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Acceleration of spheroidisation in a medium carbon steel processed by equal channel angular pressing

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Abstract

A medium carbon alloy steel was processed using equal channel angular pressing (ECAP) with a view to accelerating the softening process during subsequent annealing. The steel was pressed at 600 °C for up to four passes. The original well-aligned lamellar cementite became bent and discontinuous with increasing number of passes. Complete spheroidisation was achieved after annealing at 720 °C for only 30 minutes, resulting in the same hardness as that obtained after 72 hours without ECAP. This can potentially lead to significant cost reduction and environmental benefit for the industry.

Key words: equal channel angular pressing, medium carbon alloy steel, spheroidisation, heat treatment, severe plastic deformation

1. Introduction

Among severe plastic deformation (SPD) based processes, equal channel angular pressing (ECAP) is one of the most effective, widely used to refine microstructures to produce bulk ultrafine grained metals and alloys [1]. On the other hand, ECAP has been successfully used to consolidate particles into bulk ultrafine and nanostructured materials [2, 3]. Although most experiments are conducted on non-ferrous alloys of single phases, SPD, in particular ECAP, has been employed to refine the ferrite matrix grains in steels (mostly low carbon plain steels), leading often to improved mechanical properties. For example, grain sizes of 0.2–0.3 μm were obtained in low carbon steels after multiple passes of ECAP [4, 5]. An ultrafine grained (UFG) dual phase steel processed by ECAP followed by intercritical annealing displayed superior properties of high strength, large elongation and rapid strain hardening [6]. Tensile strengths over 1000 MPa were achieved in a low carbon steel processed by ECAP at room temperature for up to 10 passes [7].

Strengthening, however, is not always the intended outcome in the processing of steels. Some heat treatments, such as annealing and spheroidisation, are designed to soften steels to improve their formability. This is particularly important during wire drawing of high strength rods for the production of, for instance, fasteners. The term 'spheroidisation' is defined as the transformation of cementite (Fe_3C) from a lamellar morphology in the eutectoid structure to a spheroidal one, driven by a decrease in the interfacial area between the cementite and ferrite phases and an overall reduction in the free energy of the system. The kinetics of cementite spheroidisation in many commercial steels is often very slow, requiring typically days of time and thus increasing the cost of production. It is highly desirable that this process be accelerated. Fortunately, SPD is also demonstrated to be capable of affecting the morphology of the cementite phase in steels. Hono et al. reported that heavy cold drawing could completely dissolve the cementite phase in a hypereutectoid steel [8]. In particular, Shin and coworkers showed that the temperature and time for the eutectoid cementite in a low carbon steel to decompose and spheroidise may be significantly reduced by prior ECAP [9, 10].

In the present investigation, ECAP was used to process a medium carbon alloy steel used for fastener applications with a view to significantly reducing the 72 hours needed to soften it to be suitable for the subsequent drawing process. Medium carbon steels are

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much stronger and thus require ECAP to be conducted at higher temperatures. The steel after ECAP was subjected to annealing at the usual spheroidisation temperature for up to 60 minutes and compared with that treated without ECAP.

2. Experimental materials and procedures

The experimental material was a 4037 medium carbon alloy steel with a nominal composition (wt.%) of Fe-0.37C-0.8Mn-0.27Mo-0.22Si-0.12Cr-0.018P. The ECAP specimens were cut in the form of bars of 60 mm in length. ECAP was conducted using a 90° die with sharp corners and channel cross sections of $9 \times 9\,\mathrm{mm}^2$. The specimen was lubricated in graphite and placed in the entrance channel of the die which was heated and held at 600 ℃ using a heating device controlled to ± 1 ℃. Up to 4 passes were carried out at a pressing speed of $50\,\mathrm{mm\,min}^{-1}$. The specimens were rotated 180 degrees between passes following the C route.

The middle section of the specimens after processing by ECAP was cut into pieces and annealed at 720 ℃ for up to 60 minutes for spheroidisation. The Vickers microhardness (HV) was measured by applying a load of 100 g for 25 seconds and taking the average of 10 individual readings. The surface of the specimens was prepared following standard metallography procedures and etched in a solution of 3 % nital to reveal the microstructure using optical microscopy (OM, Olympus BH2-UMA) and scanning electron microscopy (SEM, FEI Quanta 200 FEG ESEM). For higher resolution, thin films of the steel were made by focused ion beam (FIB) for transmission electron microscopy (TEM, Tecnai F20).

