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The effect of oxide particles on the strength and ductility of bulk iron with a bimodal grain size distribution

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ABSTRACT

The strength and ductility of bulk nanostructured and ultrafine-grained iron containing 0.39% oxygen by weight was determined by tensile tests. Samples were obtained by consolidation of milled iron powder at 500 °C. Heat treatments were designed to cover a wide range of grain sizes spanning from 100 to 2000 nm with different percentages of coarse and nanostructured grain areas, which was defined as a bimodal grain size distribution. Transmission electron microscopy was used to determine the diameter, volume fraction and location of oxides in the microstructure. The strength was analysed following two approaches. The first one was based on the strong effect of oxides and involved the use of a mixed particle-grain boundary strengthening model, and the second one was based on simple grain boundary strengthening. The mixed model underestimated the strength of nanostructured samples, whereas the simple grain boundary model worked better. However, for specimens with a bimodal grain size, the fitting of the mixed model was better. In this case, the more effective particle strengthening was related to the dispersion of oxides inside the large ferrite grains. In addition, the bimodal samples showed an acceptable combination of strength and ductility. Again, the ferrite grains containing oxides promoted strain hardening due to the increase in dislocation activity.

1. Introduction

The development of nanocrystalline (NC) and ultrafine-grained (UFG) materials with a good balance of strength and ductility is one of the main topics in current structural materials research. In the case of NC and UFG iron and steels, several processes based on the severe plastic deformation of ferrite grains have been implemented. Mechanical milling and, in particular, ball milling of iron have been extensively used to produce nanostructured powders that render NC and UFG bulk materials after a consolidation process [1–5]. The presence of an oxide layer in the powder before milling allows some oxygen content to be detected in bulk consolidated samples. Moreover, depending on the milling media used, some carbon content has also been reported [5,6]. Thus, in the analysis of the mechanical properties of consolidated ball-milled iron, the presence of finely dispersed particles in the nanostructure of specimens must be taken into account.

Generally, the yield strength of industrial iron and low-carbon steel is evaluated by a classical Hall-Petch relationship, $\sigma_y = \sigma_0 + k_y \cdot D^{-1/2}$ [7,8], and the increase in strength is related mainly to grain boundary strengthening. For Armco iron and low-carbon steels, the grain size dependence of the yield strength has been demonstrated by high values of the Hall-Petch coefficient k_y, between 550 and 600 MPa · $\mu m^{1/2}$ [9,10].

Recently, the role of particles in strengthening ball-milled iron has been investigated, and a new model has been proposed [11]. In this approach, the contribution of grain boundary strengthening is reduced when the grain size decreases below 2 μm; thus, a large percentage of the strength is attributed to the particles. We can define this model as a mixed particle-grain boundary strengthening model. Particles in the NC range would provide a large contribution to the overall strength of ball-milled iron when finely dispersed inside ferrite grains instead of being located at grain boundaries. At the same time, advances in thermomechanical processing have allowed for UFG irons to be obtained, in which the precipitation of carbides and oxides is suppressed [12]. In these UFG and industrial irons, grain boundary strengthening of ferrite has been demonstrated to depend strongly on the presence of solute carbon. It has been reported that the presence of only 60

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ppm of solute carbon changes the Hall-Petch coefficient k_y associated with pure iron from approximately 150 MPa $\mu m^{1/2}$ to 600 MPa $\mu m^{1/2}$ [13]. Applying these values for k_y, the Hall-Petch relationship appears to be obeyed in industrial irons for grain sizes below 1 μm and even at 0.2 μm [14–15]. Therefore, it can be concluded that this approach allows for NC and UFG iron to be strengthened based on simple grain boundary strengthening.

The increase in strength produced in NC and UFG irons after grain refinement is generally accompanied by a decrease in ductility. Under compression, in addition to high strength, moderate ductility has been observed even for average grain sizes close to the NC range [3,16–18]. It must be noted that, in these cases, the strain hardening has been observed to decline with the refinement of grain size, being virtually zero below 300 nm. At the same time, shear banding is clearly the main mode of deformation as the grain size reaches the UFG range [3,17,19].

Currently, different severe plastic deformation (SPD) techniques are used to obtain UFG iron from relatively pure materials such as Armco iron or interstitial-free steels (IF) [20]. In the case of UFG iron obtained by equal-channel angular extrusion (ECAE), the resulting microstructure consists of elongated grains with a cellular dislocation substructure [21,22]. This microstructure has the tendency to shear band, and the related strong shear localisation has been identified as the main cause of the low ductility observed in tensile tests. The same problem has been observed in UFG irons produced by high pressure torsion (HPT) and accumulative roll bonding [23–26], in which high strength is accompanied by low uniform deformation. In this case, the elongated and aligned structure results in a high mechanical anisotropy that leads to limited fracture toughness along certain orientations [23]. The use of heat treatments after the SPD deformation of UFG iron helps improve the total elongation of samples and even strain-hardening regions without a significant reduction in strength, but the total increase in uniform elongation is small [24,27,28]. On the other hand, the presence of carbon and cementite formation appears to be effective because UFG steels processed by caliber warm rolling have shown tensile elongation with Lüders band formation and subsequent strain hardening at carbon contents above 0.15 wt%, forming globular cementite particles [29].

