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Review on the EFDA work programme on nano-structured ODS RAF steels

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Abstract: The 2008–2009 work programme of the European research project on nano-structured oxide dispersion strengthened (ODS) reduced activation ferritic (RAF) steels is being organized along the four following programmatic lines: (1) improve the present generation of nano-structured ODS RAF steels; (2) start the industrial fabrication of the present generation of nano-structured ODS RAF steels; (3) develop an optimised generation of nano-structured and nano-grained ODS RAF steels; (4) investigate the stability of present and optimised generation of nano-structured ODS RAF steels under creep and irradiation. This paper presents the main objectives of current R&D activities being performed within the European research project on nano-structured ODS RAF steels, the main obtained results and the main future activ-ities in the case of the four programmatic lines mentioned just above.

1. Introduction

As the upper temperature for use of reduced activation ferritic/martensitic (RAFM) steels for structural applications in fusion power reactors, such as the European EUROFER 97 RAFM steel, is presently limited by a drop in mechanical strength at about 550 °C (see e.g. [1]), Europe, Japan and the US are actively research-ing steels with high strength at higher operating temperatures, mainly using stable oxide dispersion. In addition, the numerous interfaces between the matrix and the oxide particles are expected to act as sinks for the irradiation-induced defects and transmuta-tion elements such as He and H [2]. Main R&D activities aim at finding a compromise between good tensile and creep strength and sufficient ductility, especially in terms of fracture toughness. It has been recently found that the oxide dispersion strengthened (ODS) RAFM steels, such as the ODS EUROFER steel, could be used for structural applications in fusion power reactors up to about 650 °C (see e.g. [3]). The nano-structured ODS reduced activation ferritic (RAF) steels appear as very promising materials as they are expected to be used for structural applications in fusion power reactors up to about 750 °C (see e.g. [4]).

The plate supporting the tungsten tiles in the European dual-coolant lithium–lead (DCLL) breeding blanket concept [5] and the cartridge within the finger-like parts of the European He-cooled divertor concept [5] are presently foreseen to be made of ODS EUROFER [6,7]. The use of ODS RAF steels with a higher creep strength up to about 750 °C and a reasonable fracture toughness at ambient and intermediate temperatures will provide these com-ponents with additional integrity margin and lifetime.

The 2008–2009 work programme of the European research pro-ject on nano-structured ODS RAF steels is being organized along the four following programmatic lines: (1) improve the present generation of nano-structured ODS RAF steels; (2) start the indus-trial fabrication of the present generation of nano-structured ODS RAF steels; (3) develop an optimised generation of nano-structured and nano-grained ODS RAF steels; (4) investigate the stability of present and optimised generation of nano-structured ODS RAF steels under creep and irradiation.

This paper presents the main objectives of current R&D activi-ties being performed within the European research project on nano-structured ODS RAF steels, the main obtained results and

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the main future activities in the case of the four programmatic lines mentioned just above.

2. Improve the present generation of nano-structured ODS RAF steels

2.1. Main objectives

The main objectives of this programmatic line are (i) to optimize parameters of manufacturing by powder metallurgy, including mechanical alloying followed by either hot isostatic pressing (HIPping) or hot extrusion and thermal–mechanical treatments, in order to obtain a dense population of small nano-clusters and sub-micrometre grains, both conditions being required for high creep strength and reasonable fracture toughness after irradiation, and (ii) to fabricate small batches of about 1 kg at the laboratory scale.

