders were mechanical alloyed after milling for 30 h under a rotation of 300 rpm. Y-Ti-O phase was obtained after annealing the MA powders at 1100 °C. The tensile strength of the HIPed 14Cr-ODS steels at room temperature was about 1120 MPa. Microstructure observation showed that there were amount of nano dispersion particles in the base alloy which resulted in the high hardness and tensile strength.

1. Introduction

Based on safety and efficiency, the Generation IV forum has selected six innovative concepts for nuclear reactors. SCWR (Supercritical Water Reactor), which offered many advantages including the use of a single phase coolant with high enthalpy, the elimination of components and a much higher efficiency, was considered as a promising design and frequently investigated by researchers [1-3].

The design for the SCWR called for advanced structural materials. The main candidate structural materials for the SCWR are Ni-based alloys, ODS steels, ferritic/martensitic and austenitic steels. Ferritic steel is well known to have excellent irradiation resistance due to its bcc structure [4-5]. However, its high temperature strength needs improvements. Nano oxide particle was added into the ferritic matrix to improve the high temperature strength and irradiation resistance properties, and ODS steels was developed [6].

The ODS steels is usually fabricated in a method of mechanical alloying due to its low cost and facile realization. Meanwhile, consolidation of MA powders by hot isostatic pressing (hipping) can avoid the anisotropy of the microstructure, compared with hot extrude. In this paper a 14Cr-ODS steels was fabricated, and the properties and the microstructure were investigated.

2. Experimental

Pre-alloyed 14Cr ferritic steel powders, Ti and Y₂O₃ were used as starting materials. The composition of the mixed powders was designed as Fe-14Cr-1W-0.2Si-0.5Ti-0.35Y₂O₃, as shown in table 1.

| Table 1 Chemical composition of powders, in weight percent |
|-----------------|---|---|---|---|---|---|
| Materials       | C  | Si | Cr | W  | Ti | Y₂O₃ | Fe  |
| 14CrNA          | 0.06 | 0.24 | 14.20 | 1.80 | 0.09 | —    | Bal.|
| 14CrODS         | 0.06 | 0.24 | 14.20 | 1.80 | 0.50 | 0.35 | Bal.|
The starting materials were mechanical alloyed in a nitrogen atmosphere using a ball mill at 300 rpm for 30 h. The ball-to-powder mass ratio was 5:1. The mechanical alloyed powders were then consolidated by hot isostatic pressing at a temperature of 1150 °C under a pressure of 120 MPa for 3 h. The powders before and after milling were investigated using a scanning electron microscope (SEM, LEO-1450) and X-ray diffraction (XRD, D/MAX-RB). Microstructure and chemical composition were characterized by Transmission Electron Microscope (TEM, JEM-2010) with an energy dispersive spectroscopy (EDS). Samples for TEM were prepared by mechanical polishing and electrochemical thinning using 7% perchloric acid + 93% ethanol as electrolyte. Tensile tests were carried out at room temperature and 700 °C with a strain rate of $5 \times 10^{-2} \text{ s}^{-1}$.

3. Results and discussion

3.1 Precipitated phase

The contents of oxygen induced in the process of mechanical alloying played an important role in the formation of nano size particles. The size of the nano particle increased as the introduction of the excess oxygen. The nano particle related with the excess oxygen played a negative role in the densification and mechanical properties of the samples. The oxygen content of the pre-alloyed powders was about 0.069%. The oxygen content of the ODS powders was about 0.21%. The amount of oxygen induce in the ball milling process was about 0.067%.

Fig. 1 shows the XRD patterns of the pre-alloyed powders, mixed powders before and after mechanical alloying, as well as ODS powders after annealing. It is concluded from the figure that the main phase for the pre-alloyed powders is iron. There are Ti and $Y_2O_3$ peaks in addition to iron peak for the mixed powders. However, the Ti and $Y_2O_3$ peaks disappeared after ball milling for 30 h. Thus, ODS powders can be fabricated using mechanical alloying.

