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Development and characterisation of a new ODS ferritic steel for fusion reactor application

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ABSTRACT

This paper describes the microstructure, tensile properties and Charpy impact resistance of a reduced activation oxide dispersion strengthened ferritic steel Fe–14Cr–2W–0.3Ti–0.3Y_2O_3 produced by mechanical alloying of a pre-alloyed, gas atomised steel powder with Y_2O_3 particles, compaction by hot extrusion at 1100 °C, hot rolling at 700 °C and heat treatment at 1050 °C for 1 h. At room temperature the material exhibits a high ultimate tensile strength of about 1420 MPa and high yield strength of about 1340 MPa in the transverse direction. In the longitudinal direction the values are about 10% lower, due to the anisotropy of the microstructure (elongated grains in the rolling direction). At 750 °C the material still exhibits relatively high yield strengths of about 325 MPa and 305 MPa in the longitudinal and transverse directions, respectively. The material exhibits reasonable uniform and total elongation values over the temperature range 23–750 °C, in both transverse and longitudinal directions. The material exhibits weak Charpy impact properties in the transverse direction. Charpy impact properties are slightly better in the longitudinal direction, with upper shelf energy of about 4.2 J and a ductile-to-brittle transition temperature of about 8.8 °C.

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1. Introduction

Reduced activation oxide dispersion strengthened (ODS) ferritic steels for structural application in the future fusion reactors have been first developed and characterized about one decade ago [1,2]. Based on these first experiments at the laboratory scale, about 100 kg of an ODS ferritic steel with the composition of 12Cr–3W–0.4Ti–0.25Y_2O_3 (referred to as 12YWT) was then produced by Kobe Steel Ltd. in Japan [3]. Tensile tests performed on this material revealed a high yield strength of about 1300 MPa, an ultimate tensile strength of about 1400 MPa and an uniform elongation of about 4.5% at room temperature in longitudinal (parallel to rolling) direction [3,4]. The creep strength and high temperature stability of this alloy were also found to be very good. Unfortunately, the ductile-to-brittle transition temperature was determined to be about 100 °C. Therefore, a new material was developed by Oak Ridge National Laboratory in the USA [5,6]. As compared to the 12YWT alloy, this new material, referred to as 14YWT, contained higher amounts of chromium (14 wt%) and yttrium (0.3 wt%) and a number of manufacturing parameters were modified, namely the temperature of hot extrusion, the reduction in thickness per pass during hot rolling as well as the final heat treatment. Tensile properties of the 14YWT material were found slightly better than those of the 12YWT alloy, whereas the ductile-to-brittle transition temperature was significantly reduced down to about −150 °C.

In this work an European Fe–14Cr–2W–0.3Ti–0.3Y_2O_3 ODS ferritic steel was produced by mechanical alloying of a pre-alloyed, gas atomised powder with yttria particles, hot extrusion and hot rolling, and characterized in terms of microstructure, and tensile and Charpy impact properties. Batch of 1.5 kg of the material was produced.

2. Experimental procedure

A pre-alloyed Fe–14Cr–2W–0.3Ti (wt%) powder was prepared by Aubert & Duval (France) by argon gas atomization. The pre-alloyed ferritic steel powder was mechanically alloyed with 0.3Y_2O_3 (wt%) particles at Plansee (Austria) in an attritor ball mill for 48 h in a hydrogen atmosphere. The mechanically alloyed 14Cr ODS steel powder was sieved (<150 μm), put into a low carbon steel can and degassed at 300 °C. Then, about 1.5 kg can was closed and extruded at 1100 °C. A square-shape cross-section extruded bar was then hot rolled at 700 °C to get a thickness reduction of 20% and annealed at 1050 °C for 1 h. More detailed information about manufacturing process of the steel is provided in Ref. [7].

Flat tensile specimens, 25 mm in length, 0.5 mm in thickness, and with a gauge length and width of 8 mm and 1.5 mm, respectively, were cut out from the hot extruded bar in both longitudinal

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Table 1
Chemical composition of the ODS ferritic steel powder (in wt%).

