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Influence of Y_2O_3 and Fe_2Y additions on the formation of nano-scale oxide particles and the mechanical properties of an ODS RAF steel

Z. Oksiuta [a,*], M. Lewandowska [b], P. Unifantowicz [c], N. Baluc [c], K.J. Kurzydlowski [b]

[a] Bialystok Technical University, Bialystok, Poland
[b] Warsaw University of Technology, Warsaw, Poland
[c] Ecole Polytechnique Fédérale de Lausanne (EPFL), Centre de Recherches en Physique des Plasmas, 5232 Villigen PSI, Switzerland

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ABSTRACT

The main goal of this work was to manufacture an oxide dispersion strengthened (ODS) reduced activation ferritic steel from a pre-alloyed, gas atomised Fe–14Cr–2W–0.2Ti (in wt.%) powder mechanically alloyed with either 0.3%Y_2O_3 or 0.5%Fe_2Y particles and consolidated by hot isostatic pressing, and to investigate its microstructure, microhardness and Charpy impact properties.

A lower oxygen content was measured in the ODS Fe_2Y steel than in the ODS Y_2O_3 steel. However, the mean size of nanoclusters in the ODS Fe_2Y steel was found larger, whereas density was smaller, than in the ODS Y_2O_3 steel. In addition, the nanoclusters in the ODS Fe_2Y steel appear less stable upon thermal annealing at 1350 °C for 1 h. Vickers microhardness measurements revealed that after HIPping the ODS Y_2O_3 is about 40% harder (366 $HV_{0.1}$) than the ODS Fe_2Y (260 $HV_{0.1}$). After heat treatment at 1350 °C the microhardness of both alloys was found smaller by about 30%. The ODS Fe_2Y steel was found to exhibit a much better Charpy impact behaviour, with an upper shelf energy of 8.8 J and a ductile-to-brittle transition temperature of −24 °C. The differences in mechanical properties were discussed in terms of the oxygen content as well as in the mean size, number density and crystallographic structure of the nanoclusters.

1. Introduction

Oxide dispersion strengthened (ODS) reduced activation ferritic (RAF) steels are promising candidate materials for first wall and breeding blanket applications in the future fusion reactors. These materials are attractive due to their excellent high temperature mechanical properties and good resistance to neutron irradiation [1–3].

Powder metallurgy (PM) technique yields the formation of small grains and a high density of nanoclusters enriched with Y, Ti and O. However, there are still some problems with the manufacturing route of these materials. It is commonly known that the main issue during fabrication by PM, especially during mechanical alloying (MA), is to control and maintain the level of oxygen and carbon contents as low as possible [4,5]. Several studies have been performed on ODS reduced activation ferritic/martensitic (RAF/M) and ODS RAF steels in order to improve understanding of the influence of nanocluster composition on the microstructure and mechanical properties as well as on the nanocluster stability at high temperatures [6]. From the literature, it is known that the Y–Ti–O nanoclusters are thermally very stable up to about 1300 °C for short annealing times of about 1 h. However, there is little information available about the impact of the Fe_2Y intermetallic compound on the mechanical properties and thermal stability of ODS RAF steels. Fe_2Y is used instead of yttria during the mechanical alloying process to reduce the oxygen content. In this paper the effects of two different types of reinforcing powders, namely yttria (Y_2O_3) nano-particles (size 20–40 nm) and iron-yttrium (Fe_2Y) intermetallic compound particles (size <45 μm), have been investigated in terms of thermal stability up to 1350 °C and Charpy impact properties.

2. Experimental procedure

A pre-alloyed ODS powder with the chemical composition of Fe–14Cr–2W–0.2Ti, produced by gas atomization in argon, was MA with 0.3Y_2O_3 or 0.5Fe_2Y (in wt.%) in a planetary ball mill for 20 h, in a hydrogen atmosphere, followed by hot isostatic pressing (HIPping) at 1150 °C under a pressure of 200 MPa for 3 h. After consolidation the ODS RAF steel samples were annealed for 1 h, in an argon atmosphere, at a temperature ranging between 850 and 1350 °C.

* Corresponding author at: Bialystok Technical University, Mechanical Department, Wiejska 45 C, 15-351 Bialystok, Poland. Tel.: +48 85 746 9254.
E-mail address: oksiuta@pb.edu.pl (Z. Oksiuta).

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Table 1
Chemical composition of the pre-alloyed powder and ODS ferritic steel powders after MA for 20 h in hydrogen.

