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Mechanical and microstructural behaviour of Y_2O_3 ODS EUROFER 97

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Abstract

Two small ingots of the steel EUROFER 97, one containing 0.25 wt% Y_2O_3 and the other Y_2O_3 free, have been produced by consolidating mechanically milled powder by hot isostatic pressing at 1373 K for 2 h under 200 MPa. For comparison, a third ingot was consolidated under identical conditions but using un-milled EUROFER powder. Microhardness, tensile and Charpy tests, along with TEM observations, have been performed on these materials in the as-HIPed condition and after different heat treatments. The mechanical behaviour and the microstructural characteristics of these materials suggest that the origin of the reduced impact properties of the oxide dispersion strengthened EUROFER could be the premature formation of carbides during quenching following the HIP process. This would be enhanced by the high density of structural defects produced by milling, as these favour the fast diffusion and segregation of carbon.

1. Introduction

Dispersion strengthening appears to be the most promising approach to widening the operating temperature window of the ferritic/martensitic steels. Oxide dispersion strengthened (ODS) steels produced by mechanical milling and hot isostatic pressing (HIP) are considered potential structural materials for future fusion reactors [1,2]. At present, the efforts in Europe to develop ODS steels for fusion applications are focused on the reduced activation 9CrW steel EUROFER 97. One of the aims is to find a route to produce ODS EUROFER with improved high temperature properties. The preliminary results indicate that ODS EUROFER 97 with 0.3 wt% Y_2O_3 exhibits high creep resistance and tensile strength at temperatures up to around 920 K but reduced fracture toughness and a high ductile–brittle transition temperature (DBTT) compared to conventional steels [3–6]. To assess the capabilities of ODS EUROFER, it is essential to determine whether the worsening of the impact properties is an inherent characteristic, or is related to either the production process or the thermal treatments after consolidation. Charpy impact tests performed on ODS EUROFER with Y_2O_3 particles point out that the lack of toughness could be inherent, however neither its origin nor the effect of the

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processing parameters and thermal treatments has been comprehensively investigated [6]. The aim of the present work is to investigate the microstructure and mechanical properties of ODS and non-ODS EUROFER powder consolidated by HIP, in order to determine the origin of the degradation of the mechanical properties.

2. Experimental

Gas-atomised EUROFER 97 powder (prepared by Studsvik, Sweden) having particle size <45 μm was milled with 0.25 wt% Y_2O_3 for 24 h under an Ar pressurised atmosphere in a horizontal attrition mill. Details of the procedure and composition of the milled powder have been reported elsewhere [7]. Also, a batch of the same EUROFER powder, Y_2O_3 free, was milled under identical conditions. One hundred and eighty grams of milled Y_2O_3/EUROFER powder was canned and degassed at 673 K for 24 h in vacuum ($<10^{-1}$ Pa), and then the can was sealed. For comparative experiments un-milled and milled EUROFER powders were canned for consolidation. Consolidation was performed by HIP at 1373 K for 2 h under a pressure of 200 MPa. During cooling the temperature decreased exponentially at rates which were higher than 30 K/min at temperatures above the M_s temperature (∼650 K), while the pressure decreased linearly with temperature. This HIP conditions produced a fully dense material with densities of (7.76 ± 0.06) g/cm^3 for ODS EUROFER, and (7.77 ± 0.03) and (7.79 ± 0.02) g/cm^3 for un-milled and milled EUROFER, respectively, compared to the theoretical density of 7.82 g/cm^3.

Lots of miniature flat tensile and KLST Charpy specimens were cut by electrical discharge machining, aligned along the longitudinal direction of the HIPed ingots. The tensile specimens were 0.4 mm thick, 3 mm wide with 11 mm gauge length, and the Charpy specimens $4 \times 3 \times 27$ mm^3 with a V-notch 1 mm deep. Tensile tests at room temperature, and Vickers microhardness and Charpy impact measurements were performed on specimens in the as-HIPed condition and after different heat treatments. A tempering treatment of 2 h at 1023 K was selected for comparison to the previous results in ODS EUROFER [6,8]. Normalization treatments for 30 min in the range 1253–1473 K followed by tempering, both in a vacuum of $<10^{-3}$ Pa, were also performed. The specimens were forced-air cooled after normalizing and tempering. The carbon, oxygen and nitrogen content of the specimens were measured after their respective treatments, and the microstructure was characterized by transmission electron microscopy. The tensile tests were continued to fracture at a strain rate of 6×10^{-5} s^{-1}, and the microhardness measurements used an applied load of 2.94 N. Charpy impact tests were carried out using an instrumented Charpy pendulum (50 J) in the range 123–523 K to obtain a full impact curve.

3. Results

3.1. Mechanical properties

Table 1 summarises the tensile test and microhardness results, along with the C, O and N content, for ODS EUROFER and non-ODS EUROFER. Fig. 1 shows comparative stress–strain curves. As-HIPed Y_2O_3/EUROFER, i.e. the ODS material, resulted in a hardened material with yield and ultimate tensile strengths of 1400 MPa and 1500 MPa, respectively, a uniform elongation to fracture of 1.1%, and a microhardness of 480. This material is softer and less ductile than the CRPP ODS EUROFER produced using the same powder milled under identical conditions, but consolidated at 1273 K for 1 h under 180 MPa after pre-sintering at 1543 K [9]. Tempering after HIP softened the ODS material, although the ductility remained very low. The normalization treatments did not improve the tensile strength or the ductility of the ODS material. The appearance of a yield point in the stress–strain curves for ODS EUROFER tempered after normalizing above 1253 K suggests a certain degree of recovery in the dislocation structure.

The un-milled non-ODS material, in as-HIPed state or after tempering, was more ductile than the corresponding ODS material. However, the milled non-ODS material was harder and less ductile after tempering. In this material, tempering after normalizing at 1473 K reduced the microhardness, and the yield and tensile strength to the corresponding values for the un-milled material, increasing the ductility up to an elongation to fracture of ∼7%.

Fig. 2 shows the Charpy impact curves for ODS and non-ODS EUROFER after tempering, and a comparison curve for EUROFER 97. The un-milled non-ODS material presents a DBTT shift of ∼58 K and 45 K at 3.1 J and 1.9 J, respectively, as compared to base EUROFER 97, and a decrease of the upper shelf energy of ∼1.68 J. Both milled