Elements	After gas atomisation	After mechanical alloying
Cr	13.97	13.45
W	2.01	1.92
Ti	0.201	0.33
Ni	0.02	0.13
Mn	0.355	0.38
C	0.0045	0.0043
O	0.0142	0.0914
N	0.0057	0.0085
H	–	0.0039
Y_2O_3	–	0.31

and transverse directions. Tensile tests were curried out at various temperatures ranging between room temperature and 750 °C, using a strain rate of $2 \times 10^{-5}\ s^{-1}$, in an argon atmosphere. At least three specimens were tested at each temperature. The microstructure of the alloy was studied using scanning electron microscopy (SEM) and transmission electron microscopy (TEM). The transmission electron microscope was operated at 200 kV and equipped with an energy dispersive spectrometry (EDS) system. Microhardness measurements at the polished surface of the specimens were carried out by means of a Vickers diamond pyramid (JENOPHOT 2000) and applying a load of 0.98 N for 15 s. Each result is the average of nine measurements. Instrumented Charpy impact tests were performed at various temperatures ranging between −100 °C and 300 °C. V notch KLST ($3 \times 4 \times 27\ mm^3$) specimens for impact tests were machined also in two perpendicular directions of the hot extruded bar. Chemical analysis of the powder before and after mechanical alloying was performed using wavelength dispersive X-ray fluorescence 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

The chemical composition of the powder after gas atomisation and mechanical alloying is listed in Table 1. It is well known that the mechanical alloying technique yields contamination of the milled powder. It can be seen in Table 1 that oxygen and nitrogen contents increased whereas the alloying elements Cr and W contents slightly decreased as a result from ball milling. On purpose the Ti content was increased from about 0.2% after gas atomisation up to about 0.3% after mechanical alloying in order to obtain a Ti/Y_2O_3 ratio close to 1:1.

TEM images of the 14Cr ODS ferritic steel in both longitudinal and transverse directions are presented in Fig. 1. In the longitudinal (rolling) direction elongated grains with a shape ratio from 5:1 up to 20:1 and a high density of dislocations can be seen (Fig. 1(a)). Equiaxed grains with a size from a few hundred nanometers up to a few micrometres can be seen in the transverse direction (Fig. 1(b)).

The average size and density of Y–Ti–O nano-clusters measured from TEM images were found to be about 5 ± 3.7 nm and $1.5 \times 10^{23}\ m^{-3}$, respectively. Coarse particles, up to 250 nm in diameter, were also observed in both directions and identified by means of STEM-EDX detector as Ti and/or Al oxides (Fig. 2). These oxide particles are not uniformly distributed, but usually located at the grain boundaries of the alloy. This in not surprising, since the oxygen not only arises from the mechanical alloying atmosphere but also from the oxide surface layer of the metal powder particles and can create oxides particles with solute elements such as Ti, Cr and Al. It has to be emphasized that Al should not be present in this alloy. So, it is assumed that this impurity comes from the milling balls and from the jar, probably because a steel powder of the PM2000 type was MA in the same milling device before milling the 14Cr ODS ferritic steel powder. As a consequence, a traced amount of Al from the PM2000 type steel powder remained at the surfaces of the jar and balls and caused subsequent contamination of the 14Cr ODS ferritic steel powder. To avoid in the future Al contamination it seems necessary to perform MA process in a ball mill device dedicated to such ODS reduced activation ferritic steels.

A similar microstructure was observed by Miller et al. [5] and Hoelzer et al. [8] in 12YWT and 14YWT ODS alloys. However, it appears that the present 14Cr ODS alloy contains a higher dislocation density and a more elongated microstructure in the rolling direction in comparison to the data reported in the literature for other 12–14YWT ODS steels [5,8]. It seems that the final heat treatment conditions are not sufficient to recover the dislocation microstructure in the present 14Cr ODS alloy, as it also suggested in Ref. [7].

The Vickers microhardness and density values obtained for the 14Cr ODS ferritic steel produced in this work are presented in Table 2. For comparison purposes, data obtained for other 12–14YWT ODS ferritic steels [5] are also included. A density measurement as well as microscopic observations showed that the 14Cr ODS steel is a fully dense material. Microhardness measurements revealed not significant difference in hardness between longitudinal and perpendicular directions, in spite of the difference in the morphology of grains. The microhardness of the 14Cr ODS alloy is about 10% higher than that of the 12YWT alloy [4], which was also hot extruded, hot rolled and annealed at 1000 °C, and about 15%

