FRACTURE TOUGHNESS OF HIGH-DENSITY SINTERED STEELS

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ABSTRACT

Yield stress, tensile strength and fracture toughness of a PM steel (Fe-4.0Ni-1.5Cu-0.5Mo-0.5C) have been studied as a function of sintered density in the high-level range, from 7.22 to 7.51 g/cm^3. The material was processed by different routes combining both cold and warm compaction with single and double pressing-sintering sequences. Fracture toughness was tested by mode I crack opening in a three-point bending test in accordance with the standard procedure ASTM E-399. Validity of fracture toughness tests in these materials is discussed. A quantitative analysis of porosity was correlated with tensile and fracture parameters to assess the influence of pore morphology on mechanical behavior. Microstructural characterization reveals that pores tend to be bigger and more clustered at lower densities. All the mechanical properties increased with a reduction in porosity, being this conjugated performance a remarkable advantage over wrought steels. In spite of using three different lubricants in the processing routes involved, it is found that mechanical properties depend solely on the attained sintered density regardless the route followed to achieve it.

KEY WORDS: Fracture Toughness, Sintered Steel, High-Density

RESUMEN

Este trabajo presenta el estudio del límite elástico, la resistencia a tracción y la tenacidad de fractura de un acero sinterizado (Fe-4.0Ni-1.5Cu-0.5Mo-0.5C) en función de su densidad, en el rango de 7.22 a 7.51 g/cm^3. Para obtener los diferentes niveles de densidad se emplearon seis rutas diferentes de procesamiento, en las cuales se ha variado la temperatura de compactación, el tipo de lubricante, la temperatura de sinterización y el número de etapas de compactación-sinterización. La tenacidad de fractura ha sido evaluada por ensayos de flexión en tres puntos con probetas tipo SENB siguiendo la norma ASTM E-399. Se discute la validez de los ensayos en este tipo de materiales. Se ha encontrado que todas las propiedades mecánicas del material aumentan con la densidad del sinterizado, lo cual representa un comportamiento ventajoso comparado con el de materiales macizos comúnmente utilizados en aplicaciones estructurales. Los resultados de la caracterización microestructural muestran que al aumentar la densidad, el tamaño de los poros disminuye y su distribución mejora. Adicionalmente se determina que la ruta de procesamiento empleada no influye significativamente en los resultados finales.

ÁREA TEMÁTICA PROPUESTA: Fractura de Materiales Metálicos y Hormigón.

PALABRAS CLAVE: Tenacidad de Fractura, Acero Sinterizado, Alta Densidad.

1. INTRODUCTION

Sintered steels are increasingly applied because the low production costs in large series associated with the powder metallurgy (PM) processing route. This economic benefit is granted by a near net shape and low raw material loss. In recent years, with the advent of emerging processing technologies as well as an attendant increase in material reliability, the use of PM steels has been also growing into applications that imply higher operating stresses, either static or cyclic. This is particularly true for high-density sintered steels, materials for which information and documentation on microstructure – mechanical properties relationships may still be considered scarce.

Mechanical properties of sintered materials are influenced by several factors, being density level and internal structure of pores two of the most important ones [1,2]. It is widely known that density has a dramatic effect upon mechanical behaviour [1-9]. Regarding fracture toughness-strength correlation for sintered steels, a literature review reveals significant differences in their behaviour when compared with homogeneous materials. While in wrought materials an inverse relationship of toughness with yield stress is well established; in PM materials a positive correlation between fracture toughness and density is usually reported [4,10-14], being this performance a significant structural advantage once the accompanying

improvement in tensile properties by density is accounted. These previous studies have found the referred tendency to be valid up to relative densities of about 94% when it is expected that behaviour of sintered steels become similar to that exhibited by fully dense materials.
It is the aim of this study to asses the relationship between fracture toughness and tensile properties in PM steel at high-density levels in order to explore whether this advantageous performance trend is maintained up to density levels closer to free-porosity materials.

2. EXPERIMENTAL PROCEDURE

Material evaluated is a Fe-4.0Ni-1.5Cu-0.5Mo-0.5C alloy processed by different routes. Three different lubricants were used. Both cold compaction (CC) and warm compaction (WC) were employed. Six densities were achieved combining different mixes and processing routes, as summarized in Table 1. The higher-level densities were reached using warm compaction and double pressing-double sintering process (2P2S - WC). In 2P2S routes, the first sintering was performed at 800ºC for 20 min and the second one at 1120ºC for 30 min. These latter conditions were also used in single-pressing-single-sintering (1P1S) routes. In all cases, sintering was carried out in a H_2-N_2 atmosphere.

