**ON THE ANISOTROPY OF THE DUCTILE TO BRITTLE TRANSITION BEHAVIOR IN A WROUGHT
AND IN TWO OXIDE DISPERSION STRENGTHENED FeCrAl STEELS**

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ABSTRACT

The directionality of the ductile-brittle transition behavior of two oxide-dispersion - strengthened (ODS) FeCrAl steels with similar chemical compositions, tensile properties and sub-micrometric grain sizes but different processing routes, and a Zr-particle strengthened FeCrAl steel manufactured by high vacuum melting is discussed. Despite the similarities of the ODS FeCrAl steels, strong differences in the lower and upper shelf energy and the ductile to brittle transition temperature were observed for longitudinal through thickness notched specimens. Although the lower and upper shelf energies of longitudinal surface notched specimens of ODS FeCrAl steels are similar, a strong difference in the ductile-to-brittle transition temperature is observed. For through-thickness notched and surface notched specimens taken transversely, the analyzed ODS FeCrAl steels show a similar ductile-to-brittle behavior. In general, the FeCrAl alloy strengthened with Zr-particles presents a more isotropic behavior and a higher ductile-to-brittle transition temperature than the ODS FeCrAl steels. In addition, the upper shelf energy of the FeCrAl steel strengthened with Zr-particles is significantly higher than that of the ODS FeCrAl steels.

Keywords: ductile–brittle transition behavior; delamination; mechanical alloying; oxide dispersion strengthened ferritic alloy

1. INTRODUCTION

FeCrAl steels are a subclass of ferritic stainless steels containing 15-25wt% Cr and 4-7wt% Al. These materials are usually prepared by melting and casting under high vacuum conditions. According to the phase diagram, the microstructure is ferritic at all temperature ranges. The body-centered-cubic crystalline structure of ferrite makes the ductile-to-brittle transition to decrease with increasing the loading rate and/or increasing stress triaxiality stress state. These steels are not employed for structural purposes but for high-temperature applications requiring excellent resistance to oxidation e.g., for heating-resistant elements of furnaces or for construction materials in the core of fast neutron reactors.

ODS FeCrAl steels, in contrast to FeCrAl steels are manufactured by mechanical alloying of yttria (Y_2O_3) nano-particles and elementary powders of Fe, Cr and Al elements. These steels are considered among the most promising structural steels for the nuclear industry because they combine acceptable creep strength and excellent swelling resistance in supercritical pressurized water, as a consequence of the nano-scale yttria particles dispersed in the FeCrAl matrix.^[1] However, for full structural applications, further improvements in material processing are necessary to enhance their fracture toughness. Hot-extruded bars with sub-micrometric grain size present strong anisotropy in fracture toughness with a very low upper-shelf energy level, such as 0.30 J, for transverse specimens^[2-3]. Fracture toughness can be improved by eliminating the stringer-like alumina inclusions in the source powders^[2]. Moreover, to reduce the fracture toughness anisotropy, the presence of bundles of small grains with segregation/precipitation/inclusion along the extrusion direction should be

minimized^[3]. While these measures will effectively contribute improve fracture toughness, the causes of the poor fracture toughness in the transverse direction remain unclear.

The goal of this study is to understand the causes of both the strong anisotropy in the fracture toughness and the very low upper shelf energy (USE) for transverse specimens in ODS FeCrAl steels. Accordingly, this paper examines the fracture behavior of longitudinal and transversely taken specimens of two ODS FeCrAl steels with similar chemical compositions and microstructures but with slight differences in the grain elongation and texture. These ODS steels were manufactured via mechanical alloying by different manufacturers. The behaviors of these ODS steels were compared with that of a FeCrAl steel of similar chemical composition that was manufactured via melting and casting under high vacuum conditions and strengthened with Zr-rich particles.

2. MATERIALS

The chemical compositions of the three steels supplied, as determined by X-ray fluorescence spectrometry, wet chemistry, colorimetry and inert gas fusion techniques are listed in Table 1. According to their chemical composition, these materials consist of ferrite solid-solutions with a body-centered-cubic crystalline structure. Moreover, the embrittling effect of C, N interstitials is controlled by additions of Ti for the ODS FeCrAl steels and Zr for the FeCrAl steel. Both Ti and Zr are strong carbide and nitride formers, and the resulting carbides and nitrides are more stable than chromium carbide and chromium nitride^[4].

The PM2000 steel was provided by the Plansee Group (Reutte, Austria) in the form of a hot-rolled tube with a diameter of 100 mm and thickness of 7.9 mm. The material processing involved mechanical alloying of elemental Fe, Cr, Al, Ti powders with 0.5 wt.% Y₂O₃ particles in a shielding atmosphere of H₂, encapsulation of the powder, evacuation of the capsule and hot-

isostatic pressing at 1050 °C; hot-rolling/hot-extruding at 1050 °C and air cooling to room temperature.

The MA956 alloy was provided by Special Metal Corporation (Hereford, UK) in the form of a hot-extruded bar with a diameter of 60 mm. The material processing involved mechanical alloying of elemental Fe, Cr, Al, Ti powders with 0.5 wt.% Y₂O₃ particles in a shielding atmosphere of Ar, encapsulation of the powder, evacuation of the capsule and hot-isostatic pressing at 1050 °C; hot-extrusion at 1050 °C and air cooling to room temperature.

The FeCrAl alloy was provided by Goodfellow Cambridge Ltd. (Huntington, UK) in the form of a temper-rolled square sheet with a thickness of 5 mm and side length of 200 mm. The material processing involved melting and casting under high vacuum, hot forging and finally temper-rolling. It was initially assumed that the rolling direction was parallel to the watermarks on the sheet surface.

3. EXPERIMENTAL PROCEDURE

Before machining the specimens for mechanical testing from the FeCrAl steel, a 0.5 mm-thick layer was removed from both surfaces of the sheet so that the microstructure and texture were homogeneous in the cross-section of the specimens. Tensile specimens with a gauge of 25 mm in length, 4 mm in width and 3.5 mm-thick were machined such that the tensile axis was parallel to the longitudinal (L), diagonal (D) and transverse (T) directions of the sheet, Figure 1(c). The specimens were tested at a range of temperatures between -196°C and 300°C using a crosshead speed of 0.0125 mm/s.

Fig. 1. Orientation of the bending notched specimens relative to the product shape. a) PM2000 steel b) MA956 steel c) FeCrAl steel. 'L', 'T', 'R', 'S' and 'D' indicate longitudinal, tangential (PM2000 and MA956 steels) or transverse (FeCrAl steel), radial, short and diagonal directions respectively. The orientations of the bending specimens are designated according to ASTM standard^[5] as 'LT', 'TL', 'TR', 'LS', 'TS', 'DD' and 'DS' in which the first letter indicates the specimen direction and the second the loading direction.

97

98 Single-edge notch bend specimens (4 mm in width; 3 mm in thickness; 27 mm in length; 1 mm
 99 in notch depth; 0.1 mm in notch tip radius; and 60° notch angle) were machined from the as-
 100 received materials with four notch positions for the PM2000 and MA 956 steels and six for the
 101 FeCrAl steel (Figure 1). The orientation of the notch was defined according to ASTM Standard
 102 E399 using a two-letter code^[5]. The first letter designates the normal direction to the notch
 103 plane, and the second the expected direction of the crack propagation. The notch orientations
 104 included LT, LR, TL and TR for the PM2000 and MA956 steels (Figure 1(a) and Figure 1(b),
 105 respectively) and LT, LS, DD, DS, TL and TS for the FeCrAl steel (Figure 1(c)). The tests were
 106 performed using the bending test fixture shown in Figure 2. The specimens were tested at a
 107 range of temperatures between -196°C and 300°C using a crosshead speed of 1 mm/s. The
 108 fracture toughness was estimated by integrating the area under the load-displacement curve
 109 up the point of failure.

110 Figure 1 shows a schematic representation of the criteria used for defining the transition
 111 temperature. It is commonly accepted that the USE is the average energy value for all

specimens whose test temperature is above the upper end of the transition temperature, T1. Above this temperature, fracture is mainly ductile. This T1 temperature is named fracture transition temperature (FTP). This is the temperature at which fracture changes from almost totally ductile to substantially brittle, or as in the case of PM2000 and MA956, delaminations appear, on decreasing temperature. Problem of brittle fracture is almost negligible above this temperature. In contrast to the upper self, the lower shelf is the region in which the temperature is lower than the lower transition temperature T2. This T2 temperature is named as nil ductility temperature (NDT), and is defined as the temperature below which a material fractures under cleavage mechanisms. On the basis of this the ductile-to-brittle transition temperature (DBTT) was estimated as the test temperature corresponding to the average value of upper shelf energy (USE) and lower shelf energy (LSE) of the energy vs temperature curve.

Fig. 2. Experimental setup of the bending test and criteria for defining transition temperature.

The microstructure of the FeCrAl steel was examined in terms of grain morphology, grain size, and second-phase particles using optical microscopy. The three orthogonal surfaces of the sheet LT, LS and TS (Figure 1(c)), were prepared by conventional metallographic techniques including grinding, polishing and etching with an acid solution of 20% HNO_3 , 30% HCl , 20% $\text{C}_2\text{H}_4\text{O}_2$ and 30% CH_4O .

The texture of FeCrAl steel was determined by X-ray diffraction (XRD) in a Bruker AXS D8 (Bruker GmbH, Karlsruhe, Germany). Texture analysis of PM2000 and MA956 has been described elsewhere in Ref. [7] and Ref. [11]. The diffraction studies were performed with $\text{Co-K}\alpha$ radiation on the TS cross section of the sheet, Figure 1(c). SEM characterization was performed in a Hitachi S 4800 J field emission gun scanning electron microscope (FEG-SEM) operating at 10 kV.

4. EXPERIMENTAL RESULTS

Microstructure

Previous characterization of the microstructure of the PM2000 steel revealed a bamboo grain structure with an equiaxed dimension ($\sim 0.7 \mu\text{m}$) in the transverse (TR) cross-section of the tube but significant elongation ($\sim 1.6 \mu\text{m}$) in the longitudinal (LR) cross-section (Figure 3(a))^[11]. Spherical particles of yttria ($f_{\text{vol}} = 1.3\%$) with sizes ranging from 3 nm to 40 nm are mainly located at the grain boundaries.^[7] Al_2O_3 and Ti(C,N) particles ($f_{\text{vol}} \approx 0.12\%$) up to 100 nm in size were also found^[6].

The microstructure of the MA956 steel, presented a similar morphology to that of the PM2000 steel, with equiaxed grains in the transverse (TR) cross section ($\sim 0.7 \mu\text{m}$) but lower elongation ($\sim 0.9 \mu\text{m}$) in the extrusion direction (Figure 3(b))^[7]. Two main distributions of spherical particles of yttria ($f_{\text{vol}} \approx 1.1\%$) were observed: small particles with sizes from 5 nm to 40 nm and

large particles up to 500 nm^[8]. Al₂O₃ particles 100 nm in size and Ti(C,N) particles up to a few μm in size were also found^[9]. The volume fraction of Al₂O₃ and Ti(C,N) particles was approximately 0.15%.

The microstructure of the FeCrAl alloy (Figure 3(c)) was inhomogeneous throughout the thickness of the sheet. At the center layer, the microstructure of the grains was partially recovered, whereas near the surfaces, some recrystallized grains were apparent. Moreover, the average grain size near the surface at the three orthogonal planes was slightly higher than in the center layer. The morphological analysis of the microstructure revealed that the grains were ellipsoidal in shape with a maximum dimension of 29 μm at the LT plane, followed by 22 μm at the LS plane and 17 μm at the TS plane. Because Zr has a strong chemical affinity with oxygen, nitrogen, sulfur and carbon, and the Zr content of the alloy is considerably higher than its solubility limit in ferrite, second-phase particles in the form of ZrO₂, ZrC and Zr(C,N) were expected. The presence of sub-micrometric spherical particles of ZrO₂ in low-carbon steels has been reported^[10]. These particles would restrict the growth of the grains at high temperatures, resulting in a fine grain microstructure at room temperature, which is beneficial for imparting good mechanical properties to the material. In the present paper, the presence of second phase particles was analyzed in 40 fields (x500 magnification) randomly taken from the three orthogonal planes of the sheet. Carbides and carbo-nitrides of zirconium were observed (Figure 4). The particles typically have a cubic or rectangular-prism morphology and a yellow color under optical microscopy, but do not present a preferred orientation, i.e. the Zr-particles does not follow an alignment along extrusion direction such as the one followed by yttria particles has on PM2000. The average size of the particles is 2.4 μm, with maximum and minimum values of 8 μm and 1 μm, respectively. Table 2 summarizes the microstructural features of the alloys.

Fig. 3. Back-scattered SEM (PM2000 and MA956 steels) and optical (FeCrAl steel) images of the microstructures of (a),(c) and (e) transverse, and (b), (d) and (f) longitudinal cross sections of the PM2000 steel, MA956 steel and FeCrAl steel. The white arrow indicates the longitudinal direction. ((a)After [11], (b) after [7]).