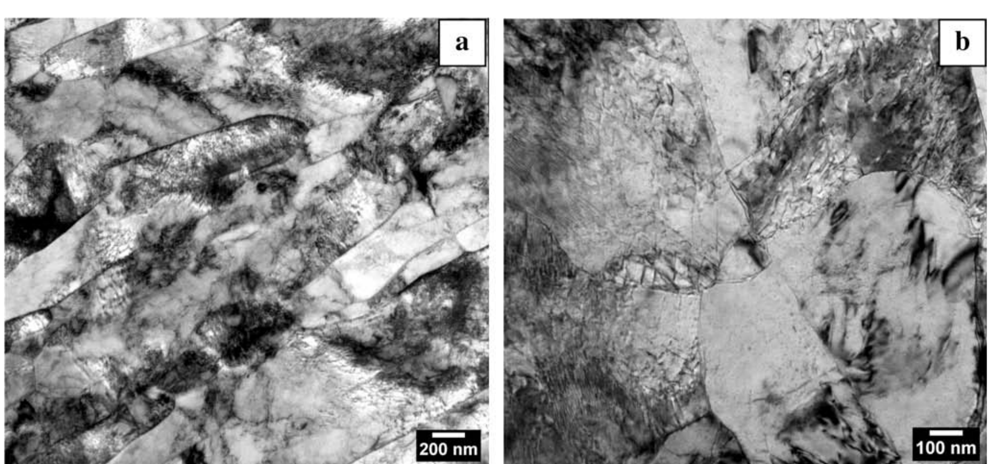

Fig. 1. TEM images of the ODS ferritic steel in (a) the longitudinal direction and (b) the transverse direction.