Two different types of test pieces were compacted and sintered: tensile specimens with a "dog-bone" geometry and fracture toughness samples with a prismatic geometry (10x5x55mm) for testing under three-point bending.

Density was determined by recourse to Archimedes principle. Metallographic specimens of all tested materials were analyzed to evaluate pore structure, shape and distribution using a conventional image analysis system. Tensile tests were conducted in a servohydraulic testing machine at a cross head displacement speed of 1 mm min^{-1}. Strain was measured using a 25-mm extensometer. The 0.2% offset yield stress (YS) and the ultimate tensile stress (UTS) were determined. Loading direction was perpendicular to the pressing one. Fracture toughness was tested by mode I crack opening in a three-point bending test in accordance with the standard procedure ASTM E-399. Crack initiating slots were made by machining. Fatigue precracks were grown up to 0.45≤a/W≤0.55 (***a*** is crack length and ***W*** is height of test piece) using a resonance testing machine at working frequencies about 100 Hz. Final fracture was generated by driving the central loading point at 100 N seg^{-1}; while crack opening displacement was measured using a clip gauge. Fracture surfaces were examined using a scanning electron microscope.

3. RESULTS AND DISCUSSION

Density and porosity

As expected, mixes processed by 2P2S showed higher densities, 0.2 g/cm^3 in average, than those processed by 1P1S with identical lubricant (see Table 1). Regarding compaction route, WC samples exhibit improved densities in comparison with the CC ones. Heating during compaction in WC routes reduces the yield stress of the powder particles. As a consequence, particle deformation is enhanced and more densification is achieved.

#	Base Powder	Lubricant	Compaction Route	Sintering Temp. [ºC]	Density [g/cm^3]
1	Distaloy AE + 0.5%C	0.6% Wax	1P1S - CC	1120 ºC	7.22
2			2P2S - CC	800 / 1120 ºC	7.40
3	Densmix + 0.5%C	Admixed	1P1S - WC	1120 ºC	7.29
4			2P2S - WC	800 / 1120 ºC	7.51
5	Distaloy AE + 0.5%C	0.58%PE + 0.02% Ad	1P1S - WC	1120 ºC	7.25
6			2P2S - WC	800 / 1120 ºC	7.48

Table 1. Materials and processing routes evaluated.

Optical micrographs revealed differences in porosity morphology in terms of pore size, shape and distribution (figure 1). Image analysis of the pore distribution (for pores whose equivalent area was higher than 25 μm^2) points out that they tend to be more clustered at lower densities. Within the higher density level, pores appeared to be smaller (figure 2a) and more homogeneously distributed. Hence, average distance between pores (neck size) increases with density (figure 2b). In general, as pore size decreases the corresponding pore shape becomes slightly more spherical.

(a)
(b)

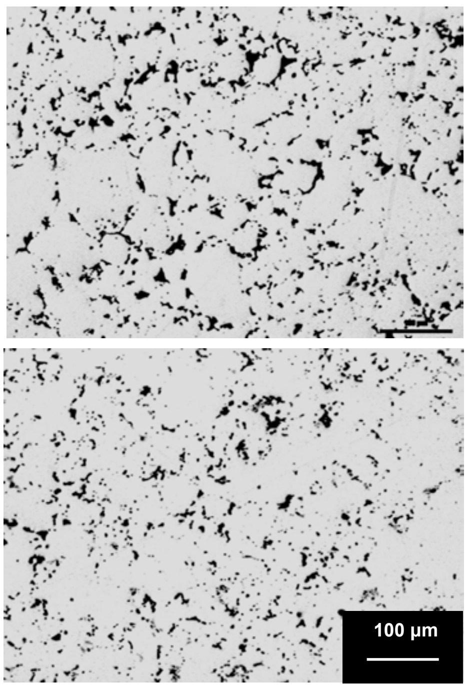

Figure 1. Typical porosity in (*a*) material 5, *ρ= 7.29 g/cm^3*; (*b*) material 6, *ρ= 7.51 g/cm^3*.