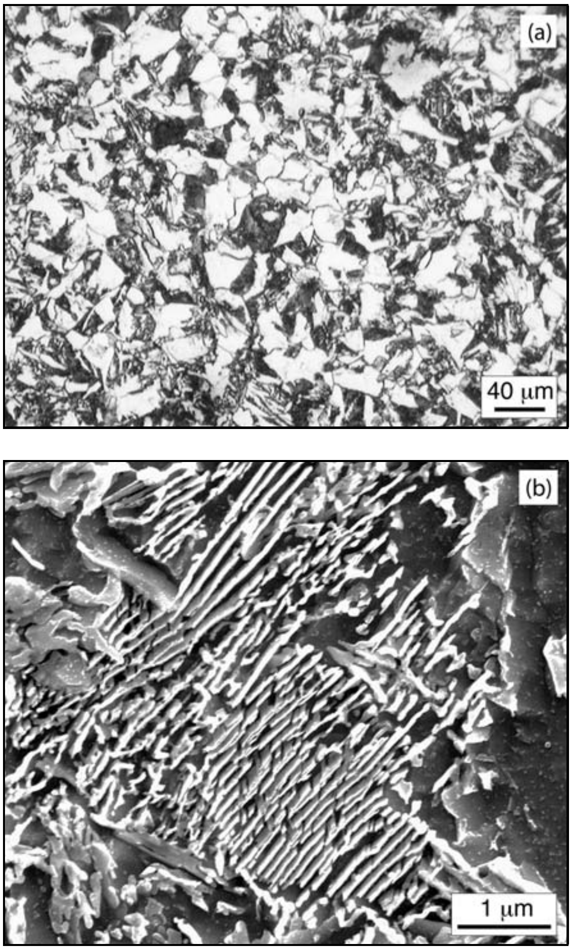

Fig. 1. Microstructures of the medium carbon alloy steel in the as-received condition, showing (a) a mixture of equiaxed ferrite (the light phase) grains and pearlite (the dark phase) colonies (OM) and (b) continuous cementite lamellae in the eutectoid structure (SEM).

3. Results and discussion

Figure 1 shows the microstructures of the as-received steel which had been normalised. The typical mixture of ferrite (the light phase) grains and pearlite (the darker phase) colonies can be identified in Fig. 1a, and they appeared equiaxed with sizes ranging from 10 to 40 μm. Upon close examination by SEM, the eutectoid structure was revealed in Fig. 1b with well aligned continuous lamellae of cementite (the white phase). When such a material was subjected to annealing at 720 ℃ (just under the eutectoid temperature) for one hour, little change was observed in the optical microstructure shown in Fig. 2. Indeed, the present industry practice was to anneal at 720 ℃ for 72 hours to cause a complete spheroidisation of the eutectoid cementite, as shown in Fig. 3, with cementite particles of the order of 1 μm in size distributed in the ferrite matrix. This confirms the sluggish

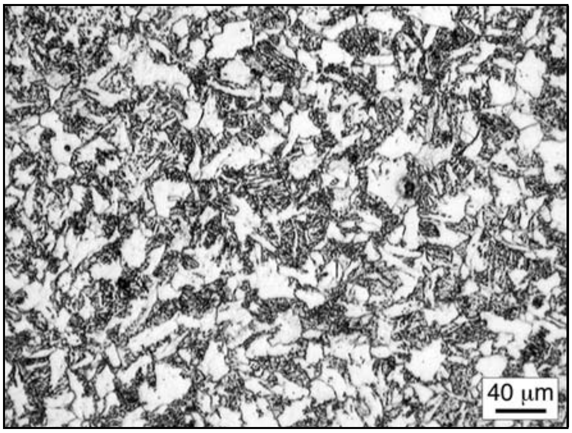

Fig. 2. Optical microstructure after annealing at 720 ℃ for 1 hour, displaying little difference from that of the as-received material.