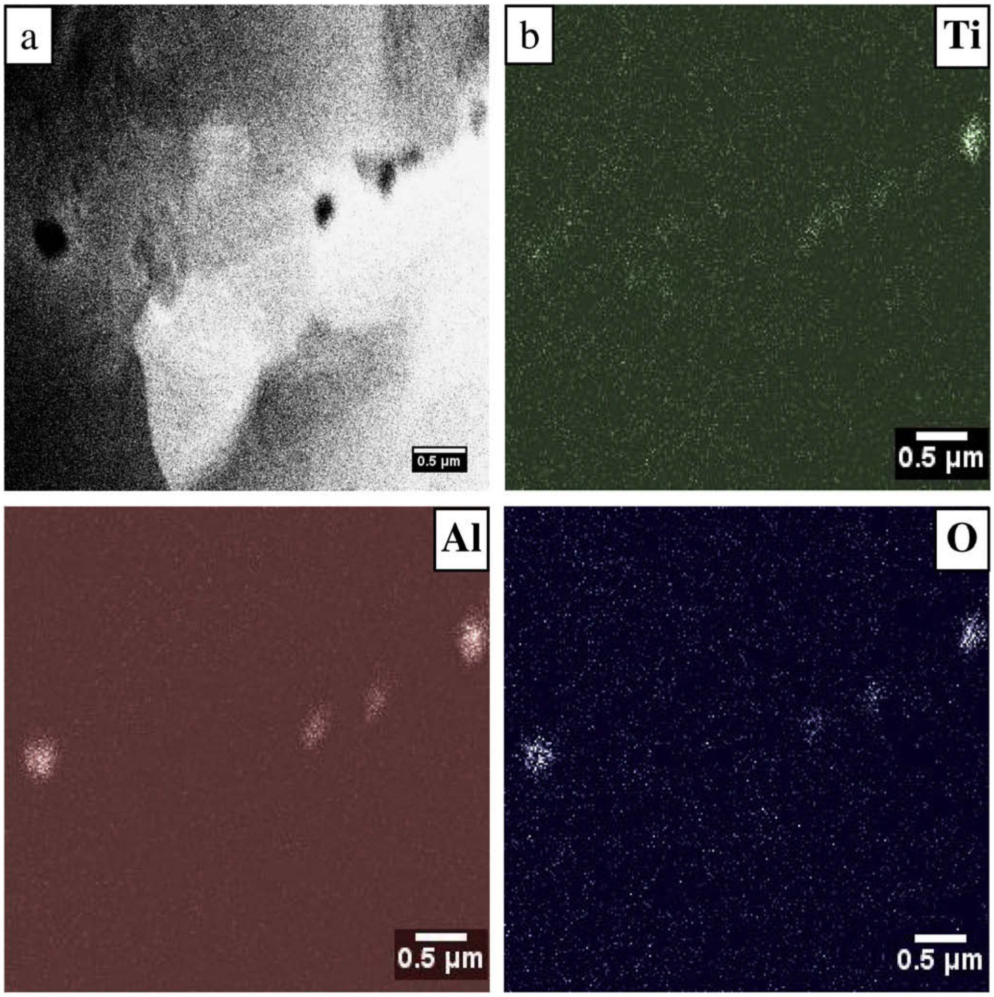

Fig. 2. STEM image of the ODS ferritic steel: (a) general view and (b) elemental chemical mapping.

Table 2
Microhardness and density data for the ODS ferritic steels.

Material	Vickers microhardness, $HV_{0.1}$	Density, g/m^3
14Cr ODS	Longitudinal, 448 ± 12 Transverse, 452 ± 10	7.82
12YWT ODS[a] as processed	Longitudinal, 411 ± 16	–
14YWT ODS[a] HE + HT 1000 °C	Longitudinal, 386 ± 34	–

[a] After Miller et al. [5]. HE: hot extruded, HT: heat treated.

higher than that of the 14WYT material, which was hot extruded and annealed at 1000 °C [6].

Results of tensile tests on the 14Cr ODS steel in both perpendicular directions as a function of test temperature are shown in Figs. 3 and 4.

At room temperature the ODS material in the transverse direction exhibits a high ultimate tensile strength of about 1420 MPa and a high yield strength of about 1340 MPa. In the longitudinal direction the values are about 10% lower, which means that this alloy is stronger in the transverse direction than in the longitudinal one, at least in the temperature range 23–600 °C. A more pronounced decrease in strength with increasing testing temperature is observed above 450 °C. At 750 °C, the highest testing temperature, the ODS material still exhibits a relatively high yield strength of about 325 MPa and 305 MPa in the longitudinal and transverse directions, respectively. The data presented here are comparable to the ones reported by McClintock et al. [6] for 12-14YWT ODS steels.

The most evident differences between the two investigated directions, due to anisotropy of the microstructure after hot rolling, are emphasised in the uniform and total elongation of the material (Fig. 4). The uniform elongation of the 14Cr ODS steel in the longitudinal direction continuously decreases with increasing testing temperature (Fig. 4(a)). Note that from room temperature up to 300 °C the uniform elongation decreases by about 30%. The uniform elongation seems to be less affected by a further increase in temperature up to about 600 °C. Above 600 °C the uniform elongation decreases more significantly with increasing temperature. At the highest testing temperature of 750 °C the uniform elongation in the longitudinal direction was measured to be about 2.5%.

In the transverse direction, however, one observes a totally different behaviour of the uniform elongation. At room temperature the uniform elongation is more than two times lower than in the longitudinal direction. As the testing temperature increase the uniform elongation increases continuously, and from about 550 °C the alloy exhibits a higher uniform elongation in the transverse direction than in the longitudinal one.

In spite of the high strength of the 14Cr ODS steel at room temperature the material exhibits a good total elongation of about 4.5% and 10.8% in the transverse and longitudinal directions, respectively (Fig. 4(b)). The total elongation first decreases with increasing temperature, slightly in the transverse direction and more