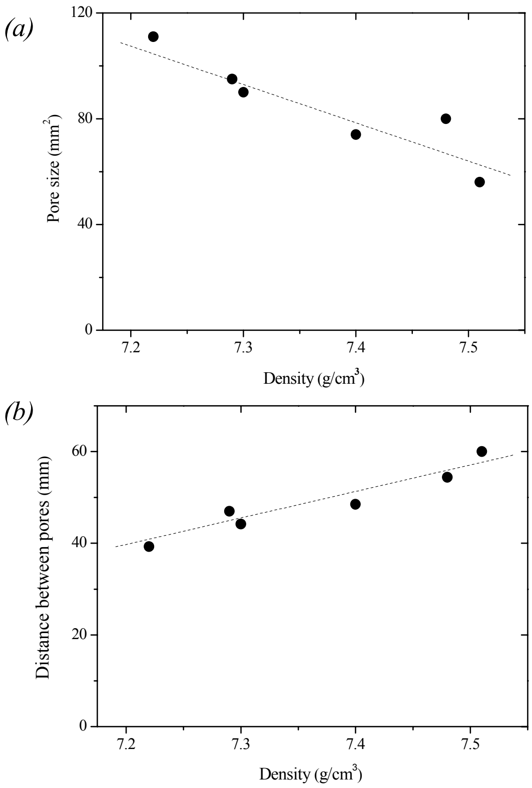

Figure 2. Porosity morphology as a function of density (*a*) pore size; (*b*) distance between pores.

Tensile properties

The average yield and ultimate tensile stresses were found to increase with density as shown in Fig. 3. The relationship observed between tensile properties and density is roughly linear. From the results, it may be indicated that the measured properties are independent of the processing routes by which similar densities are achieved, in agreement with previous works reported in the literature [13,15].

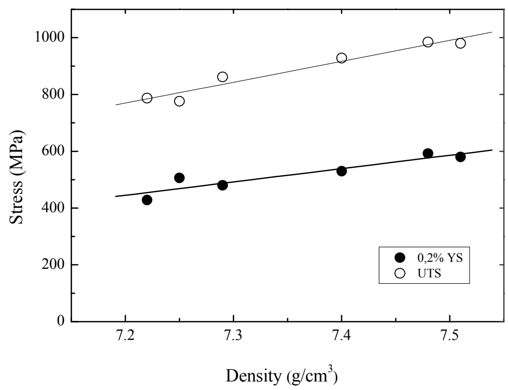

Figure 3. Effect of density on tensile properties.

Regarding density effects on strength, it may be estimated that every 0,1 g/cm^3 rise in density approximately results in a 48 and 77 MPa increase in YS and UTS, respectively.

Attempting to rationalize the strength-density dependence, relative strength increment, $\Delta\sigma/\sigma$, was calculated for each processing route studied and compared with $\Delta\sigma/\sigma$ estimated by just accounting effective load-bearing section (determined assuming spherical pores [16]). The results (see Table 2) clearly indicate that the enlargement of the cross-sectional area by reduction in porosity does not fully explain the correlation. Yield stress values are higher than expected, and strain hardening as well as notch strengthening effects at the microstructural scale (e.g. refs. [17,18]) are speculated to be possible reasons for this discrepancy. Such a rationalization is based on the fact that the interpore regions, where plastic deformation takes place, may be regarded as a set of small circumferentially notched specimens [10] and notched samples of ductile materials endure higher stresses as a consequence of the triaxiality of the stress state in front of the notch root which constraints the plastic deformation.

Processing Route	Materials	$\Delta\rho$ [g/cm^3]	$\Delta\sigma_y/\sigma_y$ experimental [%]	$\Delta\sigma_y/\sigma_y$ estimated [%]
A	$1 \rightarrow 2$	0.18	23.8	6.0
B	$3 \rightarrow 4$	0.22	20.8	7.6
C	$5 \rightarrow 6$	0.23	17.0	8.0

Table 2. Relative Yield Stress increment for each processing route studied.