Fig. 4. Typical Zr-rich particles of the FeCrAl steel and EDS results for two particles on ductile fracture surface of FeCrAl wrought steel sample tested at 155 °C.

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177 Texture

Figure 1 indicates the ratio between the directions for sample machining and texture measurements. In this sense, RD stands for rolling (or extrusion) direction, ND for normal direction and TD for tangential direction. The section of the orientation distribution function (ODF) obtained from a transverse cross-section of the PM2000 steel tube shown in Figure 5(a) reveals a strong incomplete α -fiber (RD||<110>) texture in which the {001}<110> orientation is dominant^[11]. A less intense {111} <011> orientation is also detected as it was previously reported^[11].

The section of the ODF obtained from a transverse cross section of the MA956 steel bar shown in Figure 5(b) reveals a strong α -fiber (RD||<110>) texture^[11]. Texture gradients along the radial direction are only observed very close to the surface^[11]. Moreover, a secondary {111} <011> orientation is detected, which is slightly stronger than that of PM2000 steel as it was previously reported^[11].

Sections of the ODF in the center of the thickness of the FeCrAl steel sheet and near the sheet surface are shown in Figure 5(c) and Figure 5(d), respectively. The texture in the center (Figure 5(c)) is a combination of an incomplete, broadened and weak α -fiber and a slightly less intense {332}<113> orientation, whereas near the surface (Figure 5 (d)), the texture is a combination of a weak Goss orientation ({011}<100>) and a slightly less intense {001}<110> orientation. The purpose of Figures 5(c) and 5(d) is to illustrate the texture gradient through the thickness of the FeCrAl sheet. However, as it was mentioned before, during machining of mechanical test samples it was removed a 0.5 mm-thick layer from both surfaces of the sheet in order to remove the heterogeneity in texture, so that the microstructure and texture were homogeneous in the cross-section of the specimens. The last column of Table 2 summarizes the main components of the texture of the steels.

Fig. 5. Orientation distribution function ($\phi_2 = 45^\circ$ section) of the (a) PM2000 steel, (b) MA956 steel, (c) FeCrAl steel (center of the thickness), and (d) FeCrAl steel (surface). ((a) and (b) After [11])

Mechanical testing

Tensile properties. Table 3 summarizes the tensile properties as a function of the test temperature for longitudinal (L) specimens of the PM2000^[7] and MA956^[11] steels and longitudinal (L), transverse (T) and diagonal (D) specimens of the FeCrAl steel. The strengths of the two ODS FeCrAl steels are similar to each other but considerably higher than that of the FeCrAl steel. The uniform elongation and strain hardening rate are similar for the three steels but the total elongation of the FeCrAl alloy is significantly higher than those of the ODS FeCrAl steels. Reducing the test temperature significantly increases both, the strength and the ductility and moderately decreases the strain hardening rate.

In the case of the FeCrAl steel, the specimens tested at a temperature of -196 °C, broke before reaching the yield strength (YS) of the material. The YS and ultimate tensile strength (UTS) are highest for specimens taken in the T direction. Moreover, the differences in YS and UTS when changing the tensile direction are small and can be attributed to the material texture^[12-13].

Although the elongation is slightly lower for the transverse specimen than for the longitudinal and diagonal specimens, the strain hardening rate is nearly identical in the L, D and T directions.

In relation to the anisotropy in tensile properties of the ODS FeCrAl steels, previous studies on hot-extruded bars manufactured by mechanical alloying, have shown that uniform and total elongations in the tangential direction are about 20% lower than in the longitudinal direction, whereas the YS is about 5% higher^[2,3], which is in good agreement with the above results for the FeCrAl steel.

Bending tests. The DBT curves of the PM2000 steel are shown in Figure 6(a), and the corresponding values of LSE, USE and DBTT are summarized in Table 4. The through-thickness notched specimens (LT and TL specimens) exhibits lower USE values than the corresponding surface notched specimens with LR and TR specimens. The DBTT values for the longitudinal specimens (LT and LR specimens) are lower than those of the tangential (TL and TR samples) specimens. The DBT curve for the LR sample presents a peak at a temperature of 60°C.

The DBT curves of the MA956 steel are shown in Figure 6(b), and the corresponding values of LSE, USE and DBTT are presented in Table 4. The tangential specimens with the TL and TR notch positions exhibit lower USE values than the corresponding longitudinal specimens with LT and LR notch positions. This difference is particularly striking for the through-thickness notched (TL and LT specimens) specimens. The DBTTs of the longitudinal (LT and LR notch positions) specimens are lower than those of the tangential (TL and TR specimens) specimens. The DBT curve for the LR notch position presents a peak at -25°C.

The DBT curves of the FeCrAl steel are shown in Figure 6(c) and the corresponding values of LSE, USE, and DBTT are presented in Table 4. The LT, TL and DD samples exhibit nearly identical DBT behaviors i.e., regarding the fracture behavior of the through-thickness notches, the material is isotropic. The USE values of the surface notches (LS, TS and DS specimens) are significantly higher than those of the through-thickness notches. Moreover, the LS sample exhibits the highest USE value but, in contrast to the PM2000 and MA956 steels, presents no peak in the USE region of the DBT curve. The LSE and DBTT values of all analyzed orientations are nearly identical.

Fig. 6. Effect of specimen orientation on the ductile-to brittle transition curve of (a) PM2000 steel (b) MA956 steel (c) FeCrAl steel.

Fracture analysis of bending specimens

PM2000 steel. For the LT sample at a test temperature of -196°C , fracture occurred by transgranular cleavage along a plane inclined between 30° and 45° relative to the notch plane (Figure 7(a)). When the regions near the initiation sites were studied at successively higher magnifications the microstructural feature triggering the fracture process could not be detected (zoom in Figure 7(a)).

From -60°C to 200°C the fracture occurred along the notch plane by coalescence of microvoids with the presence of crack divider delamination (Figure 7(b)). The crack divider delamination leads to a decrease in the triaxiality of the stress state of the notched specimens, and thus, to a

decrease in both the DBTT and USE values^[14]. The number of delaminations decreases as the test temperature is increased. At a test temperature of 300 °C the fracture morphology is fully ductile.

For the TL sample at a test temperature of -196 °C, intergranular brittle fracture along the notch plane was mostly observed (Figure 7(c)). From test temperatures of -60 °C to 200 °C, the area fraction of the main fracture surface occupied by ductile features increases as the test temperature is increased. Within this temperature interval, the delamination number (crack divider type) decreases as the test temperature increased. At a test temperature of 300 °C, the fracture morphology is fully ductile.

For LR and TR samples and test temperatures ranging between -196 °C and room temperature, areas of intergranular brittle fracture interspersed with ductile steps are observed on a plane inclined approximately 70° relative to the notch plane (Figure 7(d)).

For the LR sample and test temperatures between room temperature and 100°C strong delamination parallel to the rolling plane of the crack arrester type is observed (Figure 7(e)). At higher test temperatures, only incipient crack arrester delamination is observed in the notch tip. This delamination type entails bifurcation of the main fracture along the delamination plane and restarting of the fracture process in the actual unnotched ligament. As a result, enormous energy consumption can occur during the bending test, particularly in the upper shelf region of the DBT curve^[14]. At test temperatures other than -196°C no delamination was observed for the TR notched specimens.

Figure 7(f) shows a schematic illustration of the splitting in both LT and LR samples after Kimura et al. ^[15], with the corresponding orientation of TR plane regarding the L, T and R system (Figure 1). In a Charpy test, the main stress component after the strike is σ_y . The other two stress components, σ_x and σ_z , are generated by the localized plastic constrain at the notch and/or the crack tip and of lower magnitude than σ_y . The splitting in LT and LR samples could

be explained on the basis that the plastic deformation in R-direction is considerably higher than in T-direction. Therefore, the plastic constrain in R-direction would lead to an increase of σ_x in the case of LT samples, and of σ_z in the case of LR sample that would cause the distribution of cracks found experimentally. Those delaminations are similar to these found under crack-divider and crack-arrester basic geometries.

Fig. 7. Selection of electron fractographs showing the relevant aspects of the fracture

surfaces of single notch bend specimens of the PM2000 steel: (a) initiation of cleavage fracture (left) and details of the fracture initiation zone (right) of the LT specimen tested at -196oC; (b) microvoid coalescence zones bounded by crack divider delamination of the LT specimen tested at -60oC; (c) zones of intergranular brittle fractures bounded by crack divider delamination of the TL specimen tested at -196oC; (d) fracture initiation (left) and details (right) of the intergranular brittle fracture of the LR specimen tested at -70oC; (e) fracture initiation by microvoids coalescence followed by various crack arrester delamination and final intergranular brittle fracture of the LR specimen tested at 80 °C; and (f) schematic illustration showing delaminations geometries found in LT and LR samples tested according with Cartesian x,y,z and L,T,R (Figure 1(a)) reference systems. The white arrows indicate the initiation point of the fracture.

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287 *MA956 steel.* Similar to the PM2000 steel, for the LT sample at -196 °C, fracture occurs by
 288 transgranular cleavage along a plane inclined between 30° and 45° relative to the notch plane
 289 (Figure 8(a)). In this case, however, the microstructural feature triggering the fracture process
 290 was shown to correspond to the tip of a crack divider delamination. From -60 °C to -20 °C,
 291 fracture occurs along the notch plane by coalescence of microvoids with the presence of
 292 delamination of the crack divider type (Figure 8(b)). In this steel, however, the delaminations
 293 were more irregular and far less numerous than in the PM2000 steel. From room temperature
 294 to 300 °C the fracture morphology was fully ductile, without delamination.

295 For the TL sample and temperatures ranging between -196 °C and -20 °C the fracture
 296 morphology is mostly brittle intergranular (Figure 8(c)). At test temperatures of -20 °C to
 297 300 °C, the area fraction of the main fracture surface occupied by ductile features increases as
 298 the test temperature is increased. At test temperature of 300 °C, the area fraction of the
 299 fracture surface occupied by intergranular fracture is approximately 50 %. No delamination
 300 was observed for the TL sample.

301 In contrast to the PM2000 steel, for LR specimens at a test temperature of -196°C, fracture
 302 occurs by transgranular cleavage along a plane inclined between 30° and 45° relative to the
 303 notch plane. Specimen fracture nucleated in a Ti-rich particle located very near the notch tip.
 304 As the test temperature increases from -70 °C to 300 °C, the area fraction of the main fracture

surface occupied by ductile features increases. From -70 °C to -20 °C strong delamination parallel to the rolling plane of the crack arrester type is observed (Figure 8(d)).

For the TR specimens and a test temperature of -196 °C, intergranular brittle fracture along the notch plane is observed. As the test temperature is increased from -70 °C to 300 °C the area fraction of the main fracture surface occupied by layers of ductile features increases. At a test temperature of 300 °C, the area fraction of the fracture surface occupied by intergranular fracture is about 30 %. From -70 °C to room temperature, small delamination parallel to the rolling plane of the crack arrester type, is observed (Figure 8(e)).

Fig. 8. Selection of electron fractographs showing the relevant aspects of the fracture surfaces of single edge notch bend specimens of the MA956 steel: (a) location of the initiation point (left) and detail (right) of the cleavage fracture at the end of a crack divider delamination of LT specimen tested at -196°C; (b) microvoid coalescence zones bounded by crack divider delamination of the LT specimen tested at -60°C; (c) zones of intergranular brittle fracture interspersed with thin layers of ductile fracture of the TL specimen tested at -196°C; (d) microvoid coalescence zones bounded by crack divider delamination of the TL specimen tested at -60°C; (e) zones of intergranular brittle fracture interspersed with thin layers of ductile fracture of the TR specimen tested at -196°C.

room temperature; (d) fracture initiation by microvoids coalescence followed by crack arrester delamination and final intermixed zones of cleavage and ductile fracture modes of the LR specimen tested at -20°C; (e) intermixed zones of intergranular brittle and ductile fracture modes with occasional crack arrester delamination of the TR specimen tested at -20°C. The white arrows indicate the initiation point of the fracture.

313

314 *FeCrAl steel*. For any notch orientation and for test temperatures ranging from -196°C to -20°C,
315 the main fracture surfaces revealed mostly transgranular cleavage (Figure 9(a) and Figure
316 9(b)), where fracture occurs along of the notch plane. The initiation point that caused the final
317 failure of the specimen was located by tracing back the path of river lines on the fracture
318 surface. In all cases, fracture initiated from ZrC or (C,N)Zr particles near the notch tip (Figure
319 9(a) and Figure 9(b)). At a test temperature of -196°C, more than one initiation point near the
320 notch tip was observed, whereas at the other test temperatures, the fracture initiated at a
321 single initiation point.

322 As the test temperature is increased, the fracture mode gradually varies from cleavage
323 fracture to dimpled fracture. At test temperatures higher than 150 °C, the fracture surface is
324 fully ductile, regardless of the notch orientation (Figure 9(c) and Figure 9(d)). The ductile
325 fracture nucleated in all cases from ZrC or (C,N)Zr particles.

