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Effect of mechanical alloying atmosphere on the microstructure and Charpy impact properties of an ODS ferritic steel

Z. Oksiuta *, N. Baluc

Ecole Polytechnique Fédérale de Lausanne, Centre de Recherches en Physique des Plasmas, Association Euratom-Confédération Suisse, 5232 Villigen PSI, Switzerland

ABSTRACT

Two types of oxide dispersion strengthened (ODS) ferritic steels, with the composition of Fe–14Cr–2W–0.3Ti–0.3Y_2O_3 (in weight percent), have been produced by mechanically alloying elemental powders of Fe, Cr, W, and Ti with Y_2O_3 particles either in argon atmosphere or in hydrogen atmosphere, degassing at various temperatures, and compacting the mechanically alloyed powders by hot isostatic pressing. It was found in particular that mechanical alloying in hydrogen yields a significant reduction in oxygen content in the materials, a lower dislocation density, and a strong improvement in the fast fracture properties of the ODS ferritic steels, as measured by Charpy impact tests.

1. Introduction

It is well known that oxide dispersion strengthened (ODS) ferritic and ferritic/martensitic (F/M) steels prepared by mechanical alloying (MA) and hot isostatic pressing (hipping) exhibit relatively weak Charpy impact properties and fracture toughness with respect to conventional ferritic and F/M alloys [1,2]. This is due to the presence of residual porosity, high oxygen and carbon contents yielding the formation of oxide and carbide impurities, inhomogeneous grain size and dislocation distributions. In order to produce ODS steels with improved mechanical properties, oxygen and carbon contents have to be reduced to very low values. It was reported that the origin of porosity in ODS steels might arise from MA atmosphere [3–5]. The effect of MA atmosphere on the oxygen content and mechanical properties of ODS ferritic and F/M steels have been investigated by several authors [6–9]. Three different atmospheres were commonly studied, namely argon, helium and hydrogen. In most cases it was suggested that argon atmosphere is the most promising protective medium against oxidation of the powders during MA. However, it was reported by Klimiankou et al. [3,10], for instance, that argon bubbles are present in ODS F/M steels produced by MA in argon atmosphere and hipping, and that argon as a protective atmosphere during MA is not recommended. As some major issues concerning MA atmosphere and degassing process remain open, this work was aimed at investigating the influence of MA atmosphere, namely argon or hydrogen, as well as degassing temperature, on the chemical composition, microstructure, microhardness and Charpy impact properties of the 14Cr–2W–0.3Ti–0.3Y_2O_3 ODS ferritic steel.

2. Experimental procedure

ODS ferritic steel powders with the chemical composition of Fe–14Cr–2W–0.3Ti–0.3Y_2O_3 (in weight percent) were produced by mechanically alloying elemental powders of Fe, Cr, W, and Ti (about 10 μm in size) with Y_2O_3 particles (about 20–30 nm in size) in a planetary ball mill, in pure argon (99.9999%) or hydrogen atmosphere. After MA the powders were sealed, degassed at various temperatures ranging between 150 and 850 °C until a vacuum of 10^{-2} Pa is reached, and closed in a stainless steel container. Hipping experiments were performed under a pressure of 200 MPa, at 1150 °C for 4 h.

The MA time of elemental powders with Y_2O_3 particles was optimized by means of X-ray diffractometry (XRD). Loss-of-weight measurements were performed on the mechanically alloyed powders after degassing at various temperatures. The microstructure of the powders and compacted specimens was investigated using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). Vickers microhardness measurements were performed at room temperature using a Vickers diamond pyramid and applying a load of 0.98 N for 15 s. The relative density of the specimens after hipping was measured by means of the Archimedes method. Charpy impact tests were performed on KLST specimens ($3 \times 4 \times 27$ mm^3), using an instrumented Charpy impact machine with an energy capacity of 30 J, at temperatures ranging between −100 and 300 °C. Chemical analyses were performed using wavelength dispersive X-ray fluorescence

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* Corresponding author. Tel.: +41 56 310 2957; fax: +41 56 310 4529.
E-mail address: zbigniew.oksiuta@psi.ch (Z. Oksiuta).

