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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

Table 1
Mechanical properties and impurity content for non-ODS and ODS materials produced by HIP

Treatment	Un-milled non-ODS EUROFER						Milled non-ODS EUROFER						ODS EUROFER					
	C/O/N (wt%)	HV	YS (MPa)	UTS (MPa)	ε_u (%)	ε_f (%)	C/O/N (wt%)	HV	YS (MPa)	UTS (MPa)	ε_u (%)	ε_f (%)	C/O/N (wt%)	HV	YS (MPa)	UTS (MPa)	ε_u (%)	ε_f (%)
As-HIPed	0.08/ 0.09/ 0.07	405 ± 9	977	1380	3.9	4.7	–	–	–	–	–	–	0.09/ 0.20/ 0.06	480 ± 20	1405	1499	1.1	1.1
Tempering after HIP	0.09/ 0.09/ 0.06	240 ± 9	660	790	5.1	12.7	0.11/ 0.19/ 0.06	430 ± 14	–	1065	0.6	0.6	0.10/ 0.22/ 0.06	330 ± 20	1040	1320	3.2	3.8
Tempering after normalizing at 1253 K	0.09/ 0.07/ 0.06	208 ± 13	592	736 ± 2	5.2	14.7	–	–	–	–	–	–	0.09/ 0.20/ 0.06	300 ± 20	994	1148	2.3	2.3
Tempering after normalizing at 1323 K	0.08/ 0.05/ 0.05	224 ± 8	649	789	4.9	11.2	–	–	–	–	–	–	0.08/ 0.14/ 0.06	323 ± 14	768	1167	4.0	6.1
Tempering after normalizing at 1373 K	–	–	–	–	–	–	–	–	–	–	–	–	0.09/ 0.19/ 0.05	322 ± 12	968	1144	3.0	4.3
Tempering after normalizing at 1423 K	–	–	–	–	–	–	–	–	–	–	–	–	0.11/ 0.19/ 0.04	319 ± 12	798	1073	2.2	2.2
Tempering after normalizing at 1473 K	0.08/ 0.05/ 0.04	190 ± 7	620	746	4.1	11.9	0.10/ 0.17/ 0.05	203 ± 14	634	785	4.5	7.3	0.09/ 0.19/ 0.04	300 ± 20	884	1015	2.0	2.1

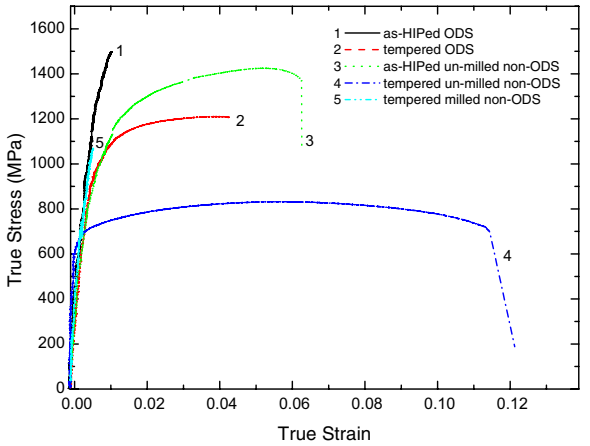

Fig. 1. Comparative stress–strain curves for HIPed EUROFER with various thermal treatments.

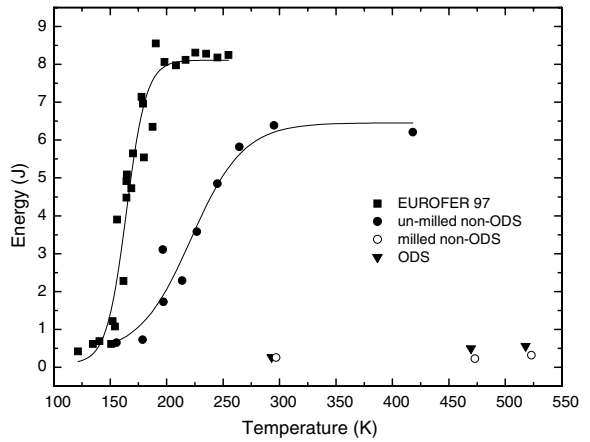

Fig. 2. Variation of absorbed energy in Charpy impact tests versus temperature for base EUROFER 97 and tempered non-ODS and ODS EUROFER.

