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Role of Cr and Ti contents on the microstructure and mechanical properties of ODS ferritic steels

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Abstract

Six oxide dispersion strengthened (ODS) ferritic steels, with the composition of Fe-(12-14)Cr-2W-(0.1-0.3-0.5)Ti-0.3Y_2O_3 (wt.%), have been prepared by mechanically alloying elemental powders of Fe, Cr, W, and Ti with Y_2O_3 nano-particles followed by hot isostatic pressing. The influence of the chemical composition on the microstructure and mechanical properties of various materials was studied. It was found that the chromium content has a significant influence on the microstructure and mechanical properties of the compacted ingots. The 14Cr ODS steel exhibits slightly higher ultimate tensile strength and yield strength values than the 12Cr ODS steel. The total elongation and uniform elongation of both materials, in general, decrease with raising the test temperature, although in the case of the 12Cr ODS steel the elongation is about 30% higher than that of the 14Cr ODS material. In what concerns the effect of titanium content it can be concluded that variations between 0.1 and 0.3% have no visible effects on the microstructure and Charpy impact properties of compacted specimens. However, the microstructure of specimens with 0.5%Ti contains large TiO_2 particles with a size in the range of 50-500nm, which have detrimental influence on the mechanical properties of that material.

Introduction

Reduced activation oxide dispersion strengthened (ODS) ferritic steels with 12-14Cr (wt.%) have recently gained peculiar attention for various high-temperature structural applications in future fusion reactors. These materials are attractive due to their excellent high temperature mechanical properties and high resistance to irradiation-induced swelling [1-2].

The properties of ODS steels are strongly correlated with Cr, Ti and W contents of the alloy. In general, these main alloying elements extend the δ ferrite loop in the Fe-C phase diagram, suppressing the austenite. The presence of chromium in ODS steels greatly improves the corrosion resistance by forming a very thin, protective oxide layer on the surface of the steels. Chromium added alone to steels has a small influence on the solid solution strengthening of the alloy. Titanium plays an important role in the dissolution of Y_2O_3 particles and subsequent formation of complex nanoclusters enriched with Y, Ti and O. This has a strong and positive influence on the creep resistance of alloys at high temperatures [3].

In this work, the microstructure, Charpy impact and tensile properties of six ODS ferritic steels with the composition of Fe-(12-14)Cr-2W-(0.1-0.3-0.5)Ti-0.3Y_2O_3 (wt%) after MA and HIPping were investigated.

Experimental procedure

Six materials, with the composition of Fe-(12-14)Cr-2W-(0.1-0.3-0.5)Ti, were prepared by mechanically alloying in a planetary ball mill (Retsch PM100) elemental powder of Fe, Cr, W and Ti with 0.3Y_2O_3 (wt%) nano-particles in an argon or a hydrogen atmosphere and hot isostatic pressing (HIP) at 1150°C under a pressure of 200 MPa for 4h, followed by annealing at 850°C for

1h in vacuum and cooling down slowly to ambient temperature, to obtain ingots with more than 99% relative density. Microscopic observations were performed using scanning electron microscopy (SEM) and transmission electron microscopy (TEM, JOEL 2010). Charpy impact tests were conducted at various temperatures between -100°C and 300°C using KLST specimens (3×4×27 mm^3). The ductile-to-brittle transition temperature (DBTT) was determined by using the equation: y=(USE-LSE)/2+LSE, where USE is the upper shelf energy and LSE is the lower shelf energy. Tensile tests were performed at temperatures between 23ºC and 900ºC in a Zwick ZO10T universal machine using flat specimens (1.5×0.5×8 mm^3) and a strain rate of 0.1 mm/min.

Results and discussion

Fig. 1 presents typical TEM images of the microstructure of ODS ferritic steels. In general, all materials are characterized by a bimodal size distribution of equiaxed grains (bcc, α-Fe), containing areas with a high dislocation density and dislocation free areas. Residual porosity (< 1%), and chromium oxides and carbides, usually located at the grains boundaries were also observed. Moreover, in both 12-14Cr ODS materials with the highest titanium content (0.5wt.%) larger titanium oxide particles, about 50-500nm in diameter, were observed (see Fig. 2).