2.2. Main results

Main recent activities focused on the production of ODS RAF steels with the composition in the range of Fe–(12–13–14)Cr–(1–2)W–(0.3–0.5)Ti–0.3Y_2O_3 (in weight percent) by mechanical alloying, canning and degassing of the milled powders, compaction of the powders by HIPping, and thermal–mechanical treatments, by using various devices and conditions. In a first step, mechanical alloying and HIPping parameters have been optimized in the aim to obtain dense ingots with a microstructure composed of a homogeneous distribution of small grains containing a high density of small nano-clusters enriched with Y, Ti and O (see Fig. 1), and as few impurities (O, C, etc.) as possible. For instance, the optimal manufacturing parameters for a Fe–14Cr–2W–0.3Ti–0.3Y_2O_3 ODS RAF steel produced by mechanically alloying elemental powder particles with Y_2O_3 particles in a planetary ball mill and HIPping are summarized in Fig. 2 [8]. All manufactured materials exhibit a high tensile strength and a reasonable ductility up to about 750 °C (see Fig. 3). However, the microstructure of 12Cr materials appears less stable than the one of 13Cr and 14Cr materials, in the sense that there is a risk of martensitic phase transformation in 12Cr materials in the case of significant carbon contamination during mechanical alloying. In addition, 0.5Ti materials contain large TiO_2 particles that may cause embrittlement effects. Although the tensile properties appear very good, the fracture properties are still too weak, in terms of fracture toughness as well as in terms of ductile-to-brittle transition temperature (DBTT) and upper shelf energy (USE) as measured by means of Charpy impact tests (see Table 1). For instance, after HIPping and heat treatment the Fe–14Cr–2W–0.3Ti–0.3Y_2O_3 material prepared from elemental powder particles mechanically alloyed with Y_2O_3 particles in a planetary ball mill exhibits a DBTT of about 22 °C and an USE of about 3.2 J. Preliminary thermal–mechanical treatments, such as hot pressing or hot rolling, were found to yield a higher tensile strength and a strongly improved Charpy impact behaviour, when considering a given type of powder and manufacturing process (see Table 1).

On the other hand, a Fe–14Cr–1W–0.3Ti–0.3Y_2O_3 ODS RAF steel has been prepared by mechanically alloying Fe–14Cr–1W–0.3Ti pre-alloyed powder particles with Y_2O_3 particles, followed by hot extrusion of the milled powder in the shape of cylindrical bar, and characterized in terms of microstructure and Charpy impact,

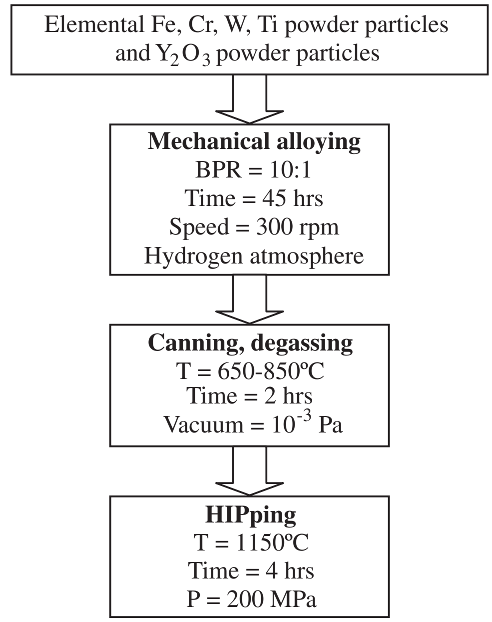

Fig. 2. Optimal processing conditions for a Fe–14Cr–2W–0.3Ti–0.3Y_2O_3 ODS RAF steel prepared by mechanically alloying elemental powder particles with Y_2O_3 particles in a planetary ball mill and HIPping.