non-ODS and ODS materials remain brittle up to 523 K. Table 2 shows the absorbed impact energies at room temperature for the milled non-ODS and ODS materials tempered after normalizing. These treatments do not produce any qualitative change in the absorbed energy.

3.2. Microstructural characteristics

Fig. 3 shows TEM images for the different materials. Both milled non-ODS and ODS materials in as-HIPed state present a similar microstructure. The TEM images reveal submicron-sized grains containing a high density of dislocations. No obvious lath martensite structure is observed, although very fine microtwins similar to those observed inside martensite plates are found in some areas of as-HIPed ODS EUROFER, as Fig. 3(a) reveals. In the ODS material, a fine dispersion of Y_2O_3 particles appears quite homogeneously distributed in the matrix. The size distribution of these particles is depicted in Fig. 4.

Table 2
Charpy impact energy at RT for milled non-ODS and ODS EUROFER after different heat treatments

Treatment	Impact energy at RT (J)	
	Milled EUROFER	ODS EUROFER
Tempering after HIP	0.26	0.27
Tempering after normalizing at 1323 K	–	0.31
Tempering after normalizing at 1373 K	–	0.36
Tempering after normalizing at 1423 K	–	0.36
Tempering after normalizing at 1473 K	0.53	0.56

Tempered ODS EUROFER exhibits a microstructure consisting of a few martensite laths in some areas and mainly unrecovered nanometer-size grains, like the ones observed in the as-HIPed material, as can be seen in Fig. 3(b). This microstructure also appears in the milled non-ODS material tempered. Carbide coalescence is found in both materials, as well as in the corresponding un-milled non-ODS material. Fig. 3(c) shows the effect of normalization treatment at 1473 K on the microstructure of the ODS material. These treatments seem to favour the dislocation recovery and the appearance of polygonal subgrains replacing the original martensite laths. The same features occur in the milled non-ODS material tempered after normalizing but the carbide coarsening is more prominent. This coarsening would produce a depletion of the strengthening elements in the matrix, accounting for the noticeable softening observed in this material in comparison to the ODS material.

The un-milled non-ODS material tempered retains the microstructure of EUROFER 97 as Fig. 3(d) shows. A lath martensite structure with a lath width of ~500 nm is evident. The grain size of the prior austenite in this material, measured by optical microscopy, is (14 ± 2) μm, somewhat larger than that corresponding to the reference base material (7–11 μm). The carbides in this material, that are larger than those observed in the base EUROFER 97, appear distributed in the matrix. However, agglomeration of carbide particles is found in some areas.