326 The fracture surfaces of through-thickness notched (LT, TL and DD) specimens tested at
327 temperatures ranging from -60 °C to 150 °C exhibit delamination of the crack divider type,
328 although to a much lesser extent than that observed for the LT and TL notched specimens of
329 the PM2000 and MA956 steels. Within the same temperature range, the LS, TS and DS notched
330 specimens exhibited one or two delaminations of the crack arrester type (Figure 9(e) and
331 Figure 9(f)) similar to the LR and TR samples of the PM2000 and MA956 steels.

Fig. 9. Selection of electron fractographs showing the relevant aspects of the fracture surfaces of single edge notch bend specimens of the FeCrAl steel: (a), (b) initiation of cleavage fracture in Zr-rich particles of the LT specimen tested at -20°C and TL specimen tested at 60°C respectively (see the arrow); (c), (d) macroscopic image of ductile fracture with crack divider delamination and detailed micrograph showing ductile fracture nucleation in Zr-particles of LT specimen tested at 150°C ; (e), (f) fracture initiation by coalescence of microvoids followed by crack arrester delamination and final cleavage fracture with small areas of ductile fracture of the DS specimen tested at 80°C and the TS specimen tested at 60°C respectively. The white arrows indicate the initiation point of the fracture.

5. DISCUSSION

The ductile-to-brittle transition in ferritic steels is associated with two different failure modes.

At high temperature, in the USE region of the DBT curve, ductile fracture occurs by nucleation

and coalescence of microvoids, thus producing ductile tearing. This process requires extensive plastic deformation, and therefore a large amount of energy is consumed by the specimen during fracture. Void nucleation and growth are determined by the combination of stress triaxiality state and plastic strain produced by void nucleation and growth^[16]. The most important microstructural parameter controlling the ductile fracture is the volume fraction of the second-phase particles nucleating microvoids^[17]. The DBT is a result of competition between plastic flow and cleavage or intergranular fracture and can be defined as the point where the yield stress equals the cleavage stress or intergranular fracture stress. In the case of ferritic steels of similar chemical composition, the crystallographic grain size is the most important microstructural parameter controlling the DBTT in the case of cleavage-to-ductile transition^[18], whereas in the intergranular-to-ductile transition case, the amount of segregated impurities, the orientation of the long dimension of the grain relative the applied stress, and the grain boundary morphology are the most important parameters^[19,20]. Thus, when cleavage occurs, the DBTT is different from that when intergranular brittle fracture occurs. The DBTT in the case of cleavage fracture is usually considerably lower than in the case of intergranular fracture^[19]. The stress triaxiality state is the main factor controlling the DBTT, in the sense that the DBTT increases with increasing the stress triaxiality, while the USE decreases^[21]. In this regard, the residual strain, i.e., the residual elastic strain remain stored in the material in the end of its manufacturing process, may affect the DBTT by changing the constrain conditions under which fracture occurs in the lower transition region^[22]. The effect of the residual strains is to increase the stress triaxiality state and then to decrease the fracture toughness in the lower transition region, however, the fracture toughness remains unaltered in the upper transition region because in this region the residual strains are relaxed by plastic strain.

In the low temperature region, the LSE region, brittle fracture is usually considered to occur by transgranular cleavage, however, intergranular brittle fracture can occur if the grain boundary is weakened by segregation of impurity atoms^[19] or because of the grain morphology^[20]. These

two fracture micromechanisms, cleavage and brittle intergranular, differ significantly. Cleavage fracture occurs by the dynamic propagation of slip-induced microcracks nucleating in a second phase or microstructural heterogeneity^[23]. Cleavage fracture is tensile stress controlled, and the local cleavage stress below the notch tip is independent of the temperature and strain rate for slip-induced cleavage^[24]. Thus, the cleavage strength is an intrinsic property of the material. As in cleavage fracture, intergranular fracture is a tensile stress controlled process^[19], however, the fracture strength of the grain boundary depends on the segregation-induced weakening intensity^[19] and, when the applied stress is normal to the long direction of the grains, on the aspect ratio of the grains, alignment of the grains and the smoothness of the grain boundaries.

[20, 25]

On the other hand, there are various factors which must be taken into account when discussing the anisotropic behavior of DBT: inclusion shape, grain shape, microstructural anisotropy due to chemical segregation with bunching of elongated grain structure, grain boundary alignment, the morphology of grain boundaries and crystallographic texture^[25-28]. The inclusion shape is disregarded in the present study due to the low S content of the studied steels and because the second phase particles found in the microstructural analysis did not present a preferential orientation. The grain shape itself is important because a high aspect ratio promotes material delamination, which is a very important source of anisotropy^[29-31]. Delamination decreases the stress triaxiality state ahead of the notch tip, which in turn produces a decrease in both DBTT and USE values^[27-32]. With respect to material delamination, the chemical segregation to the grain boundary, the grain boundary alignment and the morphology of the grain boundaries are also important promoters^[19, 20, 25]. The crystallographic texture, on the other hand, can affect the morphology of delaminations^[33]. The fiber texture of MA956 means that there is no preferred transverse orientation, and therefore delaminations in the transverse direction are more difficult. In contrast, there is no fiber texture in PM2000, but with some preferred $\langle 110 \rangle$ orientation, which could explain why

is prone for delamination in the transverse direction. Regarding the longitudinal direction, both materials, i.e. MA956 and PM2000 maintain a preferred $\langle 110 \rangle$ orientation, being more intense in PM2000. Again, this is why PM2000 is prone for delaminations.

Comparative analysis of ODS steels. Both ODS steels have similar chemical composition and tensile properties, but the PM2000 steel exhibits grains more elongated with an identical transverse dimension and an incomplete α -fiber in which $(100)\langle 110 \rangle$ is the main orientation, instead of a full α -fiber texture. Thus, no great differences between the parameters of the DBT curves for the longitudinal specimens are expected. However, as shown in Figure 6(a), Figure 6(b) and Table 4, significant differences are observed for the LT and LR specimens. In the case of the LT sample, the differences in USE could be partially justified on the basis of the higher volume fraction of the second-phase particles and, more importantly, by the higher delamination tendency of the PM2000 steel. The higher delamination tendency of PM2000 steel is due to the higher elongation of the grains compared to the MA956 steel. The difference in DBTT is attributed to the definition used for this parameter i.e., the temperature corresponding to the average value of the USE and LSE values. For the LT specimens, at the so defined DBTT, full ductile fracture with delaminations was observed in the case of PM2000 steel whereas a mixture of cleavage and ductile fracture was observed in the case of MA956 steel. Thus, no true transition occurred in the fracture mechanism of the PM2000 steel using the above DBTT definition. Moreover, a second transition in the DBT curve of PM2000 steel (Figure 6(a)) between -60°C and -196°C in which full ductile fracture with delaminations and transgranular cleavage fracture were observed, respectively. Furthermore, this second transition occurred in the same temperature range as the DBT of MA956 alloy. It is therefore suggested that the DBT is similar for the LT notch position of both ODS steels.

In the case of the LR sample, a slight difference in USE value is observed. Moreover, this difference is opposite to that expected on the basis of the difference in the volume fraction of

second-phase particles of both ODS steels. However, a marked difference in DBTT values was observed that could be attributed to the difference in the fracture mechanism in the LSE region: intergranular fracture in the case of PM2000 steel and cleavage fracture in the case of MA956 steel. As noted above, DBTT was considerable higher for intergranular fracture than for cleavage fracture. The energy peak at the beginning of the USE region of the DBT curves is due to the formation of crack arrester delamination due to the enormous energy consumption during the process of delamination → unnotched bending → fracture reinitiation during bending test. The temperature difference at the peak position in the DBT curve is likely related to the different fracture mechanism in the LSE region.

The DBTT of transverse specimens is higher than that of longitudinal specimens, which could be explained partially by the extra elongation of the grains in the longitudinal direction^[27]. However, the differences in the fracture mechanism operating in the DBT region of the longitudinal and transverse specimens revealed that the weakening intensity of the grain boundaries greatly contributed to the differences in DBTT. Except for LT notch position of PM2000 steel, the USE values of the longitudinal specimens of both steels were significantly higher than the transverse USE values, which could be due to the presence of a mix of intergranular and ductile features in the USE region of transverse specimens.

Significant differences in the USE values for the TL and TR specimens in both steels were observed. The difference in USE values for the TL and TR specimens could be justified in the case of the PM2000 steel by the presence of crack divider delamination in the TL-notched specimens; however, in the case of MA956 steel no delamination were observed for TL-notched specimens. One may speculate that the presence of crack arrester microdelamination in TR-notched specimens could contribute to a higher absorbed energy as compared to the TL-notched specimens.

Comparative analysis of ODS and FeCrAl steels. Comparing the results for the FeCrAl steel with those for the ODS FeCrAl steels (Figure 6 and Table 4) the great in-plane-isotropy of the DBTT of FeCrAl steel is notable. The USE values of the through thickness notched (LT, TL and DD) specimens can also be considered isotropic in the sheet plane. Greater differences in USE values were observed between through-thickness notched specimens and surface-notched specimens and between surface-notched specimens of different orientations, which could be attributed to incipient crack arrester delaminations of the surface notched specimens.

In general, the USE values of the FeCrAl steel are higher than those of the ODS FeCrAl steels, mainly due to the low volume fraction of second-phase particles (one order of magnitude lower) of the FeCrAl steel, but also to the difference in UTS values and to the delamination propensity of ODS steels. Figure 10 shows the difference in dimple size between the FeCrAl steel and the ODS steels. Since the delamination propensity was maximal for PM 2000 steel, which had the lowest USE value, and delamination increases in direct proportion to the aspect ratio, particular attention must be given to this parameter during material processing.

Fig. 10. Scanning electron micrographs showing the differences in ductile fracture morphology for the LT notch position at a test temperature of 300oC for (a) PM 2000, (b) MA956, and (c) FeCrAl steels.

Because both fracture mechanisms, cleavage and ductile, only compete in the DBT curves of LT and LS notched specimens of FeCrAl steel, LT and LR notched specimens of MA956 steel, and LT notched specimens of PM2000 steel, the DBTT values could be compared only for these

cases. The very low DBTT of LT and LR notched specimens of ODS steels ($\approx -100^{\circ}\text{C}$) as compared to the FeCrAl steel ($\approx 90^{\circ}\text{C}$) could be attributed to differences in grain morphology and size.

The above results indicate that an understanding of the toughness anisotropy allows a good combination of high strength and high fracture toughness to be obtained in certain directions in ODS FeCrAl steels.

Final remarks. Hot extrusion of mechanical alloyed ODS FeCrAl steels leads to three types of anisotropy in the microstructure: ^[3]

- Crystallographic texture
- Submicrometric grain size with a high aspect ratio.
- Bundles of small grains with the existence of segregation/precipitation/inclusion along the extrusion direction.

Among these factors, the latter two are the main sources of the anisotropy in DBT behavior^[34].

With respect to the second factor, a submicrometric bamboo microstructure with a high aspect ratio may weaken the grain boundaries in the longitudinal direction, which may, in the case of the longitudinal loading direction of notched samples, reduce triaxiality stress state or intergranularly deflect a propagating crack with a resultant increase in fracture toughness. By contrast, in the case of the transverse loading direction, a submicrometric bamboo microstructure with a high aspect ratio would facilitate the intergranular brittle fracture and therefore would decrease the fracture toughness^[25-29, 31, 35-36]. The third factor would contribute to a greater weakening of the grain boundaries which would produce a further decrease in the fracture toughness. In this context, two directions appear to be available. The first is to align the extrusion direction with the maximum tensile service stress axis. The second is to reduce the microstructural anisotropy to reduce further the anisotropy in fracture toughness. Controlling the manufacturing process using ultra-clean raw powders

might reduce the anisotropy in the fracture toughness, while multidirectional processing techniques must be used to obtain a microstructure with equiaxed grains.

6 CONCLUSIONS

The directionality of the brittle-ductile transition of two ODS FeCrAl steels processed via different routes, i.e., as a tube (PM2000) and as a bar (MA956), was analyzed and compared with that of a Zr-rich particle strengthened FeCrAl alloy manufactured by high-vacuum casting. The results reported in this work support the following conclusions:

1. The USE values of FeCrAl steel are higher than those of ODS FeCrAl steels mainly due to the low volume fraction of second phase particles (one order lower) of FeCrAl steel but also due to the differences in UTS values and to the delamination propensity of ODS steels.
2. Remarkably, the DBTT of the LT and LR notched specimens of the ODS steels ($\approx -100^{\circ}\text{C}$) was much lower than that of the FeCrAl steel ($\approx 90^{\circ}\text{C}$) due to differences in grain morphology and size.
3. In the case of the ODS FeCrAl steels with an LT sample, the differences in USE could be partially justified by the higher volume fraction of second-phase particles and more importantly by the higher delamination tendency of the PM2000 steel.
4. For LT sample, full ductile fracture with delamination was observed in the case of PM2000 steel at the DBTT, defined as the temperature corresponding to the average values of USE and LSE. Thus, no true transition occurs in the fracture

mechanism of PM2000. By contrast, a mix of cleavage and ductile fracture was observed in the case of the MA956 steel.