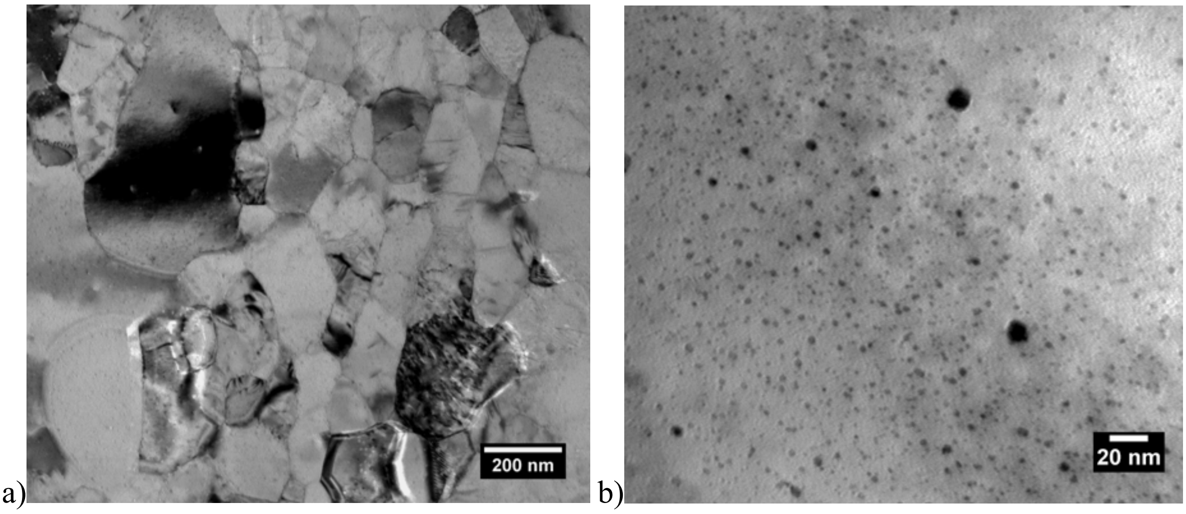


Fig. 1. TEM images of the microstructure of the Fe-12Cr-2W-0.3Ti-0.3Y_2O_3 ODS ferritic steel: a) general view and b) nano-particles.

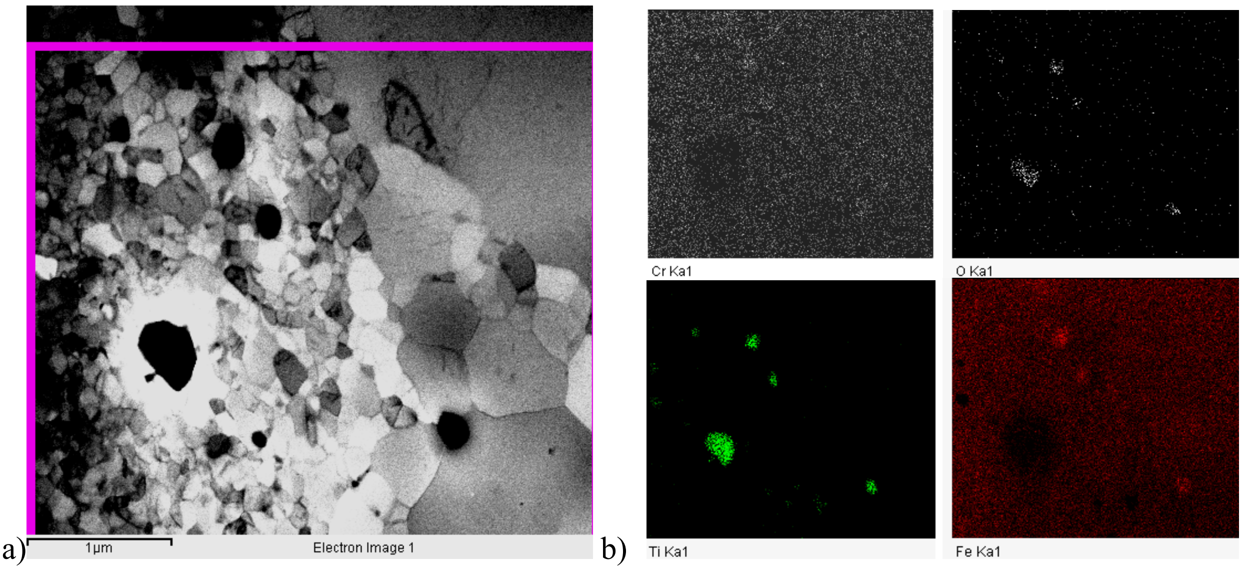


Fig. 2. STEM image of the Fe-12Cr-2W-0.5Ti-0.3Y_2O_3 ODS ferritic steel: a) general view and b) chemical mapping (TiO_2 precipitates).