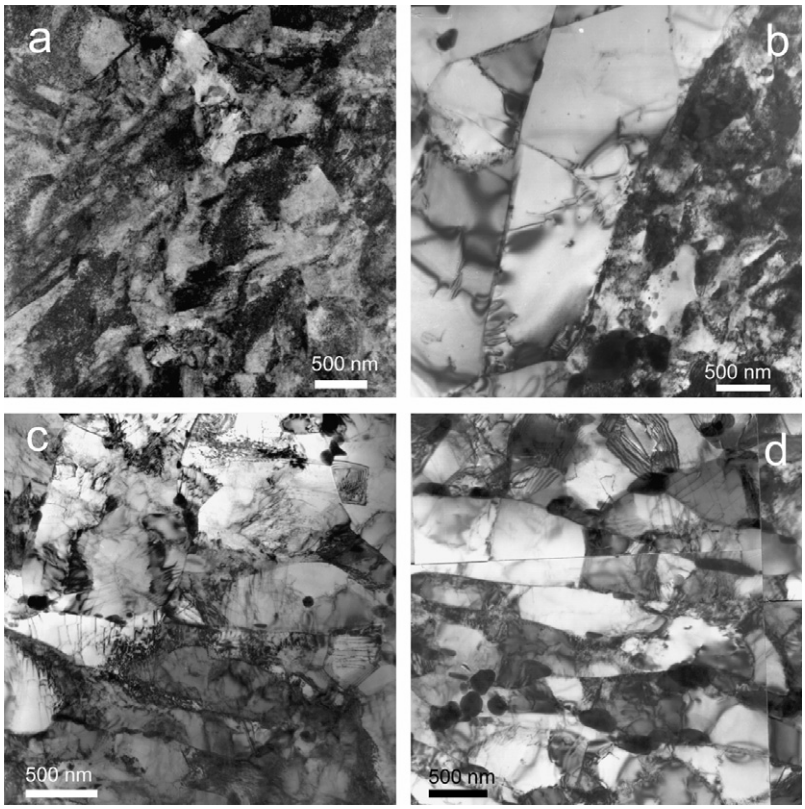

Fig. 3. TEM images of HIPed EUROFER: (a) as-HIPed ODS, (b) tempered ODS, (c) tempered ODS after normalizing at 1473 K and (d) tempered un-milled EUROFER.

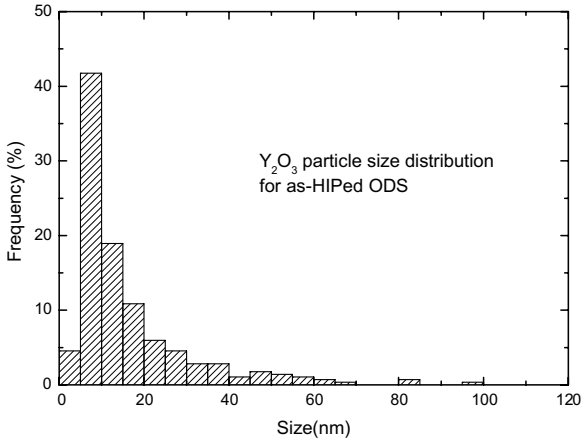

Fig. 4. Size distribution of Y_2O_3 particles in as-HIPed ODS EUROFER.

4. Conclusions

ODS EUROFER prepared following the present procedure resulted in a material harder and less ductile than those produced at Plansee and CEA [3–6,8]. Tempering at 1023 K after normalizing for 30 min in the range 1253–1473 K did not improve the tensile properties of this ODS material, in relation to properties achieved after tempering without a previous normalization treatment.

The reference tempering treatment did not improve the Charpy impact properties of ODS EUROFER and milled non-ODS EUROFER meaningfully, irrespective of normalizing at $T \leqslant 1473$ K. However, the microhardness and tensile properties of milled non-ODS EUROFER coincide with those for un-milled material when it is tempered after normalizing.

The comparative experiments reveal that normalization treatments can reduce the deleterious effect of milling on the ductility of non-ODS EUROFER, but without a qualitative increase in the Charpy impact energy. However, neither the ductility nor the impact toughness in the tempered ODS material are qualitatively changed by normalizing at temperatures up to 1473 K. Since the oxygen content in the milled non-ODS material is similar to that in the ODS material, after subtracting the oxygen amount

in Y_2O_3 form, it does not appear that the oxygen intake by itself produced by milling causes the loss of ductility and impact toughness. The TEM observations suggest that one of the possible causes of embrittlement could be the precipitation of carbides during quenching after the HIP process of the milled powder. This autotempering phenomenon should be favoured by the high density of structural defects produced by milling, which enhances the carbon diffusion and carbide precipitation. TEM observations show that tempering at 1023 K for 2 h, irrespective of normalizing, induces neither the homogeneous precipitation of fine carbides nor the homogeneous recovery required for impact toughness improvement. Fractography analyses and TEM observations are in progress in order to get more insight about HIP embrittlement of ODS EUROFER.

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