5. The difference between the USE values for the TL and TR samples in the ODS FeCrAl steels could be justified in the case of the PM2000 steel by the presence of crack divider delamination in the TL-notched specimens. However, this was not the case for the MA956 steel because no delamination was observed for TL-notched specimens. One may speculate that the presence of crack arrester microdelamination in the TR-notched specimens could contributed to a higher absorbed energy compared to the TL-notched specimens.

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FIGURE CAPTIONS

Fig. 1. Orientation of the bending notched specimens relative to the product shape. a) PM2000 steel b) MA956 steel c) FeCrAl steel. 'L', 'T', 'R', 'S' and 'D' indicate longitudinal, tangential (PM2000 and MA956 steels) or transverse (FeCrAl steel), radial, short and diagonal directions respectively. The orientations of the bending specimens are designated according to ASTM standard [5] as 'LT', 'TL', 'TR', 'LS', 'TS', 'DD' and 'DS' in which the first letter indicates the specimen direction and the second the loading direction.

Fig. 2. Experimental setup of the bending test and criteria for defining transition temperature.

Fig. 3. Back-scattered SEM (PM2000 and MA956 steels) and optical (FeCrAl steel) images of the microstructures of (a),(c) and (e) transverse, and (b), (d) and (f) longitudinal cross sections of the PM2000 steel, MA956 steel and FeCrAl steel. The white arrow indicates the longitudinal direction. ((a)After [11], (b) after [7]).

Fig. 4. Typical Zr-rich particles of the FeCrAl steel and EDS results for two particles on ductile fracture surface of FeCrAl wrought steel sample tested at 155 °C.

Fig. 5. Orientation distribution function ($\phi_2 = 45^\circ$ section) of the (a) PM2000 steel, (b) MA956 steel, (c) FeCrAl steel (center of the thickness), and (d) FeCrAl steel (surface). (a) and (b) After [11])

Fig. 6. Effect of specimen orientation on the ductile-to brittle transition curve of (a) PM2000 steel (b) MA956 steel (c) FeCrAl steel.

Fig. 7. Selection of electron fractographs showing the relevant aspects of the fracture surfaces of single notch bend specimens of the PM2000 steel: (a) initiation of cleavage fracture (left) and details of the fracture initiation zone (right) of the LT specimen tested at -196°C; (b) microvoid coalescence zones bounded by crack divider delamination of the LT specimen tested at -60°C; (c) zones of intergranular brittle fractures bounded by crack divider delamination of the TL specimen tested at -196°C; (d) fracture initiation (left) and details (right) of the intergranular brittle fracture of the LR specimen tested at -70°C; (e) fracture initiation by microvoids coalescence followed by various crack arrester delamination and final intergranular brittle fracture of the LR specimen tested at 80 °C; and (f) schematic illustration showing delaminations geometries found in LT and LR samples tested according with Cartesian x,y,z and L,T,R (Figure 1(a)) reference systems. The white arrows indicate the initiation point of the fracture.

Fig. 8. Selection of electron fractographs showing the relevant aspects of the fracture surfaces of single edge notch bend specimens of the MA956 steel: (a) location of the initiation point (left) and detail (right) of the cleavage fracture at the end of a crack divider delamination of LT specimen tested at -196°C; (b) microvoid coalescence zones bounded by crack divider delamination of the LT specimen tested at -60°C; (c) zones of intergranular brittle fracture interspersed with thin layers of ductile fracture of the TL specimen tested at room temperature; (d) fracture initiation by microvoids coalescence followed by crack arrester

delamination and final intermixed zones of cleavage and ductile fracture modes of the LR specimen tested at -20oC; (e) intermixed zones of intergranular brittle and ductile fracture modes with occasional crack arrester delamination of the TR specimen tested at -20oC. The white arrows indicate the initiation point of the fracture.

Fig. 9. Selection of electron fractographs showing the relevant aspects of the fracture surfaces of single edge notch bend specimens of the FeCrAl steel: (a), (b) initiation of cleavage fracture in Zr-rich particles of the LT specimen tested at -20oC and TL specimen tested at 60oC respectively (see the arrow); (c), (d) macroscopic image of ductile fracture with crack divider delamination and detailed micrograph showing ductile fracture nucleation in Zr-particles of LT specimen tested at 150oC ; (e), (f) fracture initiation by coalescence of microvoids followed by crack arrester delamination and final cleavage fracture with small areas of ductile fracture of the DS specimen tested at 80oC and the TS specimen tested at 60oC respectively. The white arrows indicate the initiation point of the fracture.

Fig. 10. Scanning electron micrographs showing the differences in ductile fracture morphology for the LT notch position at a test temperature of 300oC for (a) PM 2000, (b) MA956, and (c) FeCrAl steels.

TABLES:

Table 1. Chemical composition of steels investigated (wt%).

<i>Material</i>	<i>C</i>	<i>Si</i>	<i>Mn</i>	<i>P</i>	<i>S</i>	<i>Cr</i>	<i>Al</i>	<i>Ti</i>
PM2000	<0.01	0.095	0.037	0.004	<0.005	18.6	5.2	0.54
MA956	0.012	0.12	0.097	0.005	0.007	19.33	4.7	0.44
FeCrAl	0.026	0.30	0.18	0.010	0.0004	21.0	4.8	0.05
<i>Co</i>	<i>Ni</i>	<i>Cu</i>	<i>O</i>	<i>Y</i>	<i>Zr</i>	<i>N</i>	<i>H(ppm)</i>	
0.039	0.03	0.015	0.091	0.391	-----	0.003	11.0	
0.066	0.101	0.021	0.108	0.376	-----	0.02	11.0	
0.02	0.22	<0.01	-----	-----	0.19	0.014	4.1	

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635 Table 2. Geometric measures of microstructural features and texture.

Material	Grains		Second phase particles			Texture
	D_{avg} , μm	l , μm	GAR	Size, nm	f_{vol} , %	
PM2000	0.7 ^[11]	1.6 ^[11]	2.3 ^[11]	19 ^[11]	1.1 ^[11]	(100)[110]S ^[11]
MA956	0.7 ^[8]	0.9 ^[8]	1.3 ^[8]	16 ^[8]	1.3 ^[8]	<110>IIED S ^[8]
FeCrAl	17.3	26	1.5	2350	0.15	(100)[110]W ^(*)

636 D_{avg} stands for average diameter; l stands for length; GAR is grain aspect ratio; f_{vol} stands for
637 volume fraction; S stands for strong; W stands for weak

638 ^(*) Texture in the centre of FeCrAl sheet

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641 Table 3. Tensile test data. 'L', 'D' and 'T' stand for longitudinal, diagonal and transverse
 642 orientations of the specimen. Values for MA956 after Ref. [11] and values for PM2000 after
 643 Ref. [7].

MATERIAL	T, °C	YS, MPa	UTS, MPa	A _u , %	A _r , %	n
PM2000 L	-196	1430	1530	9.5	10	0.041
	-60	1100	1230	10.2	16	0.053
	-20	1050	1200	9.2	14.1	0.058
	20	1000	1170	8.5	14.8	0.064
	150	930	1100	9.1	13.1	0.067
	300	820	1050	8.7	12.6	0.088
MA956 L	-196	1405	1550	14	21.1	0.054
	-80	1100	1260	11.8	18	0.061
	-60	1070	1250	11	16.9	0.065
	20	970	1150	10.2	15	0.068
	150	870	1020	9.1	12.6	0.065
	300	820	930	8.5	13	0.055
FeCrAl L	-196	-----	1030	0	0	-----
	-70	700	787	12.5	12.5	0.057
	-32	602	725	14.5	27.5	0.076
	20	560	664	11	27.8	0.069
	162	440	590	13.1	19.1	0.101
	400	420	549	13.2	25.5	0.094
FeCrAl T	-196	-----	948	0	0	-----
	-70	755	860	13	21.2	0.061
	-27	654	780	13.1	25.2	0.072
	20	614	731	9.3	20	0.069
	140	470	645	13.5	20.5	0.106
	400	455	595	10	19.5	0.094
FeCrAl D	-196	-----	1010	0	0	-----
	-70	710	790	12.8	14.1	0.055
	-32	610	730	10	22.1	0.071
	20	567	675	10.9	26.8	0.070
	150	460	595	13	19.9	0.092
	400	418	544	10.1	23.5	0.092

644 YS stands for yield strength; UTS stands for ultimate tensile strength; A_u stands for uniform
 645 elongation; A_r stands for total elongation, n stands for strain hardening exponent; T stands for
 646 temperature.

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649 Table 4. Bending data of the parameters of the DBT curves

MATERIAL	ORIENTATION NOTCH	LSE J	USE J	DBTT °C	FRACTURE MODE (*)
PM2000	LT	0.19	2.5	18	C → D+cdd → D
	LR	0.23	4.5	30	I → I+D+cad → D
	TL	0.16	1.7	50	I → I+D+cdd → D
	TR	0.20	3.2	50	I+cad → I+D → I+D
MA956	LT	0.30	4.5	-110	C → D+cdd → D
	LR	0.13	4.1	-80	C → C+cad → D
	TL	0.21	1.7	60	I → I+D → I+D
	TR	0.17	3.2	90	I → I+D → I+D
FeCrAl	LT	0.13	5.6	70	C → C+D+cdd → D
	LS	0.12	8.8	90	C → C+D+cdd → D
	TL	0.13	5.4	65	C → C+D+cad → D
	TS	0.14	6.6	75	C → C+D+cad → D
	DD	0.11	5.2	65	C → C+D+cdd → D
	DS	0.10	7.0	70	C → C+D+cad → D

650 LSE- Lower Shelf Energy; USE- Upper Shelf Energy; DBTT- Ductile-to-Brittle-Transition
651 Temperature; C- Cleavage; D- Ductile; I- Intergranular; cdd-crack divider delamination; cad-
652 crack arrester delamination

653 (*) for example: C → C+D+cad → D, cleavage in LSE, cleavage + ductile + crack arrester
654 delamination in the DBT, ductile in USE

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**ON THE ANISOTROPY OF THE DUCTILE TO BRITTLE TRANSITION BEHAVIOR IN A WROUGHT
AND IN TWO OF OXIDE -DISPERSION -STRENGTHENED (ODS)-FeCrAl STEELS**

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ABSTRACT

The directionality of the ductile-brittle transition behavior of two oxide-dispersion - strengthened (ODS) FeCrAl steels with similar chemical compositions, tensile properties and sub-micrometric grain sizes but different processing routes, and a Zr-particle strengthened FeCrAl steel manufactured by high vacuum melting is discussed. Despite the similarities of the ODS FeCrAl steels, strong differences in the lower and upper shelf energy and the ductile to brittle transition temperature were observed for longitudinal through thickness notched specimens. Although the lower and upper shelf energies of longitudinal surface notched specimens of ODS FeCrAl steels are similar, a strong difference in the ductile-to-brittle transition temperature is observed. For through-thickness notched and surface notched specimens taken transversely, the analyzed ODS FeCrAl steels show a similar ductile-to-brittle behavior. In general, the FeCrAl alloy strengthened with Zr-particles presents a more isotropic behavior and a higher ductile-to-brittle transition temperature than the ODS FeCrAl steels. In addition, the upper shelf energy of the FeCrAl steel strengthened with Zr-particles is significantly higher than that of the ODS FeCrAl steels.

Keywords: ductile–brittle transition behavior; delamination; mechanical alloying; oxide dispersion strengthened ferritic alloy

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1. INTRODUCTION

FeCrAl steels are a subclass of ferritic stainless steels containing 15-25wt% Cr and 4-7wt% Al. These materials are usually prepared by melting and casting under high vacuum conditions. According to the phase diagram, the microstructure is ferritic at all temperature ranges. The body-centered-cubic crystalline structure of ferrite makes the ductile-to-brittle transition to decrease with increasing the loading rate and/or increasing stress triaxiality stress state. These steels are not employed for structural purposes but for high-temperature applications requiring excellent resistance to oxidation e.g., for heating-resistant elements of furnaces or for construction materials in the core of fast neutron reactors.

ODS FeCrAl steels, in contrast to FeCrAl steels are manufactured by mechanical alloying of yttria (Y_2O_3) nano-particles and elementary powders of Fe, Cr and Al elements. These steels are considered among the most promising structural steels for the nuclear industry because they combine acceptable creep strength and excellent swelling resistance in supercritical pressurized water, as a consequence of the nano-scale yttria particles dispersed in the FeCrAl matrix.^[1] However, for full structural applications, further improvements in material processing are necessary to enhance their fracture toughness. Hot-extruded bars with sub-micrometric grain size present strong anisotropy in fracture toughness with a very low upper-shelf energy level, such as 0.30 J, for transverse specimens^[2-3]. Fracture toughness can be improved by eliminating the stringer-like alumina inclusions in the source powders^[2]. Moreover, to reduce the fracture toughness anisotropy, the presence of bundles of small grains with segregation/precipitation/inclusion along the extrusion direction should be

minimized^[3]. While these measures will effectively contribute improve fracture toughness, the causes of the poor fracture toughness in the transverse direction remain unclear.