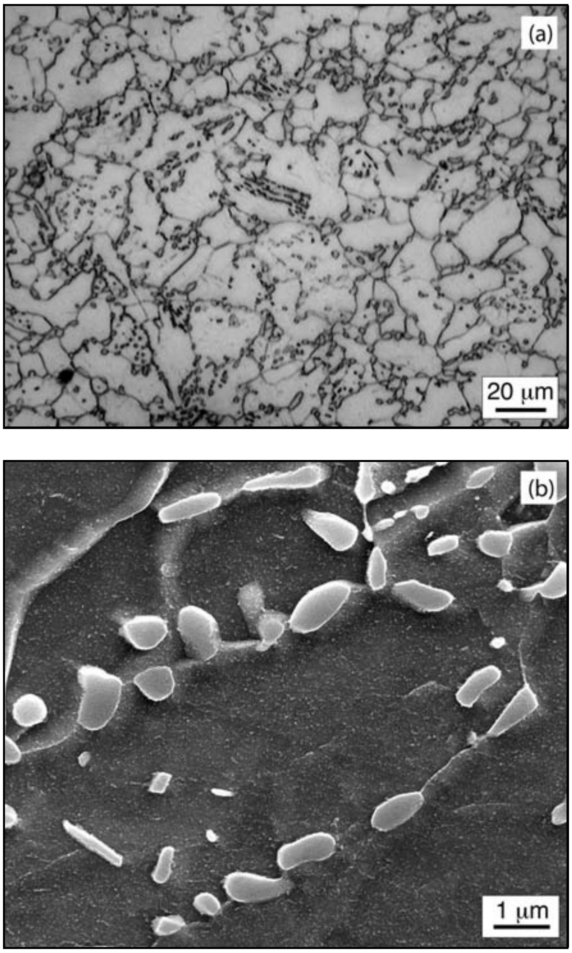

Fig. 3. Microstructures after the standard industrial annealing at 720 °C for 72 hours, showing (a) a completely spheroidised structure (OM) and (b) individual cementite particles of the order of 1 µm in size (SEM).

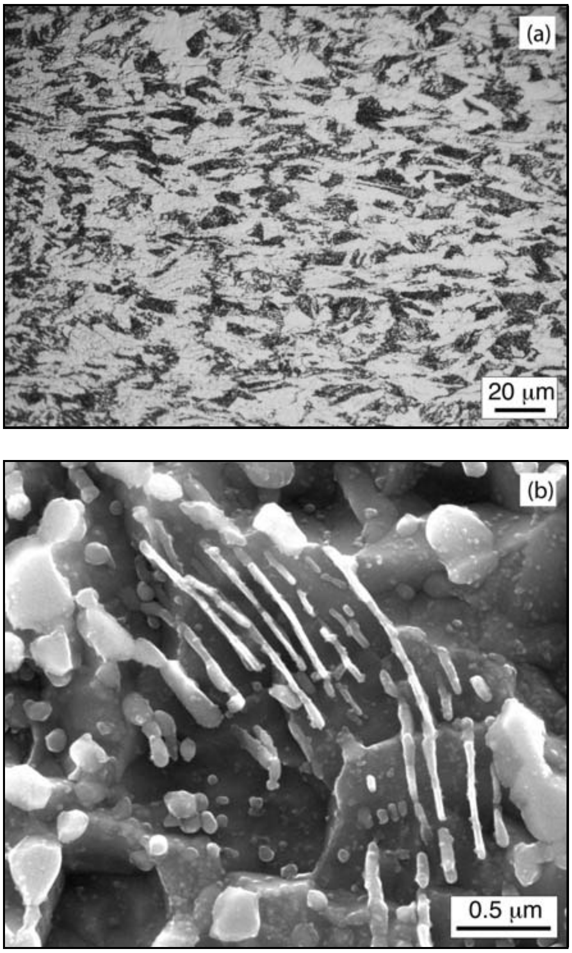

Fig. 4. Microstructures after ECAP at 600 °C for 1 pass showing (a) severely deformed ferrite grains and pearlite colonies (OM) and (b) fragmented and bent cementite plates (SEM).

transformation even at this high annealing temperature.