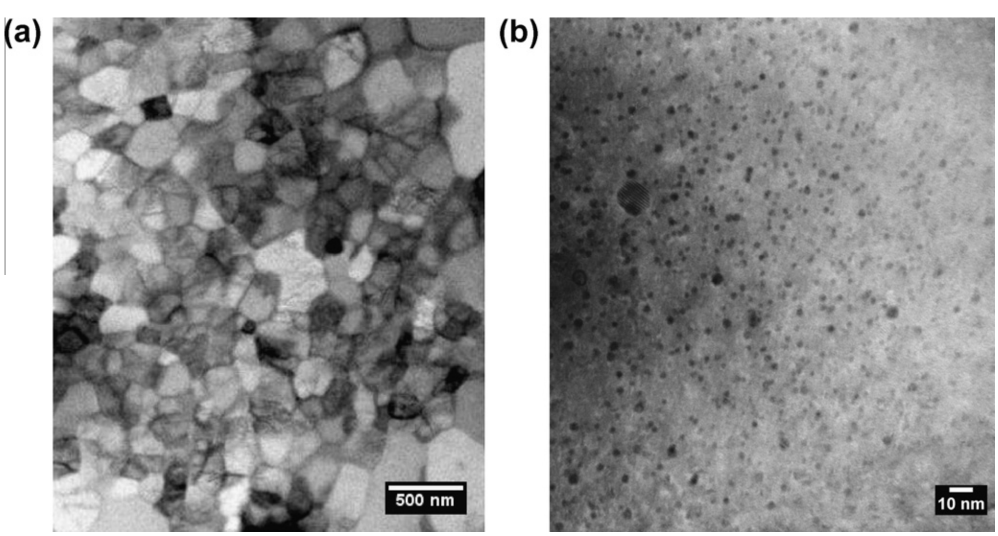

Fig. 1. Transmission electron microscopy images of the microstructure of a Fe–14Cr–2W–0.3Ti–0.3Y_2O_3 ODS RAF steel prepared by mechanically alloying elemental powder particles with Y_2O_3 particles in a planetary ball mill and HIPping: (a) general view and (b) high magnification image of 5–6 nm nano-clusters.

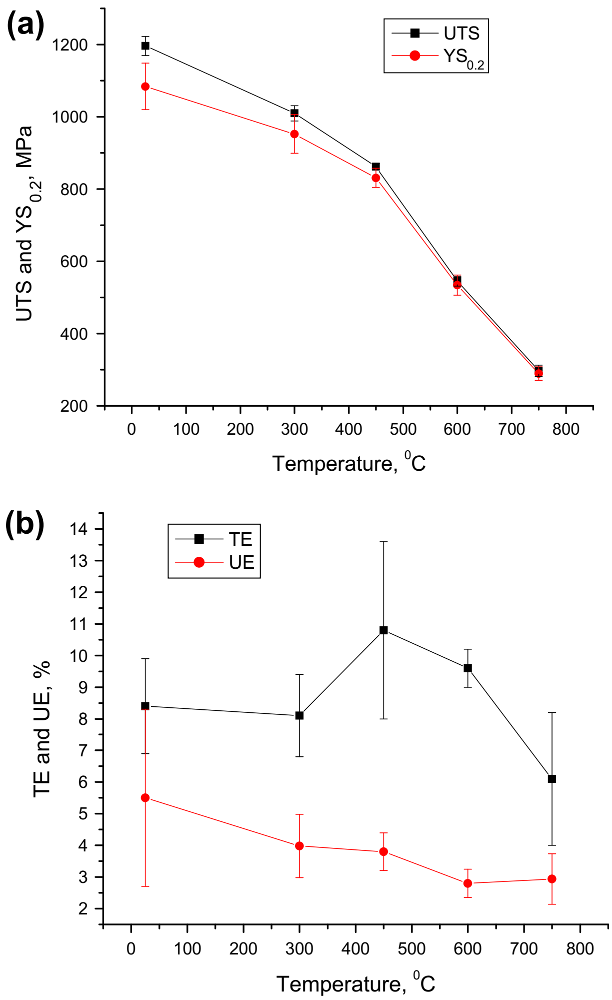

Fig. 3. Tensile properties versus test temperature of a Fe–14Cr–2W–0.3Ti–0.3Y_2O_3 ODS RAF steel prepared by mechanically alloying pre-alloyed powder particles with Y_2O_3 particles in an attritor, HIPping, hot rolling and heat treatment at 850 °C: (a) yield strength ($YS_{0.2}$) and uniform tensile strength (UTS), (b) uniform elongation (UE) and total elongation (TE).

Table 1
Effects of various thermal–mechanical treatments (hot pressing or hot rolling) on the DBTT and USE, as measured by means of Charpy impact tests, of a Fe–14Cr–2W–0.3Ti–0.3Y_2O_3 ODS RAF steel prepared by mechanically alloying either pre-alloyed or elemental powder particles with Y_2O_3 particles in either an attritor or a planetary ball mill, respectively, and HIPping: HT = heat treatment at 850 °C, HP = hot pressing at 850 °C, HR = hot rolling at 850 °C.

Type of powder, mechanical alloying device	Thermal–mechanical treatment	DBTT (°C)	USE (J)
Elemental, planetary ball mill	HT	22	3.2
Pre-alloyed, attritor	HT	140	3.1
Pre-alloyed, attritor	HP + HT	63	4.5
Pre-alloyed, attritor	HR + HT	68	4.2

tensile and fatigue behaviours. As expected, the performance of this hot extruded material, with a DBTT close to −100 °C (Charpy impact tests), is much closer to the one of the material produced by McClintock et al. [9] by mechanical alloying, hot extrusion and multi-steps hot rolling, which exhibits a DBTT well below −100 °C (fracture toughness measurements), than to the one of the materials produced by HIPping.

