

Microstructure and mechanical properties of cold drawn AISI 1016 steel processed by ECAP

D.M. Marulanda✉, J. Cuellar, C. Rojas, L.M. Acosta

Abstract

Low carbon cold drawn steel was deformed using equal channel angular pressing to evaluate its mechanical properties and microstructure, while assessing the possibility of improving properties beyond the cold drawn process. We used low carbon cold drawn steel with a composition of 0.16% C, 0.8% Mn, 0.2% Si, 0.02% P, 0.012% S and Fe balance. The process was carried out at room temperature and four passes at route Bc with a deformation of ~ 0.6 in each pass. Using scanning electron microscopy and x-ray diffraction, we evaluated the evolution of the structure before and after deformation as well as the mechanical properties of microhardness and tensile strength. A slight increase in the mechanical properties occurred when the number of passes was increased. There were changes in the original ferritic-pearlitic structure with the refinement of ferrite grains and pearlite deformation.

Keywords: Equal channel angular pressing; ECAP; low carbon steel; structure refinement; tensile strength; microhardness.

Introduction

AISI 1016 steel is an inexpensive low-carbon material that is used in manufacturing of machine parts that are not subjected to high mechanical stresses, such as shafts, chain links, pins, cemented bushes, standard screws, flanges and gears for low-stress chain drives (Selçuk et al. 2003). Surface properties of this steel can be improved by carburizing, carbonitriding or boronizing (Selçuk et al. 2003, Icziler & Tabur 2006), which entails the formation of a surface layer that improves properties such as friction, wear and corrosion resistance. However, these techniques only treat the surface of the material.

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On the other hand, the bulk properties of this steel are modified using the drawing process, which consists in reducing the section of a rod by pulling it through a conical die (Atienza et al. 2005), involves a plastic deformation of the material. In drawing deformation, the tensile strength and hardness of the steel increase, while ductility and impact resistance decrease. This drawing process is considered to be one of the most effective and flexible methods to improve surface finish, to obtain precise dimensions and specific mechanical properties (Raji & Oluwole 2011). In addition to the processes mentioned before, there are other processes that modify the steel's properties by changing its internal structure; this is called, severe plastic deformation (SPD). In severe plastic deformation, materials are subjected to enormous strains that yield particle sizes in the range of 100-1000 nm and structures such as subgrains, dislocations and crystallites in the range of $\sim 1 - 100$ nm; hence they are considered nanostructured materials (Zhu et al. 2004). Among the severe plastic deformation techniques (Langdon 2008), equal channel angular pressing (ECAP) has gained attention due to the possibility of producing full, dense and imporous bulk materials with sub-micron or even nano-scale grains (Fan et al. 2013).

Based on the preceding, the objective of this study is to demonstrate the influence of the ECAP process when it is performed on low-carbon cold drawn steel and evaluate its microstructure and mechanical properties to identify the possibility of further improving its properties after the drawing process.

Materials and methods

The material used was AISI 1016 low carbon steel of composition 0.16% C, 0.8% Mn, 0.2% Si, 0.02% P, 0.012% S and balance Fe where expressed in wt.%. We used 6 mm diameter and 50 mm long commercial steel rods cold drawn at 55%. The die used for the ECAP process was a split die manufactured with D2 tool steel, with a channel having an inner angle $\Phi = 120^\circ$ and an outer angle $\Psi = 30^\circ$, which yielded an equivalent strain of ~ 0.6 after a single pass. The rods and the channel were lubricated with molybdenum disulfide (MoS_2). The process was performed at room temperature using route Bc and completing up to four passes at a processing speed of 5 mm/min.

Following ECAP, samples were taken from the Y plane of the deformed rods, which is the plane parallel to the lateral faces at the die outlet. Microstructural evolution was observed using scanning electron microscopy (SEM) in a JEOL model JSM 6490-LV SEM working at 10 kV. Phase changes and crystallite size were studied using X-ray diffraction (XRD) in an X'PertPro Panalytical diffractometer working with the following settings: θ 2 θ varying from 10° to 90° with 0.02° step size, monochromatic $\text{CuK}\alpha$ radiation with wavelength of $\lambda = 1.5409 \text{ \AA}$, 45 kV and 40 mA.