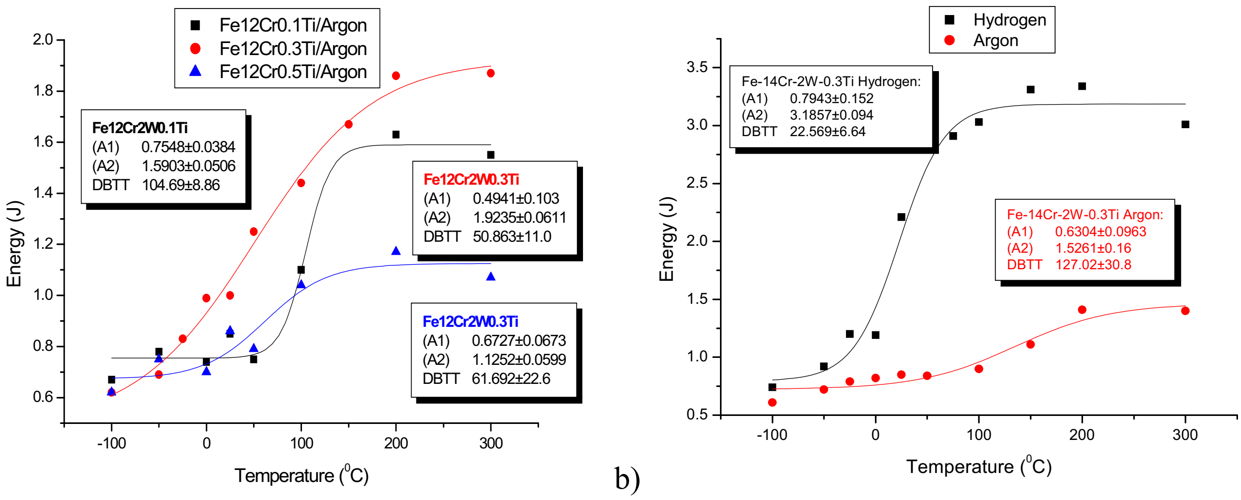
Interestingly, in the case of 12Cr ODS steels some areas with martensite were observed in ingots prepared from the powders mechanically alloyed in argon. This indicates that these materials may have a less stable microstructure than the 14Cr ODS alloys. Mukhopadhyay et al. [4] reported that a minimum Cr content of 13.5 wt.% is required to get a fully ferritic structure. Our investigation revealed however that the use of a high purity hydrogen atmosphere results in the formation of 12-14Cr ODS ferritic steels with a fully ferritic microstructure.

Table 1 presents quantitative results of image analysis of Y-Ti-O nano-particles. These results show no significant differences between 12Cr and 14Cr materials. It can also be seen that the mean size decreases and the number density of nanoparticles increases with increasing the titanium content. However, specimens with 0.5%Ti contain large Ti oxide impurities which were not considered as nano-clusters and have a detrimental influence on the Charpy impact properties of these ODS alloys. Only particles smaller than 10nm were taken into accounts as Y-Ti-O nano-particles.

Table 1
Results of images analysis of Y-Ti-O nano-particles (TEM, thin foil)

ODS material	Particle size [nm]	Particle density [m^{-3}]	Free particle distance [nm]
12Cr-2W-0.3Ti	3.35±1.9	2.31×10^{22}	47.2±29
14Cr-2W-0.1Ti	3.8±1.2	1.42×10^{22}	68.2±38
14Cr-2W-0.3Ti	3.12±2.3	2.18×10^{22}	45.2±25
14Cr-2W-0.5Ti	2.8±0.96	5.23×10^{22}	15.2±7.3

The results of Charpy impact tests are presented in Fig. 3. In general, all 12-14Cr ODS materials are very brittle and exhibit very low USE and very high DBTT values of about 1.5J and 100°C, respectively. The Charpy impact properties of the specimens with 0.5%Ti, with large TiO_2 particles, are worse in comparison to those of the 0.3%Ti ODS materials (Fig. 3a). Significant improvement in the Charpy impact properties was obtained by changing the MA atmosphere from argon to hydrogen. The USE increased by more than a factor of two (up to 3.2J) and the DBTT decreased down to 22.5°C (Fig. 3b), due to a decrease of oxygen content from 0.48% to 0.37% [5]. Heat treatment at 850ºC has a negligible effect on the Charpy impact properties of ODS materials.



a) b)

Fig. 3. Charpy impact energy vs. test temperature for: a) Fe-12Cr-2W-(0.1-0.3-0.5)Ti-0.3Y_2O_3 mechanically alloyed in argon and b) Fe-14Cr-2W-0.3Ti-0.3Y_2O_3 materials mechanically alloyed in different atmospheres.