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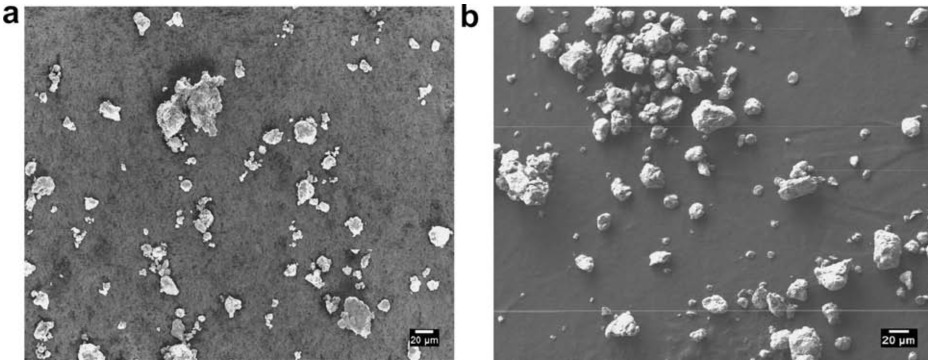

Fig. 1. Morphology of the ODS ferritic steel powder particles after MA in (a) argon atmosphere, and (b) hydrogen atmosphere.

spectroscopy (WD-XRF) as well as LECO TC-436 and LECO IR-412 analysers for detection of O, N and C contents, respectively.

3. Results and discussion

3.1. Analysis of the powder particles

After 43 h of MA it was observed that the morphology of ODS steel powder particles had changed from a spherical shape to a more irregular shape with a larger surface area (Fig. 1). SEM observations of the powders showed that MA atmosphere has no influence on the morphology of the particles. Laser analysis data showed that MA atmosphere has no influence on the mean size of the particles, equal to about 20 μm in the case of both types of powders. High magnification SEM showed that single aggregated particles actually consist of a large number of smaller ones.

Results of chemical analyses of the ODS steel powder before and after MA in pure argon atmosphere are reported in Table 1. It can be seen that the Fe, Cr, W, and Ti elemental powders used in this work contain high oxygen and carbon contents. The chemical composition of the ODS steel powder changed slightly as the result of MA.

3.2. Loss-of-weight measurements

Fig. 2 presents results of loss-of-weight measurements versus degassing temperature. It can be seen that the ODS steel powder mechanically alloyed in argon exhibits a negligible loss-of-weight at the degassing temperature of 150 °C, which slightly increases with increasing degassing temperature. A much larger loss-of-weight was measured for the ODS steel powder mechanically alloyed in hydrogen, especially at the degassing temperature of 850 °C. This unexpected behaviour results probably from reaction of hydrogen with carbon present on the powder particles surface, which can reduce metal oxides in a mixture of CO, CO_2, C_2H_2, and H_2O [11–13]. Therefore, a temperature of 850 °C seems to be sufficient for degassing ODS steel powders. Unfortunately, in spite of these efforts the oxygen content in the ODS steel powder produced in this work is still too high to ensure high performance of the material.

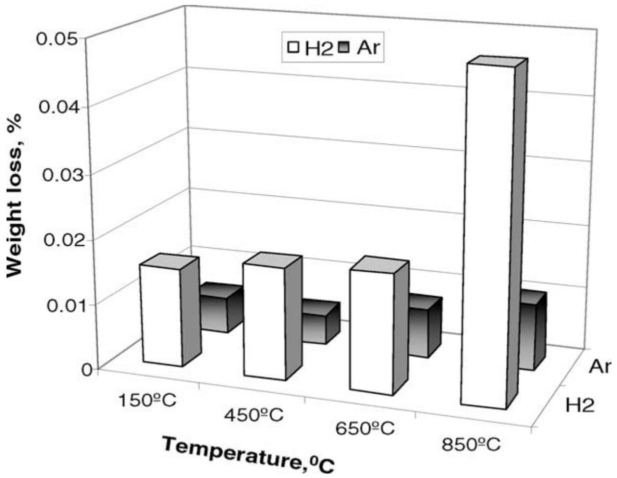

Fig. 2. Loss-of-weight versus degassing temperature for ODS ferritic steel powders mechanically alloyed in argon or hydrogen atmosphere.