Tensile testing was performed before and after the deformation process using a HUNG-TA universal testing machine model HT. Samples along the longitudinal axis of 6 mm diameter and 24 mm gage length were used according to the ASTM E8/E8M-11 standard, which indicates that on round specimens, the gage length is equal to four times the nominal diameter. Data were obtained in tests carried out at a speed of 5 mm/min and the results are the average of three measurements. Vickers microhardness was measured using an Esseyway model 600 hardness tester at a load of 50 kg F and 20 s dwell time. The reported values are the average of 30 readings, which were taken on the sample surface from the Y plane of the deformed rods, consistent with X-ray diffraction measurements.

Results

In the diffraction patterns for the samples before and after ECAP with different numbers of passes, only ferrite was observed due to the low-volume fraction of pearlite in the steel, in orientations (110), (200) and (211), according to database JCPDS 00 006 0696 (**Figure 1**). No phase transformation occurred after ECAP; however, there was a change in peak intensity and orientations even after one pass, which could indicate that a structural refinement is produced, as a result, of Fe (110) grain subdivision (Gholinia et al. 2000), producing finer grains in orientations (200) and (211).

The peak broadening presented for peaks Fe (110) and Fe (200) may also be attributed to the structural refinement in the orientation (**Figure 2**). We observed

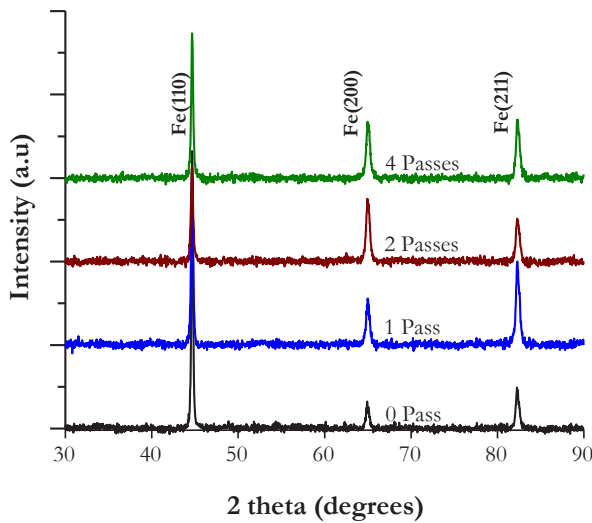


Fig. 1. XRD patterns for the samples before and after ECAP.

(Figure 2a) that the (110) peak intensity decreased after one pass while (200) peak intensity increased, which could be due to Fe (110) grain subdivision and the formation of new grains in direction (200). The same behavior was observed for orientation (211).

To assess structural refinement, the average grain size, B , was calculated using the Scherrer's formula and the full width at half maximum (FWHM) for each pass. X-ray diffraction was used to estimate crystallite size; many properties of materials depend on crystallite size, and this does not always coincide with particle size or grain size measured with other techniques (Uvarov & Popov 2013). Scherrer's formula was used to calculate crystallite size (**Equation 1**)

$$B = \frac{K\lambda}{L \cos \theta} \quad (1)$$

Where: B is the apparent crystallite size, λ is the X-ray wavelength, L is the width of the peak (FWHM in radians) and θ is the Bragg angle. K is a dimensionless constant, whose value depends on crystal shape and crystallite size distribution, and the cubic shape is commonly used with a value of $K = 0.94$. The crystallite size calculated using the Scherrer's formula was generally different to particle size or grain size, because individual particles or