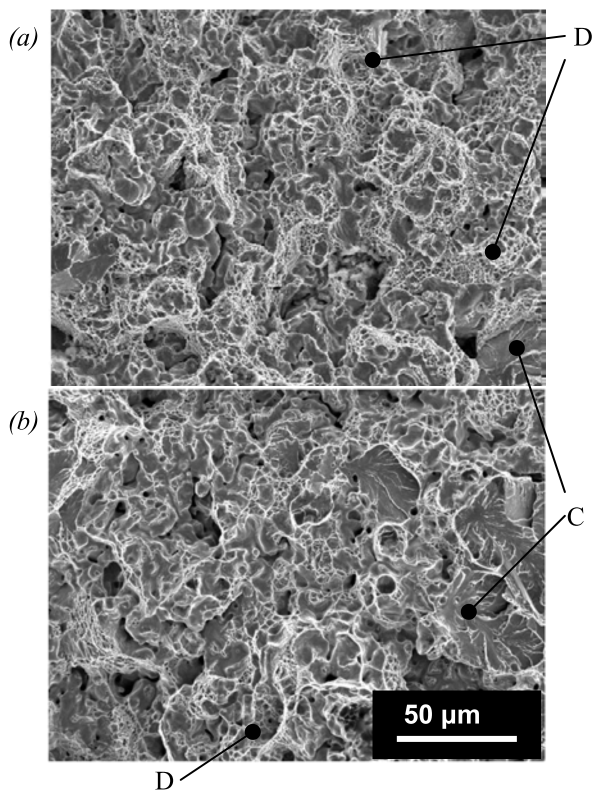

Figure 4. SEM micrograph showing dimples (D) and cleavage (C) on the fracture morphology of (*a*) material 1 (ρ = 7.22 g/cm^3) and (*b*) material 4 (ρ = 7.51 g/cm^3).

From this perspective, it should be noted that fracture is observed to occur by appreciable plastic deformation of sintered necks, as reflected by the ductile nature of the fractographic features of these load bearing units, i.e. dimples resulting from microvoid coalescence (Figure 4a). Nevertheless, as density rised, transgranular particle fracture was also found to take place (Figure 4b) indicating the increasing role played by the intrinsic microstructure of sintered steels as porosity is reduced.

Fracture Toughness

Table 3 gives fracture toughness data and also the results of the validation criterion a and $B > 2.5\ (K_Q/\sigma_y)^2$ where a is the crack length, B is the specimen thickness, K_Q is the fracture toughness and σ_y is the yield stress. Also the precrack length was checked to be within 0.45≤a/W ≤0.55 and maximum load F_{max} of the crack opening displacement-load curve to be lower than 110% of F_Q. All data are the average of at least four samples.

#	Sintered Density [g/cm^3]	Yield Stress σ_y [MPa]	Fracture Toughness, K_Q [MPa m$^{1/2}$]	$2.5\ (K_Q/\sigma_y)^2$ [mm]	F_{max}/F_Q	a/W
1	7,22	428	27.50	10.32	1.29	0.475
2	7,40	530	32.30	9.28	1.20	0.491
3	7,29	480	28.50	8.81	1.25	0.480
4	7,51	580	34.10	8.64	1.18	0.478
5	7,25	506	31.60	9.75	1.13	0.496
6	7,48	592	36.70	9.60	1.15	0.470

Table 3. Results and validation criteria of fracture toughness tests.

Tests results failed to fulfil the plane-strain criteria required for valid K_{IC} measurement in all the cases. A number of researchers have found the same problem [e.g refs. 12,19]. Taking into consideration that fracture in sintered steels is a successive rupture of sintering necks, the thickness requirement can be neglected because the constraint on plastic flow is defined by the pore spacing rather than the specimen thickness. However, the ratio F_{max}/F_Q is difficult to remain lower than 1.1 in sintered steels, especially at lowest density levels where ductile nature of sintering necks fracture is dominant. Figure 5 shows typical load-crack opening displacement curves for three density levels which provide evidence of an excessive plastic deformation before fracture.

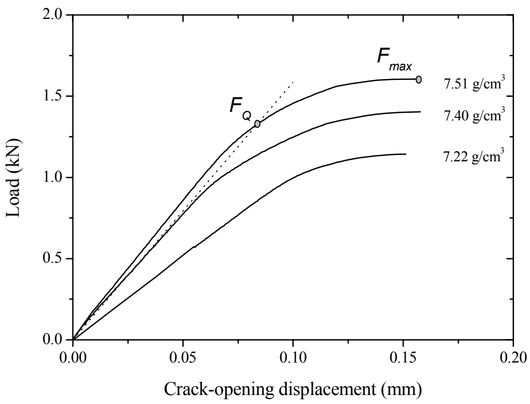

Figure 5. Load-crack opening displacement curves.