Such R&D studies have been accompanied by more systematic investigations of the tungsten content, extrusion geometry and testing direction on the micro-mechanisms of cleavage fracture of ODS ferritic steels, as well by the production and characterization of model alloys (e.g. Fe–14Cr, Fe–14Cr–0.3Y_2O_3, Fe–14Cr–0.1Y).

2.3. Future activities

Optimization of the manufacturing route will be pursued with some emphasis on thermal–mechanical treatments in order to reduce the DBTT value. Batches of about 1 kg of ODS RAF steel will be manufactured at the laboratory scale by mechanically alloying either elemental powder particles or pre-alloyed powder particles with Y_2O_3 particles, in a planetary ball mill or an attritor, followed by HIPping and adequate thermal–mechanical treatments.

3. Start the industrial fabrication of the present generation of nano-structured ODS RAF steels

3.1. Main objectives

Both presently identified applications require quantities of materials that are orders of magnitude larger than those currently manufactured at the laboratory scale and, hence, will have to be fabricated using industrial-scale methods. The main objective of this programmatic line is to assess the possibility of fabrication of semi-industrial batches of ODS RAF steels of about 10 kg in Europe, on the basis of the optimized manufacturing route that is being defined within the programmatic line described under Section 2.

3.2. Main results

As the Plansee company stopped the production of ODS ferritic steels, there is no longer any industrial company in Europe capable to fabricate large batches of ODS ferritic steels. There is a reasonable hope that the situation may change in the forthcoming 10 years, according to the project of construction of a first prototype of Generation IV reactor starting in 2020, which will most likely be a sodium-cooled fast reactor that uses an ODS ferritic steel (even if not of reduced activation chemical composition) as cladding material for the fuel element. In the mean time, recent activities have been focused on defining a strategic map for semi-industrial fabrication of batches of ODS RAF steels of about 10 kg in Europe, which could be easily extended to the fabrication of larger batches, on the basis of the optimized manufacturing route that is being defined within the programmatic line described under Section 2 and extensive contacts taken with potential industrial partners in Europe as well as with European universities and research centres.

3.3. Future activities

The fabrication of the semi-industrial batch of about 10 kg will be implemented, on the basis of the optimized fabrication route, with a delivery foreseen mid 2011. Similar batches of ODS RAF steels should be also produced at the international level, within the framework of the International Energy Agency (IEA) implementing agreement on Fusion Materials (Annex-II), and their characterization shared between different partners.

4. Develop an optimised generation of nano-structured and nano-grained ODS RAF steels

4.1. Main objectives

The main objectives of this programmatic line are (1) to develop the dynamical phase diagram of the driven Fe–Cr–W–Ti–Y_2O_3 system under mechanical alloying and subsequent thermal–mechanical treatments (see e.g. [10]), in the aim to classify the various mechanical alloying processes, compaction methods and thermal–mechanical treatments required for obtaining a dense population of small nano-clusters, and (2) to determine the optimum grain size and work-hardening level for achieving the best combination of strength, fracture toughness, ductility and radiation resistance, and to assess the fabrication route hindering grain growth (in terms of adequate thermal–mechanical treatments, for instance), as a complement to activities performed within the programmatic line described under Section 2.

4.2. Main results

In order to investigate the kinetic pathway of formation of small nano-clusters enriched with Y, Ti and O, a powder of ODS RAF steel has been prepared by an alternative processing route including reactive ball milling between Fe–Cr–W–Ti powder particles, YFe_3 particles and Fe_2O_3 particles, followed by annealing [11,12]. Nano-clusters with a size in the range of 1–2 nm were identified in the as-milled powder by means of small angle neutron scatter-ing and atom probe tomography (see Fig. 4). Their size remains constant and their volume fraction increases under annealing at temperatures up to 800 °C. It seems that ball milling yields the onset of nano-cluster nucleation that goes on during annealing. Such a powder should be further compacted by hot extrusion and submitted to thermal–mechanical treatments. Atom probe tomography analysis of the bulk MA957 ODS ferritic steel (not a reduced activation material) also revealed that the composition of the nano-clusters is size dependent [13], in agreement with the results obtained by means of transmission electron microscopy on replica [14]. In particular, the Y:Ti ratio increases slightly (to reach a value close to 1) and the O content decreases significantly when the size of the nano-clusters increases. Stochiometric and non-stochiometric compositions have been measured.