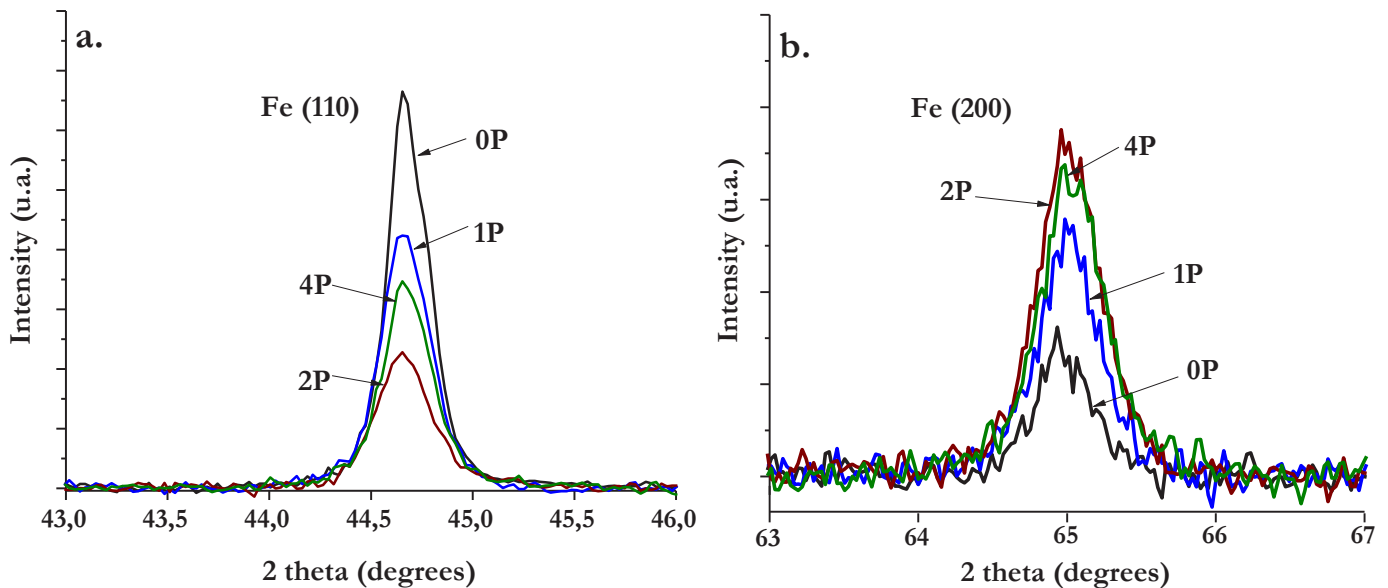


Fig. 2. Detailed XRD patterns for the peaks. **a.** Fe (110) and **b.** Fe (200). 0P: 0 pass, 1 P: 1 pass, 2 P: 2 passes, 4P: 4 passes.

grains can contain several crystallites or domains with different orientations (Uvarov & Popov 2013). On the other hand, the crystallite size in SPD metals obtained by X-ray diffraction was equivalent to the mean size of domains that scatter X-rays coherently. Consequently, X-ray diffraction makes a difference between subgrains even if the misorientations are very small (Gubicza et al. 2005).

The results obtained for crystallite size indicate a structural refinement especially in orientation Fe (100) (Table 1).

Tabla 1. Tamaño de cristalito calculado para la fase ferrita.

Orientation	Number of passes	Crystallite size (nm)
110	0	83,06
	1	68,47
	2	61,50
	4	69,00
200	0	41,60
	1	41,10
	2	37,15
	4	38,58
211	0	47,94
	1	45,54
	2	42,15
	4	44,85

The microstructure of cold-drawn steel consists of ferrite grains and pearlite colonies that are easily discernable (Figure 3a). Deformation of the microstructure was observed after the first pass possibly due to ferrite and pearlite grain subdivision. Subdivided pearlite colonies and ultrafine ferrite grains with a high density of lattice dislocations characterize the microstructure of low-carbon steel processed by ECAP (Son et al. 2005).