3.3. Analysis of compacted materials

The MA atmosphere has significant influence on the microhardness of the ODS steel powders. A larger microhardness was measured for the hipped ODS steel mechanically alloyed in argon

Table 1
Chemical composition of ODS ferritic steel powders before and after MA in argon atmosphere.

Elements	C	Si	Cr	W	Ti	Mn	Y	O	N
Before MA	0.078	0.018	14.0	1.96	0.30	0.12	0.23	0.45	0.038
After MA	0.088	0.031	13.7	1.84	0.26	0.16	0.21	0.48	0.038

Table 3
Chemical composition of hipped ODS ferritic steels mechanically alloyed in argon or hydrogen atmosphere.

Elements	C	Si	Cr	W	Ti	Mn	Ta	Y	O
MA in H_2	0.0266	0.018	14.3	2.01	0.211	<0.01	0.001	0.321	0.37
MA in Ar	0.0461	0.031	13.8	1.94	0.263	<0.01	0.001	0.308	0.49

Table 2
Vickers microhardness of the ODS ferritic steel powders, after MA in argon or hydrogen atmosphere and/or degassing at various temperatures, and of the corresponding hipped ODS ferritic steels.

Sample	$\mu HV_{0.1}$ after MA	$\mu HV_{0.1}$ HT at 450 °C	$\mu HV_{0.1}$ HT at 650 °C	$\mu HV_{0.1}$ HT at 850 °C	$\mu HV_{0.1}$ after hipping	Density after hipping, g/cm^3
14Cr Ar	938.2 ± 37.7	875.5 ± 81.8	848.8 ± 39.1	645.1 ± 81.8	406.7 ± 21.3	7.730
14Cr H_2	825.3 ± 68.7	803.1 ± 71.6	786.2 ± 29.8	517.2 ± 65.4	343.9 ± 14.0	7.770

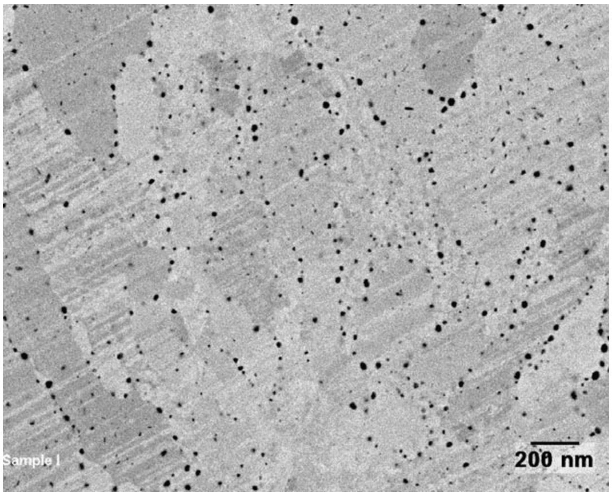

Fig. 3. SEM image of residual porosity in the hipped ODS ferritic steel mechanically alloyed in argon atmosphere.

with respect to hydrogen (Table 2). In addition, the microhardness clearly decreases with increasing degassing temperatures, in the case of both types of powders. However, the density was found higher for the hipped ODS steel mechanically alloyed in hydrogen with respect to argon (Table 2). The chemical composition of the hipped materials, as reported in Table 3, confirm indirectly oxygen and carbon reductions in the ODS steel powder during MA in hydrogen, as discussed under Section 3.2.