On the other hand, first attempts to refine the grain size using either the equal channel angular pressing (ECAP) method or high-speed hot extrusion have been successfully made on the EUROFER 97 RAFM steel and will be pursued on ODS steel variants.

4.3. Future activities

Activities on developing the dynamical phase diagram of the Fe–Cr–Y_2O_3 system and on determining the optimum grain size will be pursued. The achievement of both objectives should provide data for the fabrication of optimised nano-structured and nano-grained ODS RAF steels, with a first specification ready by the end of 2012 for the first fabrication to be carried out in the first half of 2013.

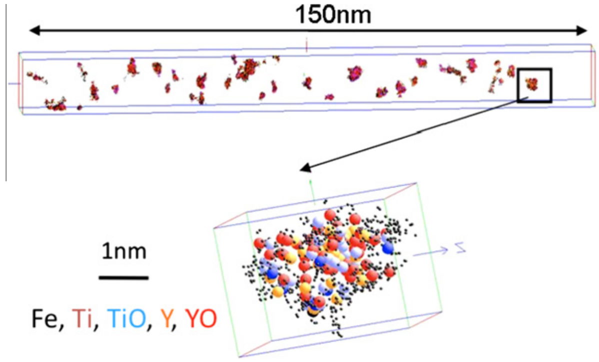

Fig. 4. Atom probe tomography analysis of the chemical composition of nano-clusters evidenced in an ODS RAF steel powder produced by reactive ball milling between Fe–Cr–W–Ti powder particles, YFe_3 particles and Fe_2O_3 particles [11].

5. Investigate the stability of present and optimised generation of nano-structured ODS RAF steels under creep and irradiation

5.1. Main objectives

The main objectives of this programmatic line are (1) to perform post-irradiation examination of the ODS ferritic steels previously irradiated in fission reactors or in spallation neutron sources, and (2) to define the characterisation programme of laboratory scale and semi-industrial European batches of ODS ferritic steel, in terms of long term annealing experiments, creep and fatigue tests, and irradiation experiments. In what concerns the investigation of radiation effects, many-beam ion irradiations should be used to a large extent in order to assess the stability of the nano-clusters and to analyse the helium behaviour in the matrix and at the grain boundaries.

5.2. Main results

In waiting for irradiated specimens of ODS RAF steels, specimens of the MA957 ODS ferritic steel in the hot extruded and cold worked condition, which had been irradiated in the Swiss Spallation Neutron Source (SINQ facility) up to about 20 dpa and high helium and hydrogen contents (50 appm He/dpa and 450 apm H/dpa), at various temperatures ranging between 115 and 360 °C, have been characterized by means of tensile tests and transmission electron microscopy observations. Promising results were obtained, in the sense that it was found that the irradiated MA957 ODS ferritic steel retained a significant ductility (with a total elongation ranging between 6% and 14%, depending on the dose) both at 25 °C and 250 °C testing temperatures.

5.3. Future activities

Preliminary ion irradiations of the most promising ODS RAF steels developed within the programmatic line described under Section 2 will be performed in the dual beam/triple beam JANNUS facility (France). The semi-industrial batches produced within the programmatic line described under Section 3 will be characterized, in terms of long term annealing and creep behaviours and following irradiation in fusion power reactor relevant conditions, i.e., up to high irradiation dose and high helium and hydrogen contents. Concerning the optimised nano-structured and nano-grained ODS RAF steels, a similar strategy and programme could start mid 2013, when the first batches will become available. A specification for the industrial fabrication of an optimised ODS RAF steel should be issued in 2014.

6. Summary

European activities in the field of ODS RAF steels focus on the development of materials combining high strength and creep behaviours at elevated temperatures with reasonable ductility and fracture toughness at low and intermediate temperatures and good radiation resistance under fusion power reactor irradiation conditions. Such R&D activities are strongly supported by fundamental investigations, in order to achieve a detailed understanding of the effects of the chemical composition and manufacturing parameters on the microstructure, and therefore the mechanical properties, of ODS RAF steels for application in DEMO and beyond. This approach should yield the industrial fabrication of an optimized ODS RAF steel in about 5 years from now.

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