Both the grain structure and the morphology of cementite were changed significantly by ECAP, as shown in Figs. 4 and 5. After ECAP for one pass at 600 °C, both the ferrite and pearlite grains were sheared (Fig. 4a), with both becoming elongated ones of approximately 1–5 µm in width and 10–15 µm in length, and the ultrafine grain structure became obvious after 4 passes (Fig. 5a), as also observed by others (e.g. [4, 5]). The pearlite colonies appeared to be severely deformed and fragmented into smaller ones with irregular shapes (Fig. 4a). Examination by SEM showed that the well aligned cementite lamellae in the as-received material had become discontinuous in many places and bent (Fig. 4b). Further ECAP to 4 passes, almost all cementite lamellae were broken into short ones or spheroids, as shown in Fig. 5b, although their distribution still revealed the locations of prior plates. This is consistent with the observation in a low carbon steel by TEM [11] that necking developed in many places along the cementite plates, leading to their fragmentation and curled shape after ECAP. The deformation and break-up of cementite might be dependent on the orientation of the cementite plates and the direction of shear during ECAP. Indeed, complicated shear patterns were found to form in a eutectic Al-Cu alloy by shearing after ECAP for multiple passes [12]. It was apparent that ECAP had gradually transformed the original lamellar cementite into a spheroidal one even before annealing at the near eutectoid temperature. This was achieved through severe plastic deformation rather than the usual diffusion-induced dissolution of the cementite phase since the ECAP temperature was much lower and time shorter. Indeed, the steel directly annealed at 720 °C for one hour (i.e. without prior ECAP deformation) showed little difference in the cementite morphology (Fig. 2). The cementite short rods or spheroids were mostly very fine with sizes ranging from approximately 50 to

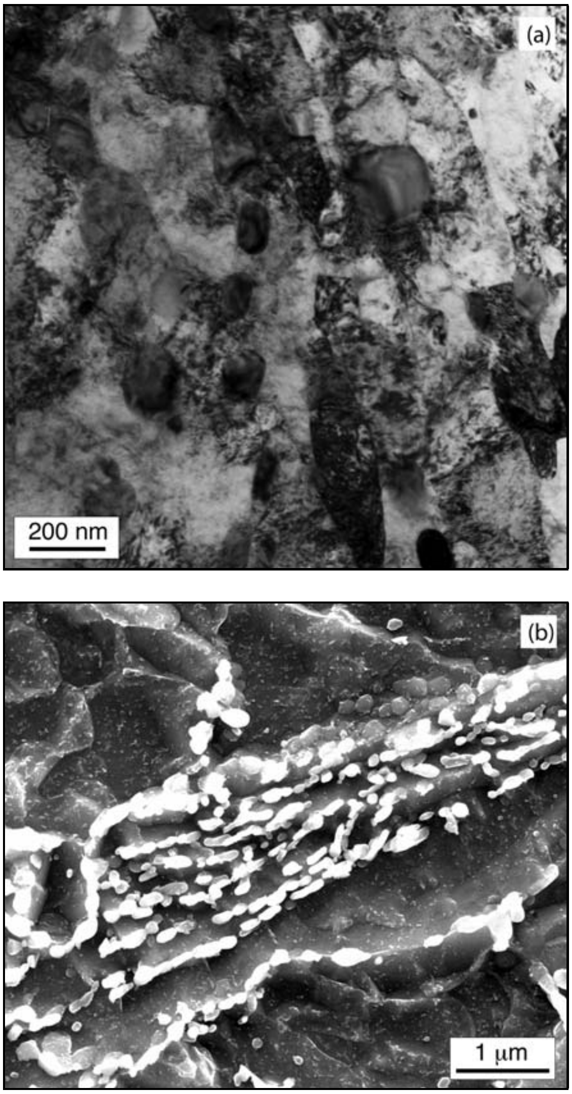

Fig. 5. Microstructures after ECAP at 600 ℃ for 4 passes showing (a) an ultrafine grain structure (TEM) and (b) mostly fragmented, discontinuous cementite (SEM).