As the ODS steel specimens have a density in the range of 99.1–99.5% of the theoretical one, spherical pores decorate the grain boundaries (Fig. 3). A higher density of pores was observed in the case of the hipped ODS steel mechanically alloyed in argon atmosphere. Residual porosity is produced by the gas atoms present during hipping, which fill the interconnected pores or are trapped by the lattice defects during MA process.

Fig. 4 presents the microstructure of both types of hipped ODS steels, mechanically alloyed in argon or hydrogen. A fully ferritic microstructure was obtained in both cases. Both types of materials are composed of grains with sizes ranging between a few hundred nanometres and a few microns, but there are also some areas where grain sizes are less than 100 nm. A high density of nano-clusters, enriched with Y, Ti, and O, less than 10 nm in diameter, as well as tangles of dislocations, were also observed. The hipped ODS steel mechanically alloyed in argon contains a higher density of dislocations than the material mechanically alloyed in hydrogen.

3.4. Charpy impact testing

Results of Charpy impact tests are presented in Fig. 5. The hipped ODS steel mechanically alloyed in argon atmosphere exhibits a very low USE of about 1.4 J and a very high DBTT of about 120 °C (Fig. 5(a)). A significant improvement in the Charpy

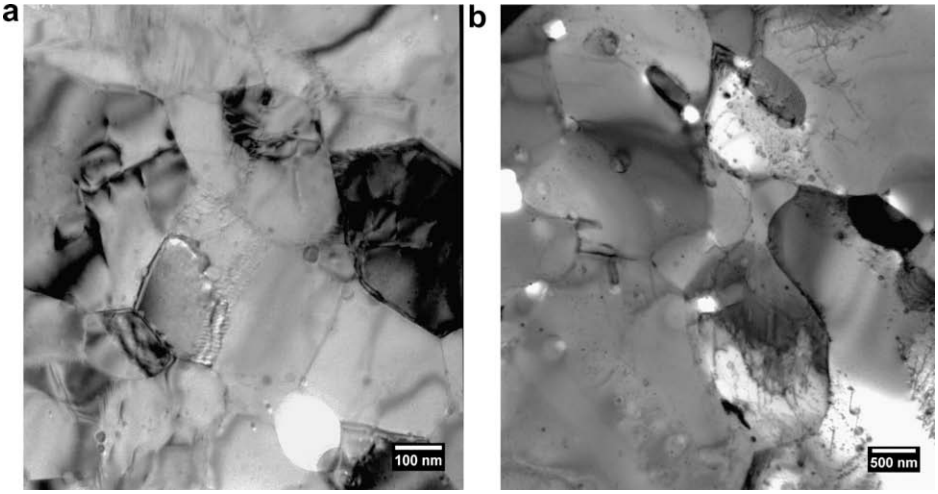

Fig. 4. TEM images of the hipped ODS ferritic steel mechanically alloyed in (a) hydrogen atmosphere, and (b) argon atmosphere.

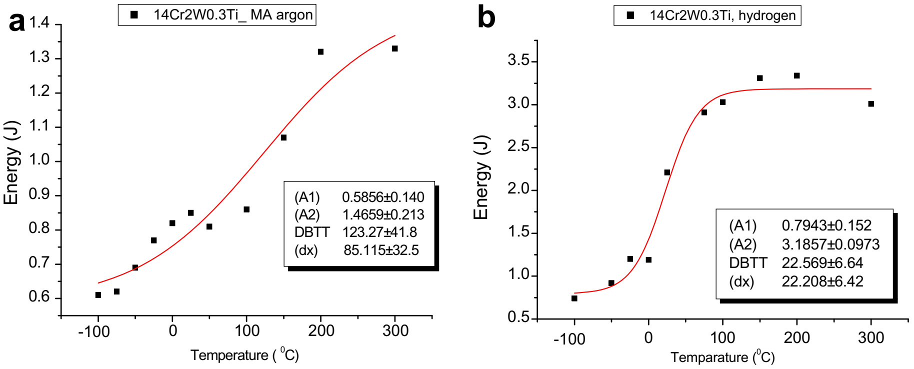

Fig. 5. Absorbed energy versus temperature, as measured by Charpy impact tests, for the hipped ODS ferritic steel mechanically alloyed in (a) argon atmosphere, and (b) hydrogen atmosphere.