Elements	Atomised	ODS Fe_2Y	ODS Y_2O_3
C	0.005	0.057	0.060
Mn	0.355	0.370	0.368
Cr	13.97	13.82	13.80
W	2.01	1.89	1.92
Ti	0.201	0.186	0.191
Y	–	0.22	0.18
O	0.0142	0.094	0.196

The microstructure was examined using transmission electron microscopy (TEM). Microhardness measurements were carried out using a Vickers diamond pyramid by applying a load of 0.98 N for 15 s. Chemical analyses were performed using gas spectroscopy analysis as well as LECO TC-436 and LECO IR-412 analysers. Charpy impact tests were performed at temperatures ranging between −100 °C and 300 °C using a Charpy impact machine with an energy capacity of 30 J and notched KLST specimens (3 mm × 4 mm × 27 mm).

3. Results and discussion

3.1. Microstructure of the ODS steels

The chemical compositions of the ODS steel powders after MA are shown in Table 1, in comparison to the one of the pre-alloyed powder. As expected, in both materials after MA the oxygen and carbon contents are significantly higher. However, the ODS Fe_2Y powder contains two times less oxygen than the ODS Y_2O_3 one. Thus, one may conclude that by using Fe_2Y intermetallic compound particles, instead of Y_2O_3, the oxygen content in the MA powders can be reduced below 0.1 wt.%.

TEM images of the as-HIPped specimens are presented in Fig. 1. The overall microstructure of both ODS materials is typical of as-HIPped materials, with a mixture of small and large α-Fe (bcc) grains with an average grain size of about 5 μm. Large oxide precipitates, mostly titanium oxides, about a few hundreds nanometers in diameter, were also observed in both materials, usually located at the grain boundaries. Some differences, however, can be observed in the size and density of the nanoclusters. The ODS Y_2O_3 material contains finer and more densely distributed nanoclusters (Fig. 1b). For this material the average nanocluster size, as determined from TEM images, is about 3.8 nm. Similar results were reported by Yamashita et al. [7]. The ODS Fe_2Y steel contains more than two times larger nanoclusters (mean size: about 9.0 nm) that appear less densely distributed (Fig. 1d).

The microstructure of the ODS ferritic steels after annealing up to 1150 °C did not change significantly. However, annealing at 1350 °C for 1 h caused meaningful grain and nanocluster coarsening in the ODS Fe_2Y material. The mean grain size increased up to about 15 μm and the nanocluster size up to 100 nm (see Fig. 2).

However, high resolution TEM observations of the ODS Fe_2Y steel after annealing revealed that some of the very fine nanoclusters actually stay unchanged (see Fig. 2c), while another significantly coarsened. EDS-TEM analyses of the fine nanoclusters revealed that they are mainly yttrium oxides, while the chemical composition of the larger ones appears not uniform. Larger nanoclusters are either titanium oxides, yttrium oxides or they have more complex chemistry of the Ti–Y–O type. Selected area electron diffraction (SAED) of the larger Y_2O_3 nanoclusters (Fig. 2d) revealed that they have an austenitic γ-fcc structure. This in not in accordance with the data reported in the literature [6], which indicates that Y–Ti–O nanoclusters have a bcc crystallographic structure. More detailed analyses need to be conducted to find out why the ODS Fe_2Y steel contains nanoclusters with a fcc structure. However, there are no doubts that these particles are less thermally stable in comparison with those evidenced in the ODS Y_2O_3 steel. This was confirmed by TEM observations (Fig. 2e). After annealing at 1350 °C the ODS Y_2O_3 alloy exhibits significantly smaller nanoclusters than the ODS Fe_2Y material. However, the average nanocluster size in the ODS Y_2O_3 steel also increased upon thermal annealing, but only up to about 10 nm. Further high resolution TEM analyses have to be performed to confirm the crystallographic structure of the nanoclusters and to get information about the coarsening mechanism at high temperatures.

3.2. Mechanical properties of the ODS steels

Vickers microhardness measurements revealed that after HIPping the ODS Y_2O_3 is about 40% harder (366 $HV_{0.1}$) than the ODS Fe_2Y (260 $HV_{0.1}$). After heat treatment at 1350 °C the microhardness of both alloys was found smaller by about 30%. This trend is in a good accordance with the literature data [8,9] however, the overall hardness presented here slightly varies with the ODS alloys manufactured using different parameters.