The goal of this study is to understand the causes of both the strong anisotropy in the fracture toughness and the very low upper shelf energy (USE) for transverse specimens in ODS FeCrAl steels. Accordingly, this paper examines the fracture behavior of longitudinal and transversely taken specimens of two ODS FeCrAl steels with similar chemical compositions and microstructures but with slight differences in the grain elongation and texture. These ODS steels were manufactured via mechanical alloying by different manufacturers. The behaviors of these ODS steels were compared with that of a FeCrAl steel of similar chemical composition that was manufactured via melting and casting under high vacuum conditions and strengthened with Zr-rich particles.

2. MATERIALS

The chemical compositions of the three steels supplied, as determined by X-ray fluorescence spectrometry, wet chemistry, colorimetry and inert gas fusion techniques are listed in Table 1. According to their chemical composition, these materials consist of ferrite solid-solutions with a body-centered-cubic crystalline structure. Moreover, the embrittling effect of C, N interstitials is controlled by additions of Ti for the ODS FeCrAl steels and Zr for the FeCrAl steel. Both Ti and Zr are strong carbide and nitride formers, and the resulting carbides and nitrides are more stable than chromium carbide and chromium nitride^[4].

The PM2000 steel was provided by the Plansee Group (Reutte, Austria) in the form of a hot-rolled tube with a diameter of 100 mm and thickness of 7.9 mm. The material processing involved mechanical alloying of elemental Fe, Cr, Al, Ti powders with 0.5 wt.% Y₂O₃ particles in a shielding atmosphere of H₂, encapsulation of the powder, evacuation of the capsule and hot-

isostatic pressing at 1050 °C; hot-rolling/hot-extruding at 1050 °C and air cooling to room temperature.

The MA956 alloy was provided by Special Metal Corporation (Hereford, UK) in the form of a hot-extruded bar with a diameter of 60 mm. The material processing involved mechanical alloying of elemental Fe, Cr, Al, Ti powders with 0.5 wt.% Y₂O₃ particles in a shielding atmosphere of Ar, encapsulation of the powder, evacuation of the capsule and hot-isostatic pressing at 1050 °C; hot-extrusion at 1050 °C and air cooling to room temperature.

The FeCrAl alloy was provided by Goodfellow Cambridge Ltd. (Huntington, UK) in the form of a temper-rolled square sheet with a thickness of 5 mm and side length of 200 mm. The material processing involved melting and casting under high vacuum, hot forging and finally temper-rolling. It was initially assumed that the rolling direction was parallel to the watermarks on the sheet surface.

3. EXPERIMENTAL PROCEDURE

Before machining the specimens for mechanical testing from the FeCrAl steel, a 0.5 mm-thick layer was removed from both surfaces of the sheet so that the microstructure and texture were homogeneous in the cross-section of the specimens. Tensile specimens with a gauge of 25 mm in length, 4 mm in width and 3.5 mm-thick were machined such that the tensile axis was parallel to the longitudinal (L), diagonal (D) and transverse (T) directions of the sheet, Figure 1(a-c). The specimens were tested at a range of temperatures between -196°C and 300°C using a crosshead speed of 0.0125 mm/s.

Fig. 1. Orientation of the bending notched specimens relative to the product shape. a) PM2000 steel b) MA956 steel c) FeCrAl steel. 'L', 'T', 'R', 'S' and 'D' indicate longitudinal, tangential (PM2000 and MA956 steels) or transverse (FeCrAl steel), radial, short and diagonal directions respectively. The orientations of the bending specimens are designated according to ASTM standard^[5] as 'LT', 'TL', 'TR', 'LS', 'TS', 'DD' and 'DS' in which the first letter indicates the specimen direction and the second the loading direction.

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98 Single-edge notch bend specimens (4 mm in width; 3 mm in thickness; 27 mm in length; 1 mm
99 in notch depth; 0.1 mm in notch tip radius; and 60° notch angle) were machined from the as-
100 received materials with four notch positions for the PM2000 and MA 956 steels and six for the
101 FeCrAl steel (Figure 1). The orientation of the notch was defined according to ASTM Standard
102 E399 using a two-letter code^[5]. The first letter designates the normal direction to the notch
103 plane, and the second the expected direction of the crack propagation. The notch orientations

included LT, LR, TL and TR for the PM2000 and MA956 steels (Figure 1(a) and Figure 1(b), respectively) and LT, LS, DD, DS, TL and TS for the FeCrAl steel (Figure 1(c)). The tests were performed using the bending test fixture shown in Figure 2. The specimens were tested at a range of temperatures between -196°C and 300°C using a crosshead speed of 1 mm/s. The fracture toughness was estimated by integrating the area under the load-displacement curve up the point of failure.

Figure 1 shows a schematic representation of the criteria used for defining the transition temperature. It is commonly accepted that the USE is the average energy value for all specimens whose test temperature is above the upper end of the transition temperature, T1. Above this temperature, fracture is mainly ductile. This T1 temperature is named fracture transition temperature (FTP). This is the temperature at which fracture changes from almost totally ductile to substantially brittle, or as in the case of PM2000 and MA956, delaminations appear, on decreasing temperature. Problem of brittle fracture is almost negligible above this temperature. In contrast to the upper self, the lower shelf is the region in which the temperature is lower than the lower transition temperature T2. This T2 temperature is named as nil ductility temperature (NDT), and is defined as the temperature below which a material fractures under cleavage mechanisms. On the basis of this ~~three~~ ductile-to-brittle transition temperature (DBTT) was estimated as the test temperature corresponding to the average value of upper shelf energy (USE) and lower shelf energy (LSE) of the energy vs temperature curve.

Fig. 2. Experimental setup of the bending test and criteria for defining transition temperature.

125

126 The microstructure of the FeCrAl steel was examined in terms of grain morphology, grain size,
 127 and second-phase particles using optical microscopy. The three orthogonal surfaces of the
 128 sheet LT, LS and TS (Figure 1(c)), were prepared by conventional metallographic techniques
 129 including grinding, polishing and etching with an acid solution of 20% HNO_3 , 30% HCl ,
 130 20% $\text{C}_2\text{H}_4\text{O}_2$ and 30% CH_4O .

The texture of FeCrAl steel was determined by X-ray diffraction (XRD) in a Bruker AXS D8 (Bruker GmbH, Karlsruhe, Germany). Texture analysis of PM2000 and MA956 has been described elsewhere in Ref. [7] and Ref. [11]. The diffraction studies were performed with Co- $K\alpha$ radiation on the TS cross section of the sheet, Figure 1(c). SEM characterization was performed in a Hitachi S 4800 J field emission gun scanning electron microscope (FEG-SEM) operating at 10 kV.

4. EXPERIMENTAL RESULTS

Microstructure

Previous characterization of the microstructure of the PM2000 steel revealed a bamboo grain structure with an equiaxed dimension ($\sim 0.7 \mu\text{m}$) in the transverse (TR) cross-section of the tube but significant elongation ($\sim 1.6 \mu\text{m}$) in the longitudinal (LR) cross-section (Figure 3(a))^[6,11]. Spherical particles of yttria ($f_{\text{vol}} = 1.3\%$) with sizes ranging from 3 nm to 40 nm are mainly located at the grain boundaries.^[75] Al_2O_3 and $\text{Ti}(\text{C},\text{N})$ particles ($f_{\text{vol}} \approx 0.12\%$) up to 100 nm in size were also found^[6].

The microstructure of the MA956 steel, presented a similar morphology to that of the PM2000 steel, with equiaxed grains in the transverse (TR) cross section ($\sim 0.7 \mu\text{m}$) but lower elongation ($\sim 0.9 \mu\text{m}$) in the extrusion direction (Figure 3(b))^[7]. Two main distributions of spherical particles of yttria ($f_{\text{vol}} \approx 1.1\%$) were observed: small particles with sizes from 5 nm to 40 nm and large particles up to 500 nm^[8]. Al_2O_3 -~~angled~~-particles 100 nm in size and $\text{Ti}(\text{C},\text{N})$ particles up to a few μm in size were also found^[9]. The volume fraction of Al_2O_3 and $\text{Ti}(\text{C},\text{N})$ particles was approximately 0.154%.

The microstructure of the FeCrAl alloy (Figure 3(c)) was inhomogeneous throughout the thickness of the sheet. At the center layer, the microstructure of the grains was partially

recovered, whereas near the surfaces, some recrystallized grains were apparent. Moreover, the average grain size near the surface at the three orthogonal planes was slightly higher than in the center layer. The morphological analysis of the microstructure revealed that the grains were ellipsoidal in shape with a maximum dimension of 29 μm at the LT plane, followed by 22 μm at the LS plane and 17 μm at the TS plane. Because Zr has a strong chemical affinity with oxygen, nitrogen, sulfur and carbon, and the Zr content of the alloy is considerably higher than its solubility limit in ferrite, second-phase particles in the form of ZrO_2 , ZrC and Zr(C,N) were expected. The presence of sub-micrometric spherical particles of ZrO_2 in low-carbon steels has been reported^[10]. These particles would restrict the growth of the grains at high temperatures, resulting in a fine grain microstructure at room temperature, which is beneficial for imparting good mechanical properties to the material. In the present paper, the presence of second phase particles was analyzed in 40 fields (x500 magnification) randomly taken from the three orthogonal planes of the sheet. Carbides and carbo-nitrides of zirconium were observed (Figure 4). The particles typically have a cubic or rectangular-prism morphology and a yellow color under optical microscopy, but do not present a preferred orientation ~~(Figure 4), i.e. "~~
i.e. the Zr-particles does not follow an alignment along extrusion direction such as the one followed by yttria particles has on PM2000. The average size of the particles is 2.4 μm , with maximum and minimum values of 8 μm and 1 μm , respectively. Table 2 summarizes the microstructural features of the alloys.

Fig. 3. Back-scattered SEM (PM2000 and MA956 steels) and optical (FeCrAl steel) images of the microstructures of (a),(c) and (e) transverse, and (b), (d) and (f) longitudinal cross sections of the PM2000 steel, MA956 steel and FeCrAl steel. The white arrow indicates the longitudinal direction. ((a)After [11], (b) after [7]).

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Fig. 4. Typical Zr-rich particles of the FeCrAl steel and EDS results for two particles on
ductile fracture surface of FeCrAl wrought steel sample tested at 155 °C.

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177 Texture

178 Figure 1 indicates the ratio between the directions for sample machining and texture
179 measurements. In this sense, RD stands for rolling (or extrusion) direction, ND for normal
180 direction and TD for tangential direction. The section of the orientation distribution function
181 (ODF) obtained from a transverse cross-section of the PM2000 steel tube shown in Figure
182 5(a) reveals a strong incomplete α -fiber (RD|| $\langle 110 \rangle$) texture in which the $\{001\}\langle 110 \rangle$
183 orientation is dominant^[611]. A less intense $\{111\}\langle 011 \rangle$ orientation is also detected as it was
184 previously reported^[611].

185 The section of the ODF obtained from a transverse cross section of the MA956 steel bar shown
186 in Figure 5(b) reveals a strong α -fiber (RD|| $\langle 110 \rangle$) texture^[11]. Texture gradients along the radial
187 direction are only observed very close to the surface^[11]. Moreover, a secondary $\{111\}\langle 011 \rangle$
188 orientation is detected, which is slightly stronger than that of PM2000 steel as it was
189 previously reported^[11].

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190 Sections of the ODF in the center of the thickness of the FeCrAl steel sheet and near the sheet
191 surface are shown in Figure 5(c) and Figure 5(d), respectively. The texture in the center (Figure
192 5(c)) is a combination of an incomplete, broadened and weak α -fiber and a slightly less intense
193 $\{332\}\langle 113 \rangle$ orientation, whereas near the surface (Figure 5 (d)), the texture is a combination
194 of a weak Goss orientation ($\{011\}\langle 100 \rangle$) and a slightly less intense $\{001\}\langle 110 \rangle$ orientation. The
195 purpose of Figures 5(c) and 5(d) is to illustrate the texture gradient through the thickness of
196 the FeCrAl sheet. However, as it was mentioned before, during machining of mechanical test
197 samples it was removed a 0.5 mm-thick layer from both surfaces of the sheet in order to
198 remove the heterogeneity in texture, so that the microstructure and texture were
199 homogeneous in the cross-section of the specimens. The last column of Table 2, summarizes
200 the main components of the texture of the steels.