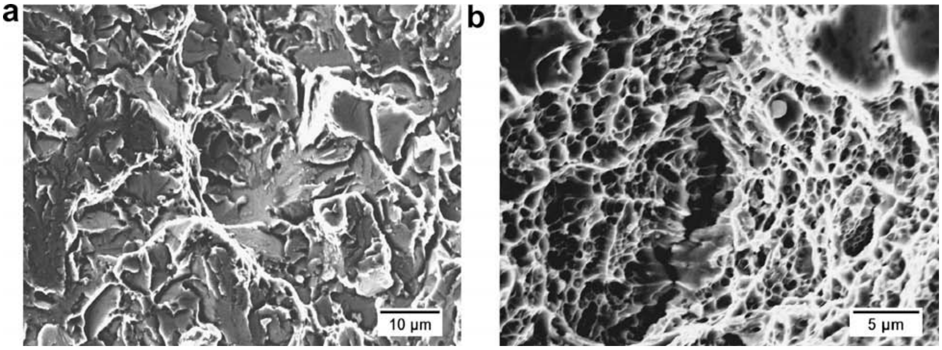

Fig. 6. SEM images of fracture surfaces of the hipped ODS ferritic steel mechanically alloyed in hydrogen atmosphere, after Charpy impact testing at (a) 25 °C (cleavage and ductile fracture), and (b) 200 °C (ductile fracture).

impact properties has been obtained by performing MA in hydrogen atmosphere, characterized by a strong increase in USE to about 3.2 J and a strong reduction in DBTT to about 23 °C, due to lower oxygen content and dislocation density in that material.

SEM observations of the fracture surfaces of Charpy impact specimens showed that the fracture modes of both types of ODS steels are not the same (Fig. 6). The main feature of fracture surfaces of the hipped ODS steel mechanically alloyed in argon is micro-porosity, which is responsible for micro-cracks initiation and propagation and may also act as cleavage fracture initiator. On the contrary, in the case of the hipped ODS steel mechanically alloyed in hydrogen, ductile striations can be seen at low magnification, while micro-dimples, about 1 μm in diameter, can be seen at medium magnification. As in this material the density of microvoids is lower than in the material mechanically alloyed in argon, cracks nucleation occur not only at the level of the pre-existing pores but also on small inclusions.

It is well known that it is relatively easy to remove hydrogen from a work-hardened powder by degassing and also during hipping, as hydrogen may diffuse through the bulk material to the surface. Assuming that at high degassing temperature reduction of oxygen may happen (at least partially) in ODS steel powders, hydrogen atmosphere is not only protective but also reductive atmosphere and should be used for MA of ODS ferritic steels. To confirm this statement more mechanical tests have to be performed.

4. Conclusions

In this work, elemental powders of Fe, Cr, W, and Ti have been mechanically alloyed with Y_2O_3 particles to produce ODS ferritic steel powders with the composition of Fe–14Cr–2W–0.3Ti–0.3Y_2O_3 (in weight percent). MA was performed either in ultra-high purity argon atmosphere or in hydrogen atmosphere. Then, the powders were degassed at various temperatures and compacted by hipping. It was found that:

- The MA atmosphere has no influence on the morphology and particle size distribution in the ODS steel powders. However, MA atmosphere and degassing temperature have a strong influence on the oxygen content, the loss-of-weight and the microhardness of the ODS steel powders.
- After hipping a higher density and smaller microhardness were measured for the ODS ferritic steel mechanically alloyed in hydrogen with respect to argon.
- Improved Charpy impact properties, with an USE of about 3.2 J and a DBTT of about 23 °C, were obtained by mechanically alloying the ODS ferritic steel powders in hydrogen atmosphere.

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