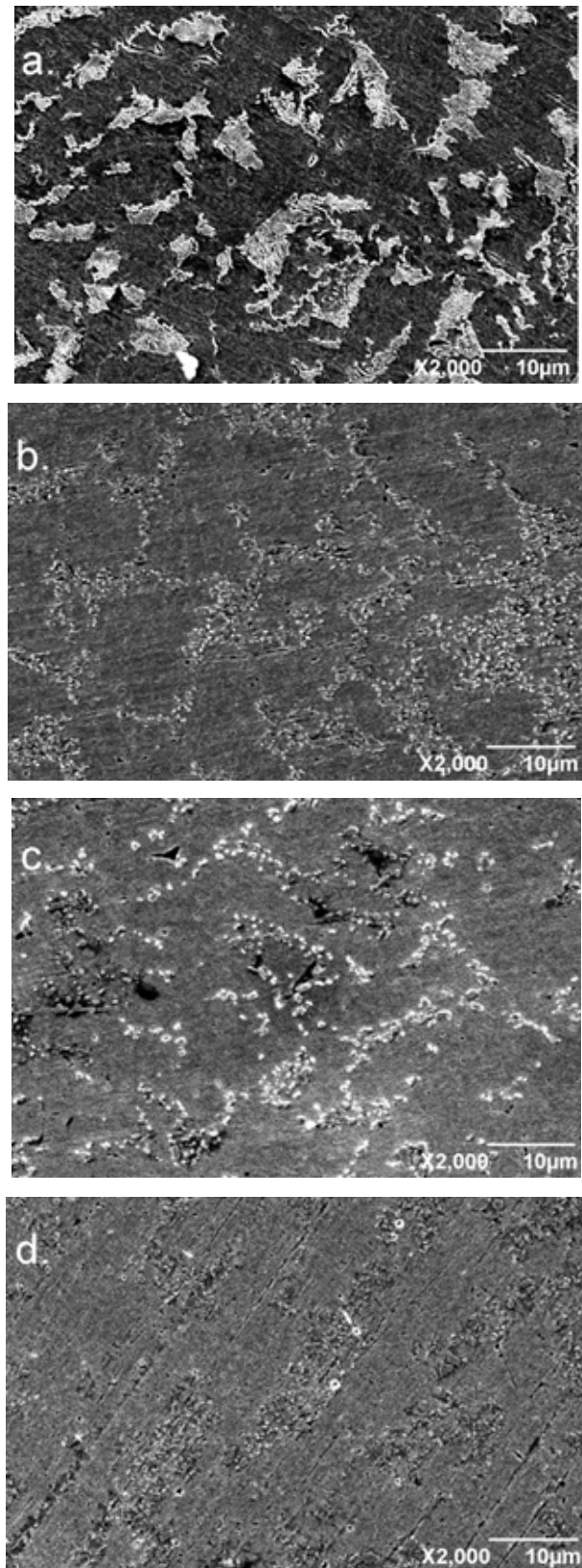


Fig. 3. SEM images of the samples before and after the ECAP process. **a.** 0 pass, **b.** 1 pass, **c.** 2 passes, **d.** 4 passes.

The tensile strength of the cold drawn steel, before the ECAP process, was in the range reported for commercial cold drawn AISI 1016 steel. The tensile strength increased up to the second pass, after four passes, it decreased below the initial value (**Figure 4**).

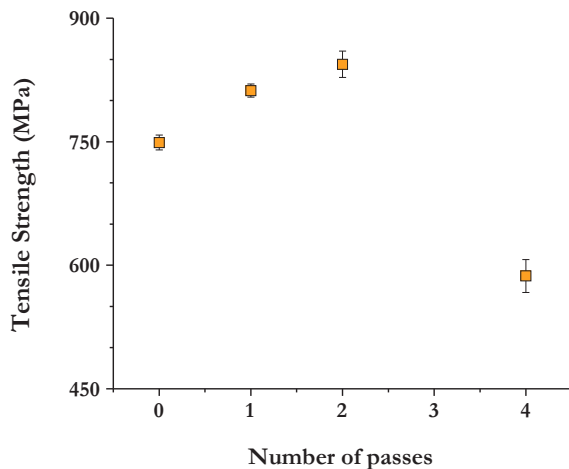


Fig. 4. Variation of tensile strength after the ECAP process.

The Vickers microhardness for the steel before processing is also comparable to the values reported for cold-drawn steel and the values gradually increase with the number of passes (**Figure 5**).