One of the strategies developed in recent years for obtaining NC and UFG metals with high tensile plasticity is bimodal grain size distribution. Structures containing nanostructured and coarse-grained zones, in which the grains are on the micrometre scale, have been suggested to prevent failure by shear localisation [30]. Nanostructured matrix grains impart high strength, whereas coarse grains help the structure to induce strain-hardening mechanisms that stabilise tensile deformation. In this manner, a material retains much of its strength, recovering part of its ductility before SPD. Bimodal structures have been obtained by different techniques, such as the annealing of copper after ECAE processing [31]; the hot consolidation of milled and unmilled powder blends of Al-7.5 Mg [32], Fe-Cr alloys [33] and oxide dispersion strengthened (ODS) ferritic steels [34]; as well as the consolidation of milled iron in a spark plasma sintering (SPS) equipment [5,17]. It should be noted that in the bimodal structures obtained by blends of milled and unmilled powders, a banded structure is formed due to the ease with which coarse-grained particles are deformed [35]. On the other hand, the structure obtained after annealing ECAE samples or by the consolidation of only milled iron results in a more equiaxed structure. Finally, in studies in which the presence of dispersed oxides in iron and iron alloys were investigated [5,17,34], the strain hardening observed in tensile tests was related to the presence of micrometre-sized ferrite grains capable of storing dislocations, together with the presence of finely dispersed oxide particles.

The mechanical milling of pure iron and subsequent warm or hot consolidation has been one of the main techniques for obtaining bulk samples of NC and UFG iron and steels with a homogeneous grain size distribution [2–4,16,18]. However, porosity and a lack of bonding between powder particles in consolidated specimens formed by these techniques have been noted as the main causes for the brittle behaviour observed, particularly in tensile tests.

In this context, the aim of the present study was to produce pure iron with a bimodal grain size distribution from mechanically milled powder and investigate its mechanical response by hardness and tensile tests to find a good combination of high strength and ductility. At the same time, microstructural observations were carried out to investigate the size, volume fraction and location of oxide particles in the ferritic matrix. These microstructural features were used to predict the yield strength based on the two abovementioned strengthening models. These predicted yield strengths were compared with the experimental ones to evaluate the effect of oxide particles on the strength in the NC and UFG ranges.

2. Materials and Methods

Commercial pure iron powder with a particle size between 75 and 160 μm was mechanically milled to obtain a nanostructured iron powder. MM was carried out for 17 h in a planetary ball mill with stainless steels vials and hardened steel 100Cr6 balls with a diameter of 10 mm under an Ar gas atmosphere. The ball to powder ratio was 27:1. The average particle size of the milled powder was approximately 80 μm, and the microhardness of the powder was evaluated by Vickers hardness tests with a load of 0.1 N. The powder had a hardness of 8.1 ± 0.7 GPa and a mean crystallite grain size of 26 ± 10 nm, as determined by X-ray diffraction using a modified Williamson-Hall method [36].

The milled powder was consolidated using a two-step process. The first process consisted in the cold compaction of the powder at 1300 MPa. The powder was not removed from the mould after cold compaction. The second step was hot static compaction at a fixed pressure of 850 MPa held for 1 h at 500 °C. The specimens measured 1 mm in height and 10 mm in diameter. Additionally, some specimens were heat treated under an Ar atmosphere between 650 °C and 775 °C for 0.5 h to obtain different grain sizes. The final chemical composition (mass%) was 0.03 C, 0.39 O, 0.94 Cr, 0.53 Ni and 0.017 P (Fe balance). Tension test specimens were cut from the consolidated samples using wire electrical discharge machining. The samples measured 0.8 mm wide, 0.8–1 mm thick and 2 mm in gage length. Tensile tests were conducted in a Deben tensile machine at an initial strain rate of $1.7 \cdot 10^{-3}\ s^{-1}$. The elongation was calculated from the gauge length and the displacement measured by the tensile machine. The hardness of the consolidated samples was evaluated by Vickers hardness tests on the upper and lower flat surfaces of the cylinders using a load of 1.96 N. At least 15 measurements were performed for each specimen. The cylinders were ground and finally polished using a diamond suspension with grain size of 6 μm.

The density was determined by the Archimedes method with water as the liquid medium. Microstructural analysis and determination of ferritic grain size were performed by scanning electron microscopy (SEM) using a JEOL JSM6400 microscope equipped with an energy dispersive X-ray spectroscopy (EDS) system (Oxford Inca X-sight) and by transmission electron microscopy (TEM) using a Philips CM30 microscope operating at 300 kV. For the preparation of TEM samples, the consolidated pieces were ground on 600-grit silicon carbide to a thickness of 250 μm and were then cut into discs measuring 3 mm in diameter. Finally, the