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Fig. 5. Orientation distribution function ($\phi_2 = 45^\circ$ section) of the (a) PM2000 steel, (b) MA956 steel, (c) FeCrAl steel (center of the thickness), and (d) FeCrAl steel (surface). ((a) and (b) After [11])

Mechanical testing

Tensile properties. Table 3 summarizes the tensile properties as a function of the test temperature for longitudinal (L) specimens of the PM2000^[7] and MA956^[11] steels and longitudinal (L), transverse (T) and diagonal (D) specimens of the FeCrAl steel. The strengths of the two ODS FeCrAl steels are similar to each other but considerably higher than that of the FeCrAl steel. The uniform elongation and strain hardening rate are similar for the three steels but the total elongation of the FeCrAl alloy is significantly higher than those of the ODS FeCrAl steels. Reducing the test temperature significantly increases both, the strength and the ductility and moderately decreases the strain hardening rate.

In the case of the FeCrAl steel, the specimens tested at a temperature of -196°C , broke before reaching the yield strength (YS) of the material. The YS and ultimate tensile strength (UTS) are highest for specimens taken in the T direction. Moreover, the differences in YS and UTS when changing the tensile direction are small and can be attributed to the material texture^[12-13]. Although the elongation is slightly lower for the transverse specimen than for the longitudinal and diagonal specimens, the strain hardening rate is nearly identical in the L, D and T directions.

In relation to the anisotropy in tensile properties of the ODS FeCrAl steels, previous studies on hot-extruded bars manufactured by mechanical alloying, have shown that uniform and total elongations in the tangential direction are about 20% lower than in the longitudinal direction, whereas the YS is about 5% higher^[2,3], which is in good agreement with the above results for the FeCrAl steel.

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224 *Bending tests.* The DBT curves of the PM2000 steel are shown in Figure 6(a), and the
225 corresponding values of LSE, USE and DBTT are summarized in Table 4. The through-thickness
226 notched specimens (LT and TL ~~notch-orientations~~specimens) exhibits lower USE values than
227 the corresponding surface notched specimens with LR and TR ~~notch-orientations~~specimens.
228 The DBTT values for the longitudinal specimens (LT and LR ~~notch-orientations~~specimens) are
229 lower than those of the tangential (TL and TR ~~notch-orientations~~samples) specimens. The DBT
230 curve for the LR ~~notch-orientations~~sample presents a peak at a temperature of 60°C.

231 The DBT curves of the MA956 steel are shown in Figure 6(b), and the corresponding values of
232 LSE, USE and DBTT are presented in Table 4. The tangential specimens with the TL and TR
233 notch positions exhibit lower USE values than the corresponding longitudinal specimens with
234 LT and LR notch positions. This difference is particularly striking for the through-thickness
235 notched (TL and LT ~~notch-orientations~~specimens) specimens. The DBTTs of the longitudinal (LT
236 and LR notch positions) specimens are lower than those of the tangential (TL and TR
237 ~~specimens~~notch-orientations) specimens. The DBT curve for the LR notch position presents a
238 peak at -25°C.

239 The DBT curves of the FeCrAl steel are shown in Figure 6(c) and the corresponding values of
240 LSE, USE, and DBTT are presented in Table 4. The LT, TL and DD ~~notch-orientations~~samples
241 exhibit nearly identical DBT behaviors i.e., regarding the fracture behavior of the through-
242 thickness notches, the material is isotropic. The USE values of the surface notches (LS, TS and
243 DS ~~specimens~~notch-orientations) are significantly higher than those of the through-thickness
244 notches. Moreover, the LS ~~notch-orientations~~sample exhibits the highest USE value but, in
245 contrast to the PM2000 and MA956 steels, presents no peak in the USE region of the DBT
246 curve. The LSE and DBTT values of all analyzed orientations are nearly identical.

Fig. 6. Effect of specimen orientation on the ductile-to brittle transition curve of (a) PM2000 steel (b) MA956 steel (c) FeCrAl steel.

Fracture analysis of bending specimens

PM2000 steel. For the LT ~~notch orientation~~ sample at a test temperature of -196°C , fracture occurred by transgranular cleavage along a plane inclined between 30° and 45° relative to the notch plane (Figure 7(a)). When the regions near the initiation sites were studied at successively higher magnifications the microstructural feature triggering the fracture process could not be detected (zoom in Figure 7(a)).

From -60°C to 200°C the fracture occurred along the notch plane by coalescence of microvoids with the presence of crack divider delamination (Figure 7(b)). The crack divider delamination leads to a decrease in the triaxiality of the stress state of the notched specimens, and thus, to a decrease in both the DBTT and USE values^[14]. The number of delaminations decreases as the test temperature is increased. At a test temperature of 300°C the fracture morphology is fully ductile.

For the TL ~~notch orientation~~ sample at a test temperature of -196°C , intergranular brittle fracture along the notch plane was mostly observed (Figure 7(c)). From test temperatures of -60°C to 200°C , the area fraction of the main fracture surface occupied by ductile features increases as the test temperature is increased. Within this temperature interval, the

delamination number (crack divider type) decreases as the test temperature increased. At a test temperature of 300 °C, the fracture morphology is fully ductile.

For LR and TR ~~notch-orientation~~sample and test temperatures ranging between -196 °C and room temperature, areas of intergranular brittle fracture interspersed with ductile steps are observed on a plane inclined approximately 70° relative to the notch plane (Figure 7(d)).

For the LR ~~notch-orientation~~sample and test temperatures between room temperature and 100°C strong delamination parallel to the rolling plane of the crack arrester type is observed (Figure 7(e)). At higher test temperatures, only incipient crack arrester delamination is observed in the notch tip. This delamination type entails bifurcation of the main fracture along the delamination plane and restarting of the fracture process in the actual unnotched ligament. As a result, enormous energy consumption can occur during the bending test, particularly in the upper shelf region of the DBT curve^[14-15]. At test temperatures other than -196°C no delamination was observed for the TR notched specimens.

Figure 7(f) shows a schematic illustration of the splitting in both LT and LR samples after Kimura et al. ^[15], with the corresponding orientation of TR plane regarding the L, T and R system (Figure 1). In a Charpy test, the main stress component after the strike is σ_y . The other two stress components, σ_x and σ_z , are generated by the localized plastic constrain at the notch and/or the crack tip and of lower magnitude than σ_y . The splitting in LT and LR samples could be explained on the basis that the plastic deformation in R-direction is considerably higher than in T-direction. Therefore, the plastic constrain in R-direction would lead to an increase of σ_x in the case of LT samples, and of σ_z in the case of LR sample that would cause the distribution of cracks found experimentally. Those delaminations are similar to those found under crack-divider and crack-arrester basic geometries.

Fig. 7. Selection of electron fractographs showing the relevant aspects of the fracture surfaces of single notch bend specimens of the PM2000 steel: (a) initiation of cleavage fracture (left) and details of the fracture initiation zone (right) of the LT specimen tested at -196°C; (b) microvoid coalescence zones bounded by crack divider delamination of the LT specimen tested at -60°C; (c) zones of intergranular brittle fractures bounded by crack divider delamination of the TL specimen tested at -196°C; (d) fracture initiation (left) and details (right) of the intergranular brittle fracture of the LR specimen tested at -70°C; (e)

fracture initiation by microvoids coalescence followed by various crack arrester

delamination and final intergranular brittle fracture of the LR specimen tested at 80 °C; and
(f) schematic illustration showing delaminations geometries found in LT and LR samples
tested according with Cartesian x,y,z and L,T,R (Figure 1(a)) reference systems. –The white
arrows indicate the initiation point of the fracture.

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289 | MA956 steel. Similar to the PM2000 steel, for the LT ~~notch-orientation~~sample at -196 °C,
290 fracture occurs by transgranular cleavage along a plane inclined between 30° and 45° relative
291 to the notch plane (Figure 8(a)). In this case, however, the microstructural feature triggering
292 the fracture process was shown to correspond to the tip of a crack divider delamination. From
293 -60 °C to -20 °C, fracture occurs along the notch plane by coalescence of microvoids with the
294 presence of delamination of the crack divider type (Figure 8(b)). In this steel, however, the
295 delaminations were more irregular and far less numerous than in the PM2000 steel. From
296 room temperature to 300 °C the fracture morphology was fully ductile, without delamination.

297 | For the TL ~~notch-orientation~~sample and temperatures ranging between -196 °C and -20 °C the
298 fracture morphology is mostly brittle intergranular (Figure 8(c)). At test temperatures of -20 °C
299 to 300 °C, the area fraction of the main fracture surface occupied by ductile features increases
300 as the test temperature is increased. At test temperature of 300 °C, the area fraction of the
301 fracture surface occupied by intergranular fracture is approximately 50 %. No delamination
302 was observed for the TL ~~notch-orientation~~sample.

303 | In contrast to the PM2000 steel, for LR ~~specimens notch-orientation~~ at a test temperature of -
304 196°C, fracture occurs by transgranular cleavage along a plane inclined between 30° and 45°
305 relative to the notch plane. Specimen fracture nucleated in a Ti-rich particle located very near
306 the notch tip. As the test temperature increases from -70 °C to 300 °C, the area fraction of the

307 main fracture surface occupied by ductile features increases. From -70 °C to -20 °C strong
 308 delamination parallel to the rolling plane of the crack arrester type is observed (Figure 8(d)).

309 For the TR [specimens notch orientation](#) and a test temperature of -196 °C, intergranular brittle
 310 fracture along the notch plane is observed. As the test temperature is increased from -70 °C to
 311 300 °C the area fraction of the main fracture surface occupied by layers of ductile features
 312 increases. At a test temperature of 300 °C, the area fraction of the fracture surface occupied
 313 by intergranular fracture is about 30 %. From -70 °C to room temperature, small delamination
 314 parallel to the rolling plane of the crack arrester type, is observed (Figure 8(e)).

Fig. 8. Selection of electron fractographs showing the relevant aspects of the fracture surfaces of single edge notch bend specimens of the MA956 steel: (a) location of the initiation point (left) and detail (right) of the cleavage fracture at the end of a crack divider

delamination of LT specimen tested at -196°C; (b) microvoid coalescence zones bounded by crack divider delamination of the LT specimen tested at -60°C; (c) zones of intergranular brittle fracture interspersed with thin layers of ductile fracture of the TL specimen tested at room temperature; (d) fracture initiation by microvoids coalescence followed by crack arrester delamination and final intermixed zones of cleavage and ductile fracture modes of the LR specimen tested at -20°C; (e) intermixed zones of intergranular brittle and ductile fracture modes with occasional crack arrester delamination of the TR specimen tested at -20°C. The white arrows indicate the initiation point of the fracture.

315

316 *FeCrAl steel*. For any notch orientation and for test temperatures ranging from -196°C to -20°C,
317 the main fracture surfaces revealed mostly transgranular cleavage (Figure 9(a) and Figure
318 9(b)), where fracture occurs along of the notch plane. The initiation point that caused the final
319 failure of the specimen was located by tracing back the path of river lines on the fracture
320 surface. In all cases, fracture initiated from ZrC or (C,N)Zr particles near the notch tip (Figure
321 9(a) and Figure 9(b)). At a test temperature of -196°C, more than one initiation point near the
322 notch tip was observed, whereas at the other test temperatures, the fracture initiated at a
323 single initiation point.

324 As the test temperature is increased, the fracture mode gradually varies from cleavage
325 fracture to dimpled fracture. At test temperatures higher than 150 °C, the fracture surface is
326 fully ductile, regardless of the notch orientation (Figure 9(c) and Figure 9(d)). The ductile
327 fracture nucleated in all cases from ZrC or (C,N)Zr particles.

328 The fracture surfaces of through-thickness notched (LT, TL and DD) specimens tested at
329 temperatures ranging from -60 °C to 150 °C exhibit delamination of the crack divider type,
330 although to a much lesser extent than that observed for the LT and TL notched specimens of
331 the PM2000 and MA956 steels. Within the same temperature range, the LS, TS and DS notched

specimens exhibited one or two delaminations of the crack arrester type (Figure 9(e) and Figure 9(f)) similar to the LR and TR ~~notch-orientation~~samples of the PM2000 and MA956 steels.

Fig. 9. Selection of electron fractographs showing the relevant aspects of the fracture surfaces of single edge notch bend specimens of the FeCrAl steel: (a), (b) initiation of cleavage fracture in Zr-rich particles of the LT specimen tested at -20°C and TL specimen tested at 60°C respectively (see the arrow); (c), (d) macroscopic image of ductile fracture with crack divider delamination and detailed micrograph

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showing ductile fracture nucleation in Zr-particles of LT specimen tested at 150oC ;
(e), (f) fracture initiation by coalescence of microvoids followed by crack arrester
delamination and final cleavage fracture with small areas of ductile fracture of the
DS specimen tested at 80oC and the TS specimen tested at 60oC respectively. The
white arrows indicate the initiation point of the fracture.