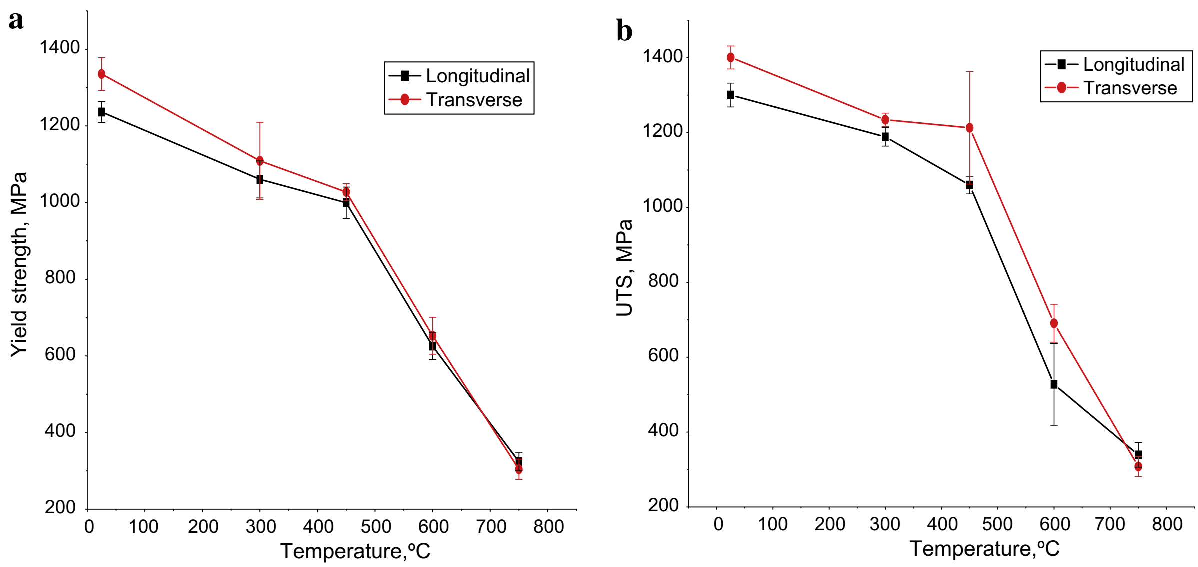

Fig. 3. Yield strength (a) and ultimate tensile strength (b) of the 14Cr ODS ferritic steel vs. test temperature.

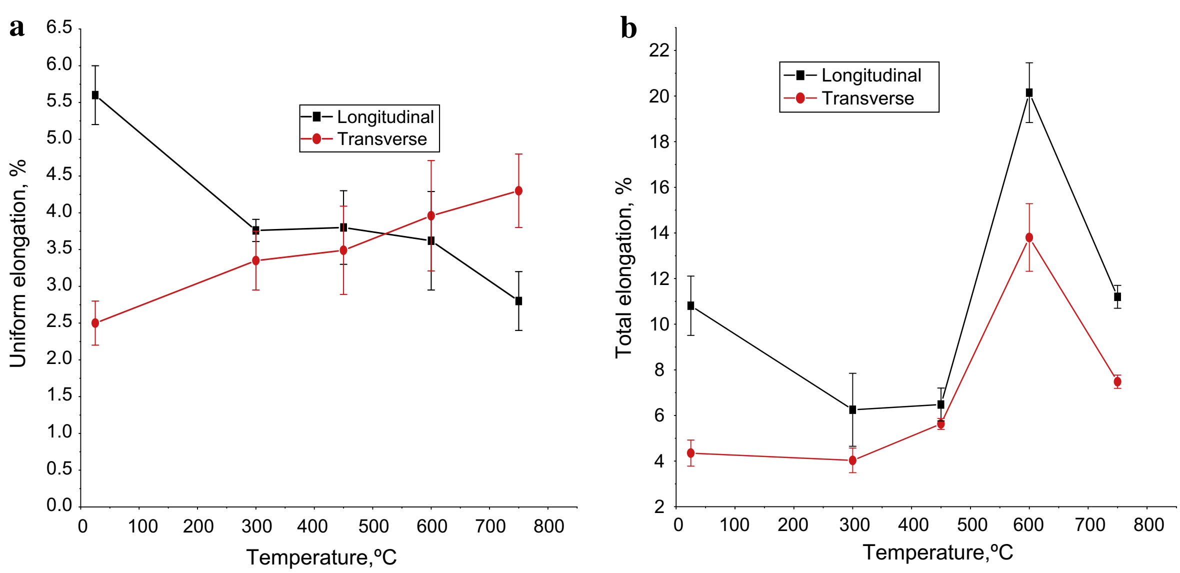

Fig. 4. Uniform elongation (a) and total elongation (b) of the 14Cr ODS ferritic steel vs. test temperature.

significantly in the longitudinal direction, and then it increases with temperature to reach a maximum value of about 20.1% and 13.8% at 600 °C in the longitudinal and transverse directions, respectively. Above this temperature (at 750 °C) the alloy exhibits a decrease of total elongation by about 50% in both directions. As expected from strength results, the total elongation in the longitudinal direction is always larger than in the transverse direction in the temperature range investigated. A similar behaviour was reported by Sokolov et al. [4] and Klueh et al. [9] for the 12YWT ODS steels and by McClintock et al. [6] for 14YWT ODS steel.