Results of Charpy impact tests are shown in Fig. 3. As expected, the HIPped pre-alloyed powder (designated A&D) exhibits the best impact properties. Surprisingly, the ODS Y_2O_3 steel exhibits a very low upper shelf energy (USE) value of about 2.4 J and a high ductile-to-brittle transition temperature (DBTT) value of about 77 °C. Heat treatment of this material at 850 °C for 1 h in argon did not improve its impact properties. The ODS Fe_2Y material shows significantly better impact properties, with a DBTT of -24 °C and an USE of 8.8 J. The oxygen content as well as the type, size and density of the nanoclusters have certainly a strong impact on the strength and fracture behaviour of ODS RAF steels. In the present case, the lower oxygen content in the ODS Fe_2Y material, associated with larger and less dense nanoclusters having eventually a peculiar crystallographic structure, yield a softer material with improved Charpy impact properties.

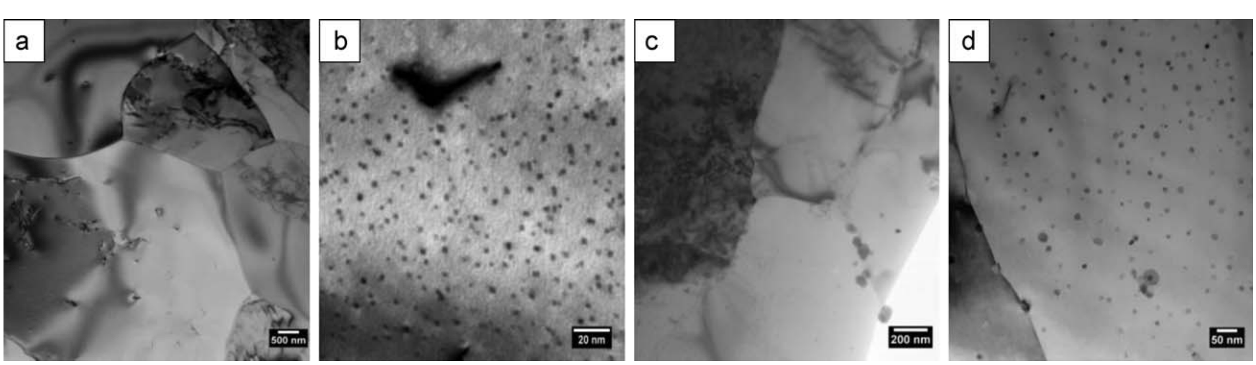

Fig. 1. TEM images of the general microstructure and nanoclusters in the ODS steels after HIPping: (a) and (b) ODS Y_2O_3, and (c) and (d) ODS Fe_2Y.

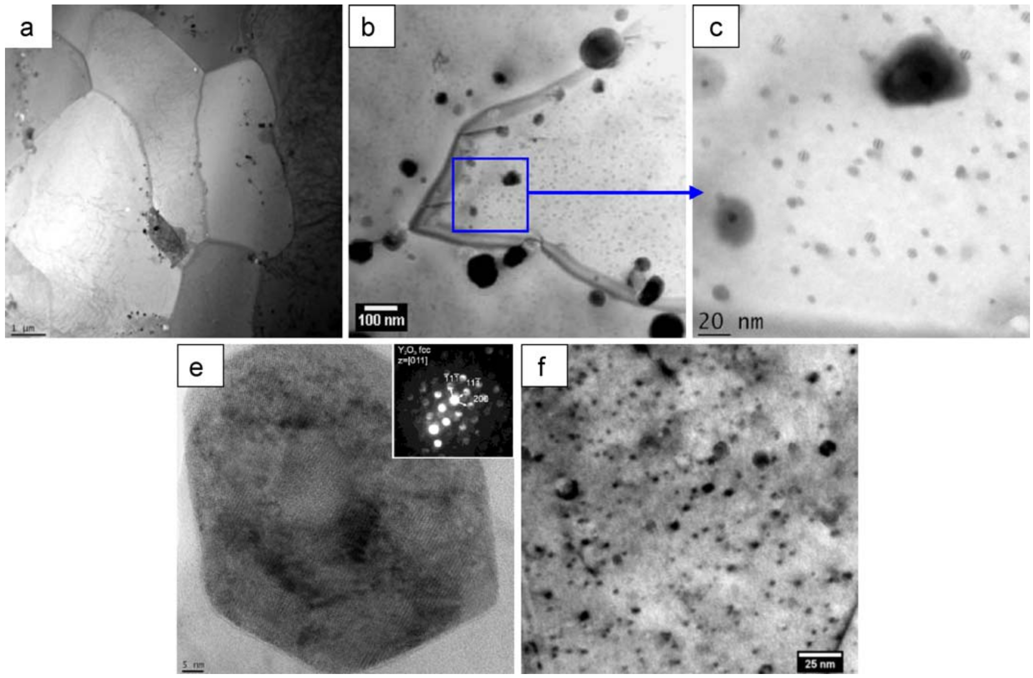

Fig. 2. TEM images of the microstructure of the ODS steels after HIPping and HT at 1350 °C: (a) ODS Fe_2Y, general view, (b) and (c) ODS Fe_2Y, nanoclusters, and (d) ODS Fe_2Y, coarse Y_2O_3 nanocluster and SED pattern, and (e) ODS Y_2O_3, nanoclusters.