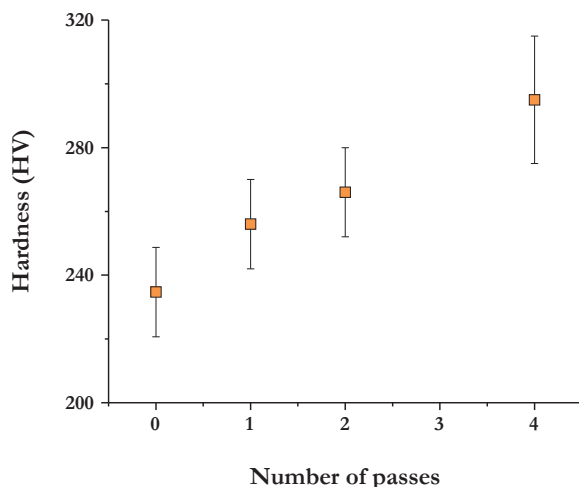


Fig. 5. Variation of hardness after the ECAP process.

Discussion

The results obtained through XRD indicate that a refinement of the ferrite crystallites in low extension is possible even after the drawing process. During ECAP, grains are refined due to the high plastic deformation they are subjected to in each pass. For this particular case of study it is expected that ferrite, which is softer than pearlite, undergoes higher plastic deformation in its microstructure (Al Abbasi 2013). Moreover, in the early stage of ECAP it is expected that ferrite should be refined by successive subdivision of dislocation walls due to the operation of multi-slip systems (Chen et al. 2012). The results obtained for crystallite size (Table 1), indicated a structural refinement, especially in orientation Fe (110), which decreased to 69 nm from 83 nm of the steel in the initial state. These values for crystallite size agree with values reported for low-carbon steels (Liu et al. 2001, Suzuki et al. 2009) and Iron (Tao et al. 2002), and also for other metals subjected to different severe plastic deformation techniques, such as aluminum (Chang et al. 2001) and aluminum alloys (Bera et al. 2013).

SEM images indicate that after two passes the pearlite is severely distorted due to the imposition of a very high deformation; this is probably because in severe plastic deformation processes the high deformation imposed causes a substantial proportion of pearlitic cementite to be dissolved at room temperature (Hong et al. 1999, Vodopivec et al. 2007). In the case studied, this effect occurs because the as-received cold drawn steel has already been subjected to plastic deformation and, therefore, when applying ECAP, the steel is more severely deformed. The higher degree of deformation causes the majority of pearlite to disintegrate after only two passes (Shin & Park 2005, Jozef et al. 2010). The formation of the cementite particles observed results from the carbon dissolution of the pearlitic cementite by the severe plastic deformation process. After four passes, the pearlite is more severely distorted, and no grain boundaries or microstructural details are observed. At this point, the dissolution of carbon from cementite is more noticeable, and it is expected that excessive carbon exists in the ferrite phase (Park & Shin 2002).

The increase in tensile strength after the first pass was observed in the tensile testing; this could be due to the low structural refinement, especially in the ferrite. However, the increase in tensile strength is not comparable to values obtained when processing low-carbon steel in a normalized state with ECAP (Marulanda et al. 2013), in this case, it is possible to obtain even three times the initial value. An additional increase in tensile strength is produced after two passes; however, after four passes it decreases to a value even lower the initial. This decrease in tensile strength may be attributed to the fragmentation of cementite particles, which causes the pearlite to lose particles and; as a result, decrease the number of sliding obstacles. According to this, a decrease in tensile strength is mainly influenced by the dispersion of cementite particles more than by the subgrain formation. On the other hand, the severe deformation endured by the steel in the drawing and ECAP processes could have negative effects. The severe deformation process could produce local damages such as micro-cracks and multiple crack initiation sites (Pang et al. 2012) that considerably affect the tensile strength of the steel after four passes. In addition, the strain hardening of the metal by increased deformation makes the steel brittle. The material has less ability to deform plastically, and the fracture of the material is probable. These results agree with results reported by other authors (Raji & Oluwole 2013).

The behavior of the variation in the tensile strength presented especially for the fourth pass, when it drastically decreases, is not the expected for cold-deformed materials where the sliding obstacles increase and therefore the tensile strength. However, it should be taken into account that the steel studied has already been severely deformed before ECAP, the cementite particles have been fragmented, and this would cause the unusual behavior.