Fig. 5 presents the results of Charpy impact tests on the 14Cr ODS steel in both longitudinal and transverse directions. The 14Cr ODS alloy, due to the anisotropy of the microstructure after hot extrusion and hot rolling, exhibits significantly different impact properties in the two perpendicular directions. Relatively low upper shelf energy of about 1.9 J and DBTT of about −7.0 °C were measured in the transverse direction. In the longitudinal direction larger upper shelf energy and DBTT values, of about 4.2 J and 8.8 °C, respectively, were measured. These values are slightly disappointing. However, the DBTT of that steel is significantly better than that of the first batch of the 12YWT ODS ferritic steel, while the DBTT of the improved 14YWT ODS steel, as it was reported by McClintock et al. [6], is about −150 °C.

It should be mentioned that the relatively low upper shelf energy obtained in this work may result from the use of a rectangular die for hot extrusion. As reported by Leblond et al. [10], a rectangular die shape, as compared to a cylindrical one, has a detrimental influence on the material flow during plastic deformation. According to Leblond et al., a Fe–14Cr–1W ODS ferritic steel extruded through a cylindrical die exhibits a twice larger upper shelf energy than the same material extruded through a rectangular die.

SEM images of the fracture surface of KLST Charpy impact specimens tested in both directions are presented in Fig. 6. Transgranular mode of fracture proceeds over the whole range of impact test temperatures. Typical brittle fracture mode can be seen at low test temperatures (−100 °C, for instance) in both directions (Fig. 6(a)

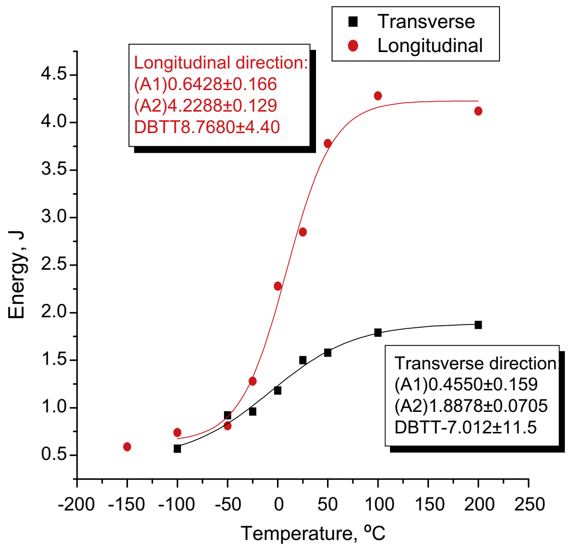

Fig. 5. Results of Charpy impact tests on the 14Cr ODS ferritic steel in the two perpendicular directions.

and (c)). The fracture mode changes from brittle to ductile with increasing the test temperature. Small areas of cleavage fracture can be also seen in both directions in the upper shelf energy region (200 °C, for instance) (Fig. 6(b) and (d)).

Cracks parallel to the rolling direction were also observed. At low magnification this phenomenon looks like delamination of the material into several zones, the thickness of a given zone reaching a few 100 µm. This seems to indicate that after hot rolling and heat treatment the microstructure consists of layers bounded by precipitates (impurities) located along the grain boundaries and aligned along the rolling direction. This phenomenon was observed in the whole range of test temperatures, in both directions, but it seems to be more extensive in the case of the longitudinal one.

It is well known that the fracture toughness of steels is not only affected by impurities (oxygen, carbon and nitrogen) content but also by the microstructure (grain size and shape, dislocation density, etc.) and fabrication route of material [11]. For example, with decreasing the grain size the upper shelf energy of steels increases and the ductile-to-brittle transition temperature decreases. On the other hand, hot extrusion and hot rolling result in heterogeneous microstructure and non uniform chemical composition, which causes a substructure of precipitates and impurities that enhances brittle fracture mode. So, great attention has to be put on better understanding and controlling the manufacturing process and conditions, such as: (i) the extrusion temperature and die shape, (ii) the temperature and degree of plastic deformation (reduction-in-thickness by pass) during hot rolling, and (iii) the parameters of the final heat treatment.