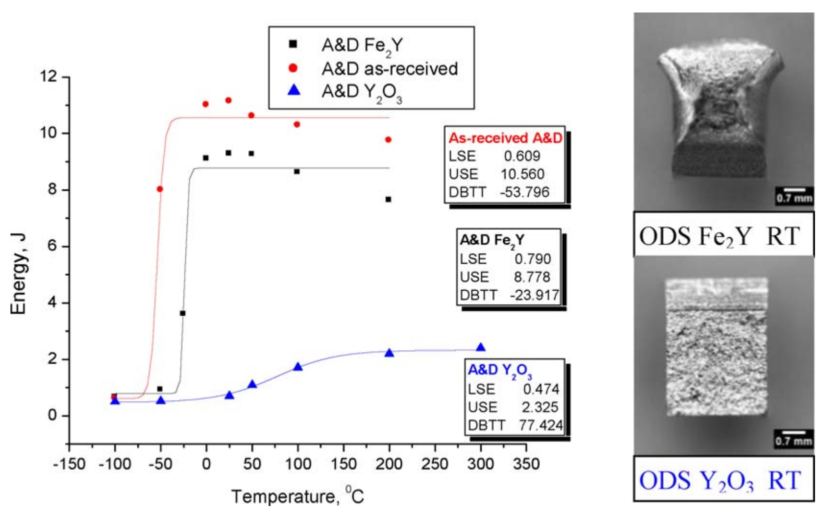

Fig. 3. Charpy impact and fracture appearance results of the ODS ferritic steels. Fracture appearance is taken from room temperature (RT).

4. Conclusions

It was found that by using Fe_2Y instead of Y_2O_3 powder particles during mechanical alloying results in a reduction of the oxygen content in ODS RAF steels. TEM observations revealed that the nanoclusters that form during HIPping are then larger and less densely distributed than in the case of ODS Y_2O_3 steel. In addition, annealing of the ODS Fe_2Y material at 1350 °C for 1 h brings about significant grain growth and nanocluster coarsening. SAED-TEM investigations of the coarser nanoclusters in the ODS Fe_2Y steel after annealing at 1350 °C revealed that they have an austenitic γ-fcc structure.

The ODS Fe_2Y material exhibits significantly better Charpy impact properties, with a DBTT of −24 °C and an USE 8.8 J, whereas the ODS Y_2O_3 material has a very low USE of about 2.4 J and a high DBTT of about 80 °C. Thus, using intermetallic Fe_2Y powder particles instead of Y_2O_3 powder particles can be an effective approach for producing a softer material with improved impact properties. However, the thermal stability of the nanoclusters in ODS Fe_2Y upon aging and irradiation as well as high-temperature creep behaviour of the material are also important concerns.

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References

[1] M.J. Alinger, G.R. Odette, G.E. Lucas, J. Nucl. Mater. 307–311 (2002) 484.
[2] M.K. Miller, D.T. Hoelzer, E.A. Kenik, K.F. Russell, J. Nucl. Mater. 329–333 (2004) 338–341.
[3] S. Ukai, A. Mizura, T. Yoshitake, T. Okuda, M. Fujiwara, S. Hagi, T. Kobayashi, J. Nucl. Mater. 283–287 (2000) 702–706.
[4] S. Ohtsuka, S. Ukai, M. Fujiwara, T. Kaito, T. Narita, J. Phys. Chem. Solids 66 (2005) 571–575.
[5] I. Monnet, P. Dubuisson, Y. Serruys, M.O. Ruault, O. Kaitasov, B. Jouffrey, J. Nucl. Mater. 335 (2004) 311–321.
[6] M. Klimiankou, R. Lindau, A. Möslang, J. Nucl. Mater. 329–333 (2004) 347–351.
[7] S. Yamashita, N. Akasaka, S. Ohnuki, J. Nucl. Mater. 329–333 (2004) 377–381.
[8] M.K. Miller, K.F. Russell, D.T. Hoelzer, J. Nucl. Mater. 351 (2006) 261–268.
[9] M.J. Alinger, G.R. Odette, D.T. Hoelzer, Acta Mater. 57 (2009) 392–406.