The increase in hardness is attributed to the decrease in grain size and, for the fourth pass, to the formation of cementite particles resulting from carbon dissolution which is more noticeable with higher deformation (Shin et al. 2002), as observed in figure 3. The distribution of these cementite particles, harder than ferrite, produces an increase in hardness, whereas the same particle dissolution produces less sliding obstacles and, therefore, the decreasing tensile strength.

Conclusion

The application of the equal channel angular pressing (ECAP) technique in cold drawn low-carbon steel, AISI 1016, is used to assess the possibility of improving its mechanical properties after the drawing process. The results show that it is possible to improve the tensile strength up to two ECAP passes. However, after four passes, when the deformation imposed on the steel is too high, and taking into account the initial drawing state, the tensile strength decreases even to a value lower the initial. This is because very high deformations produce, in this kind of steel, the dissolution of pearlitic cementite, the formation of cementite particles and excessive carbon in the ferrite. The distribution of these cementite particles, harder than ferrite, produces the increase in hardness; the same particle dissolution produces less sliding obstacles and, as a result, decreases tensile strength. Because the structural refinement obtained is low, the variation in the mechanical properties is mainly attributed to cementite particle dispersion. However, because the behavior of the tensile strength is not the usual for cold working steels, it should be further studied, based on other characterization techniques such as electron backscatter diffraction (EBSD) or transmission electron microscopy (TEM).

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Conflict of interest

This work has no conflict of interest.

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Microestructura y propiedades mecánicas del acero AISI 1016 calibrado procesado por ECAP

Resumen. Se utilizó el proceso de presión en canal angular de sección constante para deformar un acero de bajo carbono calibrado, con el fin de evaluar microestructura y propiedades mecánicas. A su vez, se identificó la posibilidad de mejorar propiedades más allá del proceso de calibrado. El material utilizado fue un acero de bajo carbono calibrado de composición 0.16 % C, 0.8 % Mn, 0.2 % Si, 0.02 % P, 0.012 % S y balance Fe. El proceso se realizó a temperatura ambiente con cuatro pasadas usando la ruta Bc, con una deformación equivalente de ~ 0.6 en cada pasada. Se estudió la evolución de la estructura antes y después de la deformación usando microscopía electrónica de barrido y difracción de rayos X y se evaluaron las propiedades mecánicas de microdureza y resistencia a la tensión. Se encontró un aumento leve de las propiedades mecánicas al aumentar el número de pasadas en el proceso. Los análisis mostraron cambios en la estructura ferrítica-perlítica original a través del refinamiento de los granos de ferrita y la deformación de la perlita.

Palabras clave: Presión en canal angular de sección constante; ECAP; acero de bajo carbono; refinamiento de estructura; resistencia a la tensión; microdureza.

Microestrutura e propriedades mecânicas do aço AISI 1016 calibrado processado por ECAP

Resumo. Utilizou-se o processo de pressão em canal angular de seção constante para deformar um aço de baixo carbono calibrado, com a finalidade de avaliar microestruturas e propriedades mecânicas. Assim, identificou-se a possibilidade de melhorar propriedades para além do processo de calibração. O material utilizado foi um aço de baixo carbono calibrado com a seguinte composição 0.16% C, 0.8% Mn, 0.2% Si, 0.02% P, 0.012% S e balance Fe. O processo realizou-se à temperatura ambiente com quatro passos utilizando a rota Bc, com uma deformação equivalente de ~ 0.6 em cada passo. Estudou-se a evolução da estrutura antes e depois da formação utilizando microscopia eletrônica de varrimento e difração de raios X e avaliaram-se as propriedades mecânicas de microdureza e resistência à tensão. Encontrou-se um aumento ligeiro das propriedades mecânicas ao aumentar o número de passos no processo. As análises mostraram alterações nas estruturas ferrítica-perlítica original a através do refinamento dos grãos de ferrita e a deformação da perlita.

Palavras-chave: Pressão em canal angular de seção constante; ECAP; aço de baixo carbono; refinamento de estrutura; resistência à tensão; microdureza.