On the basis of the results presented in this paper it is clear that relatively high hardness values (related to the large dislocation density), non-uniform chemical composition (Ti and Al oxides impurities decorating grain boundaries), as well as parameters of fabrication route (shape of the extrusion die) had a detrimental influence on the impact properties of the manufactured 14Cr ODS ferritic steel.

4. Conclusions

A Fe–14Cr–2W–0.3Ti–0.3Y_2O_3 ODS ferritic steel was manufactured by mechanical alloying, hot extrusion, hot rolling and heat treatment. The microstructure is composed of grains that appear elongated in the rolling direction and equiaxed in the transverse direction. The microstructure also contains a high dislocation density and a high density of Y–Ti–O nanoclusters. Coarse particle, which were identified as Ti and/or Al oxides, were also observed in both directions. These particles are located at the grain boundaries of the alloy.

At room temperature the ODS steel exhibits a high ultimate tensile strength of about 1420 MPa and a high yield strength of about

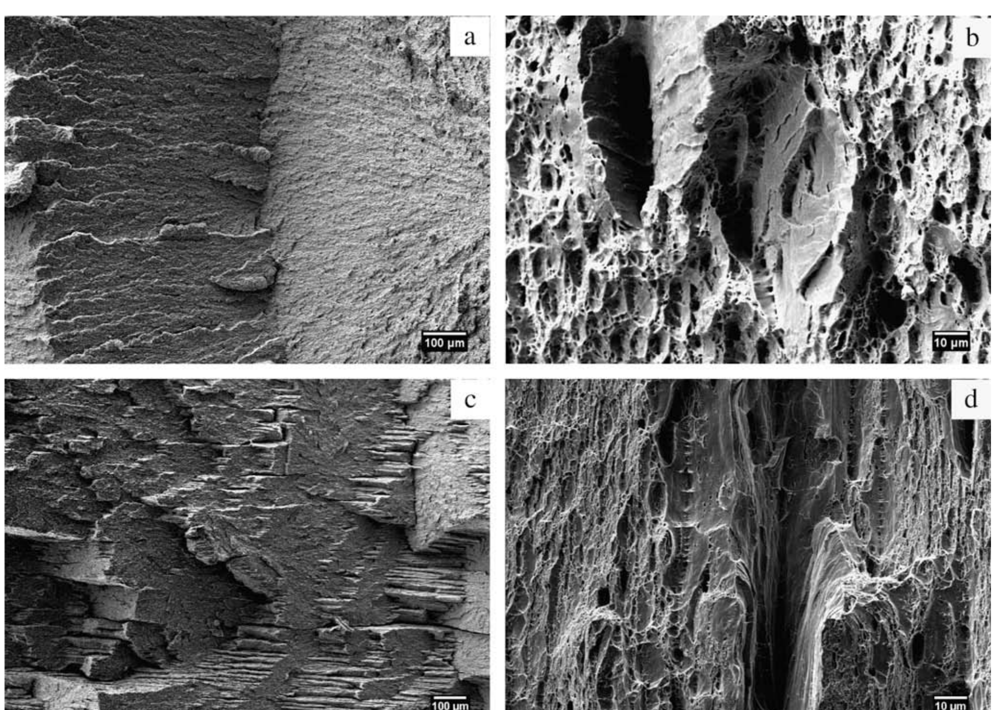

Fig. 6. Fracture surface of 14Cr ODS KLST specimens tested in (a) and (b) the longitudinal direction and in lower and upper shelf energy regions (−100 °C and 200 °C), respectively, and tested in (c) and (d) the transverse direction and in lower and upper shelf energy regions (−100 °C and 200 °C), respectively.

1340 MPa in the transverse direction. In the longitudinal direction these values are about 10% lower. Both the ultimate tensile strength and the yield strength decrease as the temperature increases and this decrease in strength is accompanied by an increase in total elongation, at least at temperatures above 300 °C. At the highest test temperature of 750 °C the material exhibits relatively high yield strengths of about 325 MPa and 305 MPa in the longitudinal and transverse directions, respectively. The uniform elongation of the material decreases continuously with increasing temperature in the longitudinal direction, whereas in the transverse direction a continuous increase in uniform elongation with temperature takes place.

The material exhibits upper shelf energy of about 1.9 J and a DBTT of about −7.0 °C in the transverse direction, while it exhibits upper shelf energy of about 4.2 J and a DBTT of about 8.8 °C in the longitudinal direction.

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