Furthermore, crack growth front was found to be nearly straight for all samples (as shown in figure 6), a finding in agreement with the idea of fracture toughness being independent of specimen dimensions for sintered steels [5,19]. This experimental fact would indicate that for sintered steels, although all ASTM requirements for a valid plane-strain result are not satisfied, K_Q may be used as a good estimate to evaluate fracture behaviour.

Within this framework, the experimental data here attained was compared in terms of a crack growth resistance parameter, K_Q, defined in accordance to ASTM standard E-399.

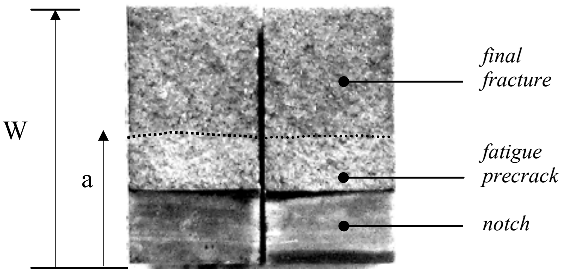

Figure 6. Fracture surface showing a nearly straight precrack front; a crack length; W specimen height.

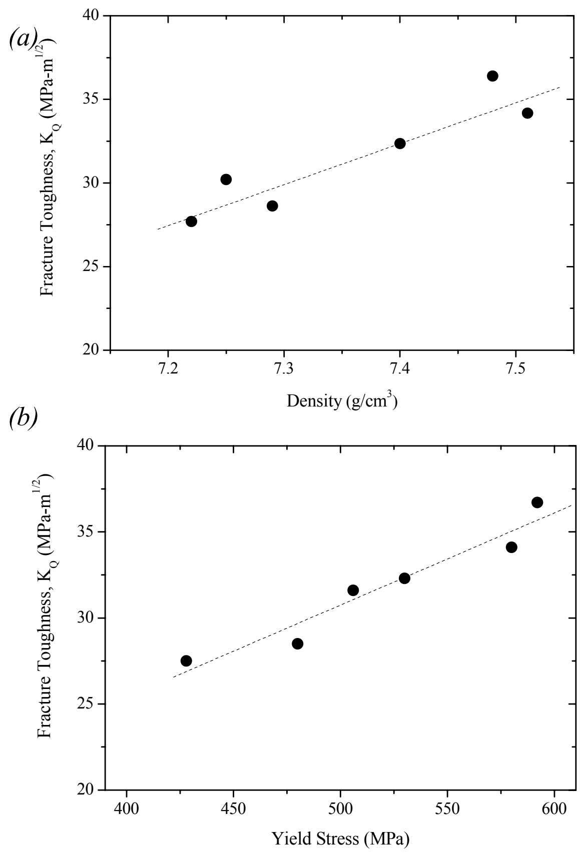

Figure 7. Relationship of fracture toughness with (*a*) density, and (*b*) yield stress.

Fracture toughness-like values, K_Q, raised as the density was increased from 7.22 to 7.51 g/cm^3. From figure 7a, a linear relationship between fracture toughness and density can be outlined. Such a trend is similar to the one discerned between yield strength and density and allows one to clearly state that the advantageous and well-established direct correlation between strength and toughness for low- and mid-density sintered steels can be extended into the high density regime, at least up to 7.50's g/cm^3 values (Figure 7b). Hence, it may be pointed out that, even under quite reduced porosity conditions, enhanced plastic deformation at sintering necks is still significant enough to continuously shield crack growth. On the other hand, yield strength rises because the high effective load bearing section associated with increasing mean distance between adjacent pores and the notch effect of pores.

4. CONCLUSIONS

The tensile properties and fracture toughness of sintered steels of composition Fe-4.0Ni-1.5Cu-0.5Mo-0.5C over a high-density range was investigated. The following conclusions can be drawn:

1. The mechanical properties evaluated increased linearly with sintered density, regardless of the processing route followed for achieving a given density level.
2. Fracture toughness exhibits a continuous increment with yield stress even at high-density levels, which is a remarkable advantage of PM over their dense counterparts.
3. The above experimental findings are directly related to the still ductile character of the sinter necks, even for sintered densities as high as 7.50 g/cm^3.

ACKNOLEWDGEMENTS

This investigation was supported by MCYT under grant Profit 0300000-2003-63 and by CDTI under grant 03-0369. Technical assistance provided by Dr. Y. Torres is gratefully acknowledged. One of the authors (J.B.) would like to express his gratitude to COLCIENCIAS and Universidad del Norte (Colombia) for the Ph.D. scholarship.

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