![XRD patterns](image)

Fig. 1 XRD patterns for the powders

It can be concluded from patterns of the annealed samples that Y-Ti-O phase formed after high temperature annealing. This process can be illustrated as follows: $Y_2O_3$ which was added into the pre-alloyed powders decomposed to Y and O atoms. Y and O resolved in the base alloy. When the mechanical alloyed powders were annealed and consolidated at high temperature, Y-Ti-O particles formed with a smaller size, and the particles dispersed uniformly.
3.2 Tensile strength

Mechanical alloying is a nonequilibrium process and makes pores forming between the powders. In this situation, the solubility of the gas in the mechanical alloyed powders is higher than that of ordinary powders. The gas in pores of the powders is hard to eliminate, while that between the powders are easy to eliminate at high temperature. Thus, the sample with high density consolidated at high temperature is possible to obtain. The theoretical density of 14Cr-ODS steel was about 7.77 kg/m³, while the actual density was about 7.58 kg/m³. Thus, the relative density of sample after consolidation was about 97.6%.

The operating condition in the reactor was very complex. It is unreasonable to acquire high strength or good ductility. Thus, it is rational to evaluate both strength and ductility at high temperature and room temperature. The tensile strength for the samples was tested at room temperature and 700 °C.

![Graph showing tensile properties for the samples of 14Cr-ODS and 14Cr-NA](image1)

**Fig. 2** Tensile properties for the samples of 14Cr-ODS and 14Cr-NA

![Fracture morphologies for the samples of room temperature and 700 °C](image2)

**Fig.3** Fracture morphologies for the samples of room temperature and 700 °C
The test results are shown in Fig. 2. UTS for the sample at room temperature and 700 °C are 1120 MPa and 325 MPa. UTS and yield strength decreased as the increasing temperature. At both temperatures, the UTS and yield strength of the ODS steel was higher than that of samples without Y2O3 addition. Especially at high temperature, UTS of the 14Cr-ODS steel was about twice of 14Cr-NA steel. It means that the addition of Y2O3 strengthens the ODS steels. Thus, the ODS steels exhibited excellent high temperature strength.

The fracture surface morphologies for the samples at high temperature and room temperature are shown in Fig. 3. There is almost no necking for both samples. Amount of cleavage surface was observed for the sample at room temperature, as well as river pattern and cleavage steps. Thus, it is typical brittle fracture for the sample at room temperature. However, there are dimple surface for the sample at high temperature. The sizes and distributions of the dimples are not evenly. This phenomenon is in related to the precipitation and nonmetallic inclusion. The hardness of the 14Cr-ODS steel and 14Cr-NA steel is shown in Table. The results show that the hardness of the 14Cr-ODS steel is higher than that of the 14Cr-NA steel. This is in accordance with that of the tensile strength.

| Samples  | Hardness/HV |
|----------|-------------|
| 14Cr-NA  | 259.8       |
| 14Cr-ODS | 373.7       |

3.3 Microstructure

The metallographic morphologies of the 14Cr-ODS steel and 14Cr-NA steel are shown in Fig. 4. A large number of boundaries were observed for the 14Cr-NA sample. The grain size was comparably with the size of the pre-alloyed powders. This means the grain boundary was the boundaries between the powders. The grain size for the samples of 14Cr-ODS steel is smaller than that of 14Cr-NA.

In order to investigate the strengthen mechanism of the nano particles, microstructure and the distribution of the nano particles were observed using TEM, which is shown in Fig. 5. It can be concluded from the figures that nano particles distributed evenly and the size of the particles was about several nano meters. Particles with a size of 20~30 nm was also observed. The chemical composition of the nano particles was indentified using EDS. Because the particle is too small, the chemical composition can only be indentified roughly. The results showed the particles might be nano Y-Ti-O particles.

![Fig.4 Metallographic morphologies for the samples 14Cr-ODS and 14Cr-NA](image-url)
4. Conclusion
Homogeneous ODS powders can be fabricated after mechanical alloying for 30 h with a speed of 380 rpm. Y-Ti-O phase precipitated after annealing the MA powders at 1100 °C for 1 h.

The tensile strength of the 14Cr-ODS steel at room temperature and 700 °C was higher than those of 14Cr-NA steel, 1120 MPa and 325 MPa respectively.

Nano particles distributed evenly in the base alloy. EDS results show that the particles should be nano Y-Ti-O particles.

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