Results of tensile tests on the Fe-(12-14)Cr-2W-0.3Ti-0.3Y_2O_3 ODS ferritic steels mechanical alloying in hydrogen are presented in Fig. 4. Both materials show a similar behaviour. With increasing the test temperature the yield strength ($YS_{0.2}$) and ultimate tensile strength (UTS) decrease [6]. The 14Cr ODS steel, however, exhibits higher $YS_{0.2}$ and UTS values (by 5% to 15%, depending on the temperature) than the 12Cr ODS steel. These tendencies are observed up to 750°C.

The uniform elongation (UE) and total elongation (TE) values, in general, decrease with raising the test temperature. However, the 12Cr ODS steel exhibits a higher TE (by about 35%) than the 14Cr ODS alloy. In addition, the total elongation of the 12Cr ODS steel exhibits a sharp increase at 600°C. At this temperature a TE value of 24% was measured, which is twice larger than for the 14Cr ODS steel. This is probably associated with a dynamic recrystallization phenomenon and/or a change in rate controlling slip systems.

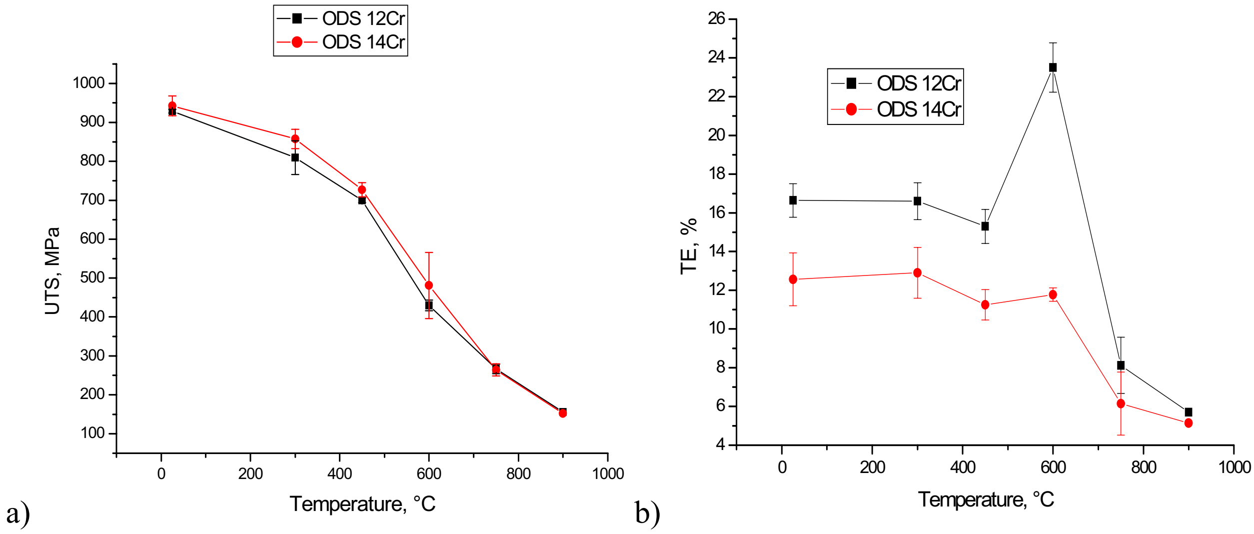


Fig. 4. Ultimate tensile stress and total elongation of the Fe-(12-14)Cr-2W-0.3Ti-0.3Y_2O_3 ODS steels vs. test temperature.

These results indicate that both ODS ferritic steel materials exhibit relatively high strength and reasonable elongation values up to about 750°C.

Summary

The following results were obtained:

- The microstructure of 12-14Cr ODS materials after hipping consists of an α-Fe (bcc) matrix with chromium oxides and carbides and Y-Ti-O nano-clusters. In the 12Cr ODS ferritic steels zones with martensitic laths are observed when the mechanical alloying is performed in an argon atmosphere.
- All compacted materials exhibit a very low USE and a relatively high DBTT. Residual porosity as well as oxide impurities have a detrimental influence on the Charpy impact properties. The use of a hydrogen atmosphere improves significantly the Charpy impact properties of the materials.
- With increasing the titanium content the number density of nano-particles also increases but TEM observations showed that 12-14Cr ODS alloys with 0.5%Ti content contain large (50-500 nm) TiO_2 particles. It is suggested not to use a Ti content above 0.3 wt.%.
- Tensile tests showed that the compacted 14Cr ODS materials exhibit a better strength than 12Cr ODS materials but the latter ones have superior ductility up to about 750ºC.

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