335

336 5. DISCUSSION

337 The ductile-to-brittle transition in ferritic steels is associated with two different failure modes.

338 At high temperature, in the USE region of the DBT curve, ductile fracture occurs by nucleation

339 and coalescence of microvoids, thus producing ductile tearing. This process requires extensive

340 plastic deformation, and therefore a large amount of energy is consumed by the specimen

341 during fracture. Void nucleation and growth are determined by the combination of stress

342 triaxiality state and plastic strain produced by void nucleation and growth^[16]. The most

343 important microstructural parameter controlling the ductile fracture is the volume fraction of

344 the second-phase particles nucleating microvoids^[17]. The DBT is a result of competition

345 between plastic flow and cleavage or intergranular fracture and can be defined as the point

346 where the yield stress equals the cleavage stress or intergranular fracture stress. In the case of

347 ferritic steels of similar chemical composition, the crystallographic grain size is the most

348 important microstructural parameter controlling the DBTT in the case of cleavage-to-ductile

349 transition^[18], whereas in the intergranular-to-ductile transition case, the amount of segregated

350 impurities, the orientation of the long dimension of the grain relative the applied stress, and

351 the grain boundary morphology are the most important parameters^[19,20]. Thus, when cleavage

352 occurs, the DBTT is different from that when intergranular brittle fracture occurs. The DBTT in

353 the case of cleavage fracture is usually considerably lower than in the case of intergranular

354 fracture^[19]. The stress triaxiality state is the main factor controlling the DBTT, in the sense

that the DBTT increases with increasing the stress triaxiality, while the USE decreases^[21]. In this regard, the residual strain, i.e., the residual elastic strain remain stored in the material in the end of its manufacturing process, may affect the DBTT by changing the constrain conditions under which fracture occurs in the lower transition region^[22]. The effect of the residual strains is to increase the stress triaxiality state and then to decrease the fracture toughness in the lower transition region, however, the fracture toughness remains unaltered in the upper transition region because in this region the residual strains are relaxed by plastic strain.

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In the low temperature region, the LSE region, brittle fracture is usually considered to occur by transgranular cleavage, however, intergranular brittle fracture can occur if the grain boundary is weakened by segregation of impurity atoms^[19] or because of the grain morphology^[20]. These two fracture micromechanisms, cleavage and brittle intergranular, differ significantly.- Cleavage fracture occurs by the dynamic propagation of slip-induced microcracks nucleating in a second phase or microstructural heterogeneity^[23]. Cleavage fracture is tensile stress controlled, and the local cleavage stress below the notch tip is independent of the temperature and strain rate for slip-induced cleavage^[24]. Thus, the cleavage strength is an intrinsic property of the material. As in cleavage fracture, intergranular fracture is a tensile stress controlled process^[19], however, the fracture strength of the grain boundary depends on the segregation-induced weakening intensity^[19] and, when the applied stress is normal to the long direction of the grains, on the aspect ratio of the grains, alignment of the grains and the smoothness of the grain boundaries. ^[20, 25]

On the other hand, there are various factors which must be taken into account when discussing the anisotropic behavior of DBT: inclusion shape, grain shape, microstructural anisotropy due to chemical segregation with bunching of elongated grain structure, grain

boundary alignment, the morphology of grain boundaries and crystallographic texture^[259-286].

The inclusion shape is disregarded in the present study due to the low S content of the studied steels and because the second phase particles found in the microstructural analysis did not present a preferential orientation. The grain shape itself is important because a high aspect ratio promotes material delamination, which is a very important source of anisotropy^[297-312].

Delamination decreases the stress triaxiality state ahead of the notch tip, which in turn produces a decrease in both DBTT and USE values^[277-329]. With respect to material delamination, the chemical segregation to the grain boundary, the grain boundary alignment and the morphology of the grain boundaries are also important promoters^[19, 20, 259]. The crystallographic texture, on the other hand, can affect the morphology of delaminations^[334].

The fiber texture of MA956 means that there is no preferred transverse orientation, and therefore delaminations in the transverse direction are more difficult. In contrast, there is no fiber texture in PM2000, but with some preferred <110> orientation, which could explain why is prone for delamination in the transverse direction. Regarding the longitudinal direction, both materials, i.e. MA956 and PM2000 maintain a preferred <110> orientation, being more intense in PM2000. Again, this is why PM2000 is prone for delaminations.

Comparative analysis of ODS steels. Both ODS steels have similar chemical composition and tensile properties, but the PM2000 steel exhibits grains more elongated with an identical transverse dimension and an incomplete α -fiber in which (100)<110> is the main orientation, instead of a full α -fiber texture. Thus, no great differences between the parameters of the DBT curves for the longitudinal specimens are expected. However, as shown in Figure 6(a), Figure 6(b) and Table 4, significant differences are observed for the LT and LR ~~specimens~~^{notch} ~~orientations~~. In the case of the LT ~~notch-orientations~~^{sample}, the differences in USE could be partially justified on the basis of the higher volume fraction of the second-phase particles and, more importantly, by the higher delamination tendency of the PM2000 steel. The higher delamination tendency of PM2000 steel is due to the higher elongation of the grains compared

to the MA956 steel. The difference in DBTT is attributed to the definition used for this parameter i.e., the temperature corresponding to the average value of the USE and LSE values. For the LT ~~specimens~~~~notch-orientation~~, at the so defined DBTT, full ductile fracture with delaminations was observed in the case of PM2000 steel whereas a mixture of cleavage and ductile fracture was observed in the case of MA956 steel. Thus, no true transition occurred in the fracture mechanism of the PM2000 steel using the above DBTT definition. Moreover, a second transition in the DBT curve of PM2000 steel (Figure 6(a)) between -60°C and -196°C in which full ductile fracture with delaminations and transgranular cleavage fracture were observed, respectively. Furthermore, this second transition occurred in the same temperature range as the DBT of MA956 alloy. It is therefore suggested that the DBT is similar for the LT notch position of both ODS steels.

In the case of the LR ~~notch-orientation~~~~sample~~, a slight difference in USE value is observed. Moreover, this difference is opposite to that expected on the basis of the difference in the volume fraction of second-phase particles of both ODS steels. However, a marked difference in DBTT values was observed that could be attributed to the difference in the fracture mechanism in the LSE region: intergranular fracture in the case of PM2000 steel and cleavage fracture in the case of MA956 steel. As noted above, DBTT was considerable higher for intergranular fracture than for cleavage fracture. The energy peak at the beginning of the USE region of the DBT curves is due to the formation of crack arrester delamination due to the enormous energy consumption during the process of delamination → unnotched bending → fracture reinitiation during bending test. The temperature difference at the peak position in the DBT curve is likely related to the different fracture mechanism in the LSE region.

The DBTT of transverse specimens is higher than that of longitudinal specimens, which could be explained partially by the extra elongation of the grains in the longitudinal direction^[22]. However, the differences in the fracture mechanism operating in the DBT region of the

431 longitudinal and transverse specimens revealed that the weakening intensity of the grain
432 boundaries greatly contributed to the differences in DBTT. Except for LT notch position of
433 PM2000 steel, the USE values of the longitudinal specimens of both steels were significantly
434 higher than the transverse USE values, which could be due to the presence of a mix of
435 intergranular and ductile features in the USE region of transverse specimens.

436 Significant differences in the USE values for the TL and TR ~~specimens notch orientations~~ in
437 both steels were observed. The difference in USE values for the TL and TR ~~specimens notch~~
438 ~~orientations~~ could be justified in the case of the PM2000 steel by the presence of crack divider
439 delamination in the TL-notched specimens; however, in the case of MA956 steel no
440 delamination were observed for TL-notched specimens. One may speculate that the presence
441 of crack arrester microdelamination in TR-notched specimens could contribute to a higher
442 absorbed energy as compared to the TL-notched specimens.

443 *Comparative analysis of ODS and FeCrAl steels.* Comparing the results for the FeCrAl steel with
444 those for the ODS FeCrAl steels (Figure 6 and Table 4) the great in-plane-isotropy of the DBTT
445 of FeCrAl steel is notable. The USE values of the through thickness notched (LT, TL and DD)
446 specimens can also be considered isotropic in the sheet plane. Greater differences in USE
447 values were observed between through-thickness notched specimens and surface-notched
448 specimens and between surface-notched specimens of different orientations, which could be
449 attributed to incipient crack arrester delaminations of the surface notched specimens.

450 In general, the USE values of the FeCrAl steel are higher than those of the ODS FeCrAl steels,
451 mainly due to the low volume fraction of second-phase particles (one order of magnitude
452 lower) of the FeCrAl steel, but also to the difference in UTS values and to the delamination
453 propensity of ODS steels. Figure 10 shows the difference in dimple size between the FeCrAl
454 steel and the ODS steels. Since the delamination propensity was maximal for PM 2000 steel,

which had the lowest USE value, and delamination increases in direct proportion to the aspect ratio, particular attention must be given to this parameter during material processing.

Fig. 10. Scanning electron micrographs showing the differences in ductile fracture morphology for the LT notch position at a test temperature of 300oC for (a) PM 2000, (b) MA956, and (c) FeCrAl steels.

Because both fracture mechanisms, cleavage and ductile, only compete in the DBT curves of LT and LS notched specimens of FeCrAl steel, LT and LR notched specimens of MA956 steel, and LT notched specimens of PM2000 steel, the DBTT values could be compared only for these cases. The very low DBTT of LT and LR notched specimens of ODS steels ($\approx -100^{\circ}\text{C}$) as compared to the FeCrAl steel ($\approx 90^{\circ}\text{C}$) could be attributed to differences in grain morphology and size.

The above results indicate that an understanding of the toughness anisotropy allows a good combination of high strength and high fracture toughness to be obtained in certain directions in ODS FeCrAl steels.

Final remarks. Hot extrusion of mechanical alloyed ODS FeCrAl steels leads to three types of anisotropy in the microstructure: ^[3]

- Crystallographic texture
- Submicrometric grain size with a high aspect ratio.

- Bundless of small grains with the existence of segregation/precipitation/inclusion along the extrusion direction.

Among these factors, the latter two are the main sources of the anisotropy in DBT behavior^[342]. With respect to the second factor, a submicrometric bamboo microstructure with a high aspect ratio may weaken the grain boundaries in the longitudinal direction, which may, in the case of the longitudinal loading direction of notched samples, reduce triaxiality stress state or intergranularly deflect a propagating crack with a resultant increase in fracture toughness. By contrast, in the case of the transverse loading direction, a submicrometric bamboo microstructure with a high aspect ratio would facilitate the intergranular brittle fracture and therefore would decrease the fracture toughness^[25-27, 295-297, 31-29, 352-364]. The third factor would contribute to a greater weakening of the grain boundaries which would produce a further decrease in the fracture toughness. In this context, two directions appear to be available. The first is to align the extrusion direction with the maximum tensile service stress axis. The second is to reduce the microstructural anisotropy to reduce further the anisotropy in fracture toughness. Controlling the manufacturing process using ultra-clean raw powders might reduce the anisotropy in the fracture toughness, while multidirectional processing techniques must be used to obtain a microstructure with equiaxed grains.

~~6. ACKNOWLEDGEMENTS~~

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495 | **67 CONCLUSIONS**

496 The directionality of the brittle-ductile transition of two ODS FeCrAl steels processed via
497 different routes, i.e., as a tube (PM2000) and as a bar (MA956), was analyzed and compared
498 with that of a Zr-rich particle strengthened FeCrAl alloy manufactured by high-vacuum casting.
499 The results reported in this work support the following conclusions:

- 500 1. The USE values of FeCrAl steel are higher than those of ODS FeCrAl steels
501 mainly due to the low volume fraction of second phase particles (one order
502 lower) of FeCrAl steel but also due to the differences in UTS values and to the
503 delamination propensity of ODS steels.
- 504 2. Remarkably, the DBTT of the LT and LR notched specimens of the ODS steels
505 ($\approx -100^{\circ}\text{C}$) was much lower than that of the FeCrAl steel ($\approx 90^{\circ}\text{C}$) due to
506 differences in grain morphology and size.
- 507 | 3. In the case of the ODS FeCrAl steels with an LT ~~notch-orientation~~sample, the
508 differences in USE could be partially justified by the higher volume fraction of
509 second-phase particles and more importantly by the higher delamination
510 tendency of the PM2000 steel.
- 511 | 4. For LT ~~notch-orientation~~sample, full ductile fracture with delamination was
512 observed in the case of PM2000 steel at the DBTT, defined as the temperature
513 corresponding to the average values of USE and LSE. Thus, no true transition
514 occurs in the fracture mechanism of PM2000. By contrast, a mix of cleavage
515 and ductile fracture was observed in the case of the MA956 steel.

5. The difference between the USE values for the TL and TR ~~notch~~ ~~orientations~~ samples in the ODS FeCrAl steels could be justified in the case of the PM2000 steel by the presence of crack divider delamination in the TL-notched specimens. However, this was not the case for the MA956 steel because no delamination was observed for TL-notched specimens. One may speculate that the presence of crack arrester microdelamination in the TR-notched specimens could contributed to a higher absorbed energy compared to the TL-notched specimens.