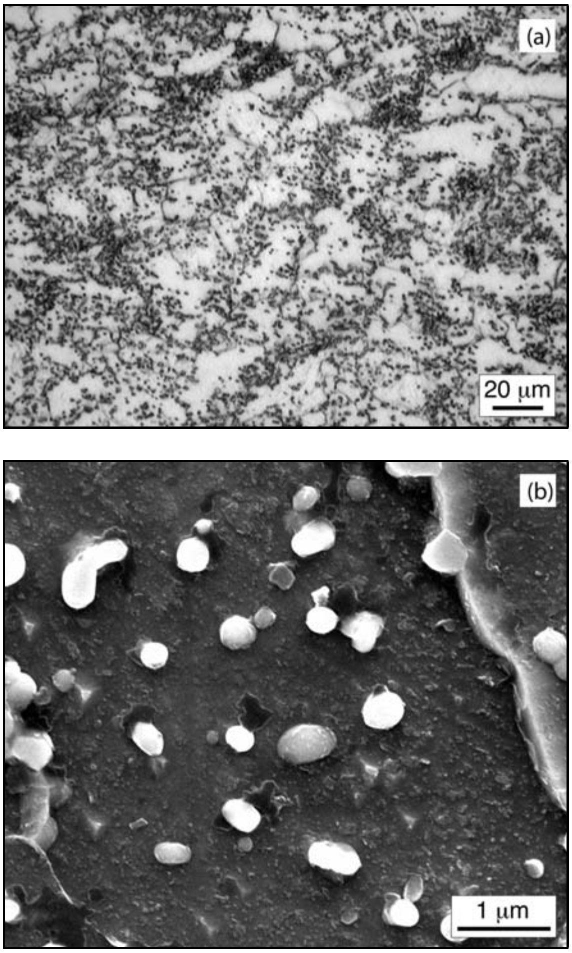

Fig. 6. Microstructures after annealing for 30 minutes at 720 ℃ of the steel processed by ECAP at 600 ℃ for 4 passes, showing (a) complete spheroidisation (OM) and (b) individual spherical cementite particles (SEM).

200 nm, similar to the thickness of the eutectoid cementite plates in the as-received material. However, compared to the observations in [11], some spheroidisation had already occurred, as evident in Fig. 5b, although the shape of the original plates can still be recognised. This is because the ECAP temperature here was significantly higher (by about 200 ℃).

The microstructures of the material which was processed by ECAP for 4 passes and subsequently annealed at 720 ℃ for 30 minutes are shown in Fig. 6. A fully spheroidised microstructure was obvious in Fig. 6a, and it appeared that the spheroidisation occurred mainly within the former pearlite colonies although there was indication of migration of cementite into the ferrite territory, probably as a result of shear deformation and/or cementite dissolution and precipitation. Figure 6b is a high magnification SEM image of the spheroidal cementite. It was apparent that it had been coarsened by annealing to approximately 200 to 700 nm in size, compared to that after ECAP for 4 passes (Fig. 5b). It has been shown [13] in a heavily cold drawn steel that C dissolution in the ferrite phase was significantly enhanced by severe plastic deformation and C atoms were preferentially distributed around dislocation lines. This enhanced solubility was attributed to much increased interfacial energy between cementite and ferrite as severe plastic deformation had increased the total interfacial area and the roughness of the interface [14].

The Vickers hardness (HV) values of the various materials are presented in Table 1. The as-received steel was only moderately strengthened by ECAP since the processing temperature was high, resulting in limited grain refinement, although it was harder with increasing number of passes. Upon annealing, the most significant decrease in hardness occurred within the

Table 1. The Vickers microhardness values of the steel in different conditions

Material	HV (MPa)
As-received	2170
ECAP at 600 ℃ for 1 pass	2350
ECAP at 600 ℃ for 4 passes	2620
ECAP at 600 ℃ for 4 passes followed by annealing at 720 ℃ for 15 min	1720
ECAP at 600 ℃ for 4 passes followed by annealing at 720 ℃ for 30 min	1600
ECAP at 600 ℃ for 4 passes followed by annealing at 720 ℃ for 60 min	1500
As-received + annealing at 720 ℃ for 72 hours	1630

very short first 15 minutes at 720 ℃. Further softening, not as drastic though, was observed after 30 and 60 minutes, respectively. Importantly, the materials after such short annealing times were as soft as that annealed for 72 hours without ECAP. That is to say, ECAP had substantially reduced the annealing time required. This outcome can be related to the much enhanced coarsening kinetics as the nano cementite phase became of the order of ten times larger after annealing (Fig. 6b), compared to that after ECAP (Fig. 5). Any grain refinement due to ECAP was also expected to have disappeared after annealing.

4. Conclusion

1. A medium carbon alloy steel with ~ 0.4 wt.% C was processed by ECAP at 600 ℃ for up to 4 passes. Both the ferrite grains and pearlite colonies were severely shear deformed by ECAP. After one pass, the originally continuous eutectoid cementite lamellae were transformed into a curly or spheroidal morphology. Further ECAP to four passes resulted in spheroidal cementite with sizes of the order of 50–200 nm. At the same time, the hardness was increased moderately with increasing number of ECAP passes.

2. Following subsequent annealing at 720 ℃ for as short as 15 minutes, the spheroidisation of cementite was completed with much coarsened sizes of the order of 200–700 nm. Correspondingly, the steel was softened to the same level as that achieved in the industry standard practice of annealing for 72 hours at the same temperature. That is to say, the softening by annealing had been significantly accelerated as a result of ECAP.

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