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FIGURE CAPTIONS

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Fig. 1. Orientation of the bending notched specimens relative to the product shape. a) PM2000 steel b) MA956 steel c) FeCrAl steel. 'L', 'T', 'R', 'S' and 'D' indicate longitudinal, tangential (PM2000 and MA956 steels) or transverse (FeCrAl steel), radial, short and diagonal directions respectively. The orientations of the bending specimens are designated according to ASTM standard [5] as 'LT', 'TL', 'TR', 'LS', 'TS', 'DD' and 'DS' in which the first letter indicates the specimen direction and the second the loading direction.

Fig. 2. Experimental setup of the bending test and criteria for defining transition temperature.

Fig. 3. Back-scattered SEM (PM2000 and MA956 steels) and optical (FeCrAl steel) images of the microstructures of (a),(c) and (e) transverse, and (b), (d) and (f) longitudinal cross sections of the PM2000 steel, MA956 steel and FeCrAl steel. The white arrow indicates the longitudinal direction. ((a)After [11], (b) after [7]).

Fig. 3. Back-scattered SEM (PM2000 and MA956 steels) and optical (FeCrAl steel) images of the microstructures of (a),(c) and (e) transverse, and (b), (d) and (f) longitudinal cross sections of the PM2000 steel, MA956 steel and FeCrAl steel. The white arrow indicates the longitudinal direction.

Fig. 4. Typical Zr-rich particles of the FeCrAl steel and EDS results for two particles on ductile fracture surface of FeCrAl wrought steel sample tested at 155 °C.

Fig. 5. Orientation distribution function ($\phi_2=45^\circ$ section) of the (a) PM2000 steel, (b) MA956 steel, (c) FeCrAl steel (center of the thickness), and (d) FeCrAl steel (surface). (a) and (b) After [11])

Fig. 5. Orientation distribution function ($\phi_2=45^\circ$ section) of the (a) PM2000 steel, (b) MA956 steel, (c) FeCrAl steel (center of the thickness), and (d) FeCrAl steel (surface).

Fig. 6. Effect of specimen orientation on the ductile-to brittle transition curve of (a) PM2000 steel (b) MA956 steel (c) FeCrAl steel.

Fig. 7. Selection of electron fractographs showing the relevant aspects of the fracture surfaces of single notch bend specimens of the PM2000 steel: (a) initiation of cleavage fracture (left) and details of the fracture initiation zone (right) of the LT specimen tested at -196oC; (b) microvoid coalescence zones bounded by crack divider delamination of the LT specimen tested at -60oC; (c) zones of intergranular brittle fractures bounded by crack divider delamination of the TL specimen tested at -196oC; (d) fracture initiation (left) and details (right) of the intergranular brittle fracture of the LR specimen tested at -70oC; (e) fracture initiation by microvoids coalescence followed by various crack arrester delamination and final intergranular brittle fracture of the LR specimen tested at 80 °C; and (f) schematic illustration showing delaminations geometries found in LT and LR samples tested according with Cartesian x,y,z and L,T,R (Figure 1(a)) reference systemsoC. The white arrows indicate the initiation point of the fracture.

Fig. 8. Selection of electron fractographs showing the relevant aspects of the fracture surfaces of single edge notch bend specimens of the MA956 steel: (a) location of the initiation point (left) and detail (right) of the cleavage fracture at the end of a crack divider delamination of LT

specimen tested at -196oC; (b) microvoid coalescence zones bounded by crack divider delamination of the LT specimen tested at -60oC; (c) zones of intergranular brittle fracture interspersed with thin layers of ductile fracture of the TL specimen tested at room temperature; (d) fracture initiation by microvoids coalescence followed by crack arrester delamination and final intermixed zones of cleavage and ductile fracture modes of the LR specimen tested at -20oC; (e) intermixed zones of intergranular brittle and ductile fracture modes with occasional crack arrester delamination of the TR specimen tested at -20oC. The white arrows indicate the initiation point of the fracture.

Fig. 9. Selection of electron fractographs showing the relevant aspects of the fracture surfaces of single edge notch bend specimens of the FeCrAl steel: (a), (b) initiation of cleavage fracture in Zr-rich particles of the LT specimen tested at -20oC and TL specimen tested at 60oC respectively (see the arrow); (c), (d) macroscopic image of ductile fracture with crack divider delamination and detailed micrograph showing ductile fracture nucleation in Zr-particles of LT specimen tested at 150oC ; (e), (f) fracture initiation by coalescence of microvoids followed by crack arrester delamination and final cleavage fracture with small areas of ductile fracture of the DS specimen tested at 80oC and the TS specimen tested at 60oC respectively. The white arrows indicate the initiation point of the fracture.

Fig. 10. Scanning electron micrographs showing the differences in ductile fracture morphology for the LT notch position at a test temperature of 300oC for (a) PM 2000, (b) MA956, and (c) FeCrAl steels.

Tables:

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Table 1. Chemical composition of steels investigated (wt%).

<u>Material</u>	<u>C</u>	<u>Si</u>	<u>Mn</u>	<u>P</u>	<u>S</u>	<u>Cr</u>	<u>Al</u>	<u>Ti</u>
<u>PM2000</u>	<u><0.01</u>	<u>0.095</u>	<u>0.037</u>	<u>0.004</u>	<u><0.005</u>	<u>18.6</u>	<u>5.2</u>	<u>0.54</u>
<u>MA956</u>	<u>0.012</u>	<u>0.12</u>	<u>0.097</u>	<u>0.005</u>	<u>0.007</u>	<u>19.33</u>	<u>4.7</u>	<u>0.44</u>
<u>FeCrAl</u>	<u>0.026</u>	<u>0.30</u>	<u>0.18</u>	<u>0.010</u>	<u>0.0004</u>	<u>21.0</u>	<u>4.8</u>	<u>0.05</u>
<u>Co</u>	<u>Ni</u>	<u>Cu</u>	<u>O</u>	<u>Y</u>	<u>Zr</u>	<u>N</u>	<u>H(ppm)</u>	
<u>0.039</u>	<u>0.03</u>	<u>0.015</u>	<u>0.091</u>	<u>0.391</u>	<u>-----</u>	<u>0.003</u>	<u>11.0</u>	
<u>0.066</u>	<u>0.101</u>	<u>0.021</u>	<u>0.108</u>	<u>0.376</u>	<u>-----</u>	<u>0.02</u>	<u>11.0</u>	
<u>0.02</u>	<u>0.22</u>	<u><0.01</u>	<u>-----</u>	<u>-----</u>	<u>0.19</u>	<u>0.014</u>	<u>4.1</u>	

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Table 2. Geometric measures of microstructural features and texture.

Material	Grains		Second phase particles		Texture	
	D_{avg} , μm	l , μm	GAR	Size, nm		f_{vol} , %
PM2000	0.7 ^[11]	1.6 ^[11]	2.3 ^[11]	19 ^[11]	1.1 ^[11]	(100)[110]S ^[11]
MA956	0.7 ^[8]	0.9 ^[8]	1.3 ^[8]	16 ^[8]	1.3 ^[8]	<110>HED S ^[8]
FeCrAl	17.3	26	1.5	2350	0.15	(100)[110]W ^(*)

D_{avg} stands for average diameter; l stands for length; GAR is grain aspect ratio; f_{vol} stands for volume fraction; S stands for strong; W stands for weak

^(*) Texture in the centre of FeCrAl sheet

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Table 3. Tensile test data. 'L', 'D' and 'T' stand for longitudinal, diagonal and transverse orientations of the specimen. Values for MA956 after Ref. [11] and values for PM2000 after Ref. [7].

MATERIAL	T, °C	YS, MPa	UTS, MPa	A _y , %	A _t , %	n
PM2000	-196	1430	1530	9.5	10	0.041
	-60	1100	1230	10.2	16	0.053
	-20	1050	1200	9.2	14.1	0.058
	L 20	1000	1170	8.5	14.8	0.064
	150	930	1100	9.1	13.1	0.067
MA956	300	820	1050	8.7	12.6	0.088
	-196	1405	1550	14	21.1	0.054
	-80	1100	1260	11.8	18	0.061
	-60	1070	1250	11	16.9	0.065
	L 20	970	1150	10.2	15	0.068
FeCrAl	150	870	1020	9.1	12.6	0.065
	300	820	930	8.5	13	0.055
	-196	-----	1030	0	0	-----
	-70	700	787	12.5	12.5	0.057
	-32	602	725	14.5	27.5	0.076
FeCrAl	L 20	560	664	11	27.8	0.069
	162	440	590	13.1	19.1	0.101
	400	420	549	13.2	25.5	0.094
	-196	-----	948	0	0	-----
	-70	755	860	13	21.2	0.061
FeCrAl	I -27	654	780	13.1	25.2	0.072
	20	614	731	9.3	20	0.069
	140	470	645	13.5	20.5	0.106
	400	455	595	10	19.5	0.094
FeCrAl	-196	-----	1010	0	0	-----
	-70	710	790	12.8	14.1	0.055
	-32	610	730	10	22.1	0.071
	D 20	567	675	10.9	26.8	0.070
	150	460	595	13	19.9	0.092
FeCrAl	400	418	544	10.1	23.5	0.092

YS stands for yield strength; UTS stands for ultimate tensile strength; A_y stands for uniform elongation; A_t stands for total elongation, n stands for strain hardening exponent; T stands for temperature.

Table 4. Bending data of the parameters of the DBT curves

<u>MATERIAL</u>	<u>ORIENTATION NOTCH</u>	<u>LSE J</u>	<u>USE J</u>	<u>DBTT °C</u>	<u>FRACTURE MODE (*)</u>
<u>PM2000</u>	<u>LT</u>	<u>0.19</u>	<u>2.5</u>	<u>18</u>	<u>C → D+cdd → D</u>
	<u>LR</u>	<u>0.23</u>	<u>4.5</u>	<u>30</u>	<u>I → I+D+cad → D</u>
	<u>TL</u>	<u>0.16</u>	<u>1.7</u>	<u>50</u>	<u>I → I+D+cdd → D</u>
	<u>TR</u>	<u>0.20</u>	<u>3.2</u>	<u>50</u>	<u>I+cad → I+D → I+D</u>
<u>MA956</u>	<u>LT</u>	<u>0.30</u>	<u>4.5</u>	<u>-110</u>	<u>C → D+cdd → D</u>
	<u>LR</u>	<u>0.13</u>	<u>4.1</u>	<u>-80</u>	<u>C → C+cad → D</u>
	<u>TL</u>	<u>0.21</u>	<u>1.7</u>	<u>60</u>	<u>I → I+D → I+D</u>
	<u>TR</u>	<u>0.17</u>	<u>3.2</u>	<u>90</u>	<u>I → I+D → I+D</u>
<u>FeCrAl</u>	<u>LT</u>	<u>0.13</u>	<u>5.6</u>	<u>70</u>	<u>C → C+D+cdd → D</u>
	<u>LS</u>	<u>0.12</u>	<u>8.8</u>	<u>90</u>	<u>C → C+D+cdd → D</u>
	<u>TL</u>	<u>0.13</u>	<u>5.4</u>	<u>65</u>	<u>C → C+D+cad → D</u>
	<u>TS</u>	<u>0.14</u>	<u>6.6</u>	<u>75</u>	<u>C → C+D+cad → D</u>
	<u>DD</u>	<u>0.11</u>	<u>5.2</u>	<u>65</u>	<u>C → C+D+cdd → D</u>
	<u>DS</u>	<u>0.10</u>	<u>7.0</u>	<u>70</u>	<u>C → C+D+cad → D</u>

LSE- Lower Shelf Energy; USE- Upper Shelf Energy; DBTT- Ductile-to-Brittle-Transition Temperature; C- Cleavage; D- Ductile; I- Intergranular; cdd-crack divider delamination; cad-crack arrester delamination

(*) for example: C → C+D+cad → D, cleavage in LSE, cleavage + ductile + crack arrester delamination in the DBT, ductile in USE

RD // L ; ND//R ; TD//T

(a)

RD // L ; ND//R ; TD//T

(b)

RD // L ; ND//S ; TD//T

(c)

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
B K	2.87	0.5382	13.61	2.45	27.14
C K	2.59	0.2012	32.78	2.28	58.85
Ti K	0.22	0.8281	0.66	0.15	0.30
Cr K	0.70	0.8714	2.04	0.22	0.84
Fe K	1.91	0.8846	5.52	0.38	2.13
Zr L	16.40	0.9212	45.40	1.97	10.73
Totals			100.00

Element	App Conc.	Intensity Corrn.	Weight%	Weight% Sigma	Atomic%
C K	2.02	0.2285	28.74	2.48	73.19
Ti K	0.18	0.8364	0.70	0.19	0.45
Cr K	0.97	0.8969	3.50	0.30	2.06
Fe K	2.40	0.9172	8.52	0.51	4.67
Zr L	16.38	0.9092	58.53	2.11	19.63
Totals			100.00

(a)

(b)

(c)

(a)

(b)

(c)

(d)

(e)

(f)

(a)

(b)

(c)

(d)

(e)

(a)

(b)

(c)

(d)

(e)

(f)

(a)

(b)

(c)