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Dissolution of cementite in carbon steels by ball drop deformation and laser heating

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Abstract

The dissolution behavior of cementite by heavy deformation in eutectoid steels with pearlitic and spheroidite structures was studied. To examine the thermal effect on the cementite dissolution, pulsed laser heating was also carried out. By ball drop deformation, the cementite dissolved and the matrix became nano-grained. After pulsed laser heating, re-austenitized region transformed to fresh martensite during quenching. The boundary between the re-austenitized region and matrix exhibited similar microstructure with that observed in specimens subjected to ball drop deformation. It was suggested that the dissolution of cementite by heavy deformation at high strain rate were probably due to thermal effect, that is, re-austenitization.

Keywords: Cementite; Pearlite; Spheroidite; Deformation; Dissolution; Structure

1. Introduction

It has been reported that a substantial proportion of the cementite in pearlite dissolves by heavy deformation at room temperature, such as cold-rolling [1], wire-drawing [2], ball milling [3-5] and high-pressure torsion [6]. Cementite dissolution is also observed on the surface of rail tracks [7,8]. Two distinct mechanisms have been proposed for the dissolution of cementite during heavy deformation. One is the thermal effect [7] and another is the deformation effect [8]. In the former case, adiabatic heating during deformation transforms the matrix to austenite and cementite dissolves into austenite. This idea arose from the finding of fresh martensite and/or retained austenite at the region where cementite is dissolved. In the latter case, cementite dissolves into ferrite at low temperature. The present study aims to investigate the individual effect of deformation and thermal treatment on the cementite dissolution. The dissolution behavior of both lamellar and spheroidal cementite is studied by ball drop deformation and pulsed laser heating.

2. Experimental procedures

Various eutectoid steels with composition ranging from 0.76 to 0.80 mass% C with pearlite and spheroidite structures were used. Ball drop deformation (a weight with a ball attached on its bottom was dropped onto the specimens) was performed with 5 kg weight and a ball of 6 mm in diameter. Pulsed laser heating was performed using CO_2 laser in air under two conditions: (1) average power of 50 W (pulse-on duration 10%, frequency 50 Hz) and scanning rate of 6000 mm/min and (2) average power 150 W (pulse-on duration 50%, frequency 1000 Hz) and scanning rate 500 mm/min. The laser beam diameter was 0.15 mm. After deformation or laser heating, specimens were characterized by optical microscope (OM), SEM, TEM and Vickers microhardness tester.

3. Results

3.1. Dissolution of cementite by ball drop deformation

Fig. 1 shows the OM micrograph of the pearlitic steel subjected to ball drop deformation. Near the surface, a bright contrast layer (often called white etching layer from its appearance under OM after etching) is seen. Fig. 2(a) shows the SEM image of the boundary between deformed pearlite structure (lower part) and white etching layer (upper part) which appears as a dark contrast featureless structure (Fig. 2(b)). The boundary between them is sharp and clear. In the featureless structure, the original cementite lamellae are hardly observed. Fig. 2(c) is the enlarged

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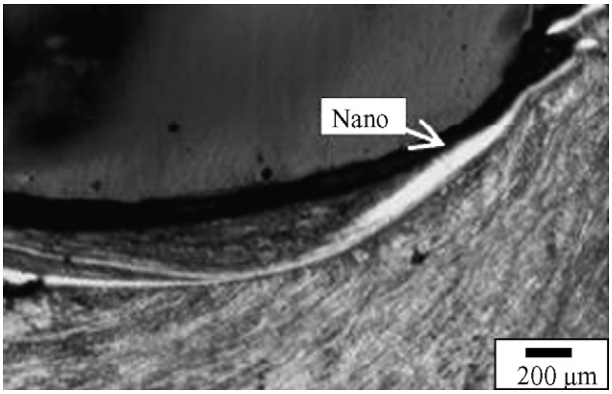

Fig. 1. OM micrograph of the Fe–0.76%C pearlitic steel after ball drop deformation (dropped 25 times from 1 m height with 5 kg weight).

micrograph of the boundary marked (c) in Fig. 2(a). In the lower part of the picture heavily deformed pearlite is clearly seen, while in the upper part a faded contrast finely spaced lamellar structure is seen. Fig. 3 shows the dark field (DF) image and the selected area diffraction (SAD) rings taken from the featureless structure region. The DF image shows that the grain size is around 100 nm. The hardness of the featureless structure was about HV 11 GPa which is substantially higher than that of the deformed pearlite structure region (about HV 7 GPa). From the structure and hardness, it is clear that nanostructuring occurred by ball drop deformation. In the SAD rings, cementite phase is hardly detected. It should be noted that the SAD rings correspond to ferrite and austenite phases. This indicates that the temperature of this region increased above Ac_1 so that the austenite phase formed.

Fig. 4 shows the dissolution of cementite particles by ball drop deformation. The upper part in Fig. 4(a) shows a featureless structure and lower part shows a deformed structure region. The featureless structure region (enlarged in Fig. 4(b)) is similar to that shown in Fig. 2. From TEM observation, it is confirmed that the featureless structure region is nanocrystalline and cementite particles are completely dissolved. In the deformed ferrite structure region even near the nanocrystalline region deformed or irregular shaped cementite particles were hardly observed. The structure with cementite particles surrounded by nanocrystalline structure was sometimes observed as shown in Fig. 4(c). The middle layer in Fig. 4(c) shows bright contrast, and the upper and bottom layers show conventional spheroidite structure. The enlarged image of the bright contrast layer (Fig. 4(d)) shows that spherical cementite particles surrounded by similar contrast ferrite region. This structure is considered to be an early stage of the formation of featureless structure shown in Fig. 4(a) and (b). It is interesting to note that cementite particles are not deformed although the surrounding ferrite is heavily deformed to become nanocrystalline.

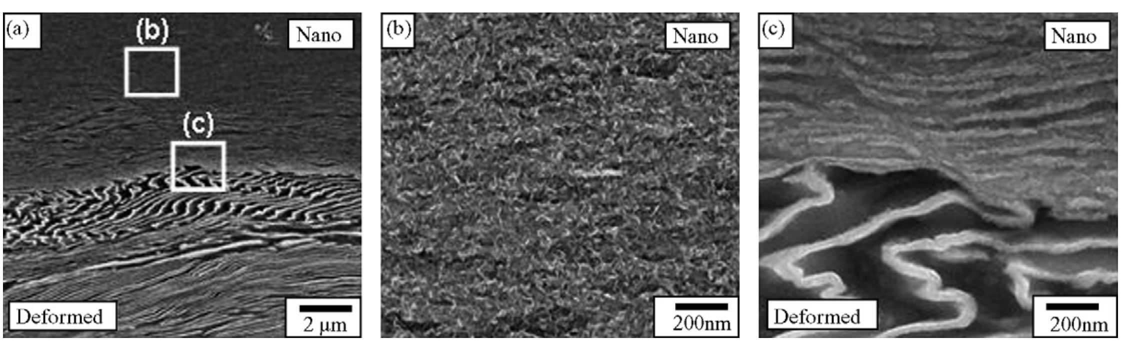

Fig. 2. SEM micrographs showing dissolution of cementite in pearlite (Fe–0.80%C) by ball drop deformation (dropped eight times from 1 m with 5 kg weight). (a) Low magnification, (b) the high magnification of nanocrystalline region and (c) high magnification of the boundary between the nanocrystalline and deformed structure regions.

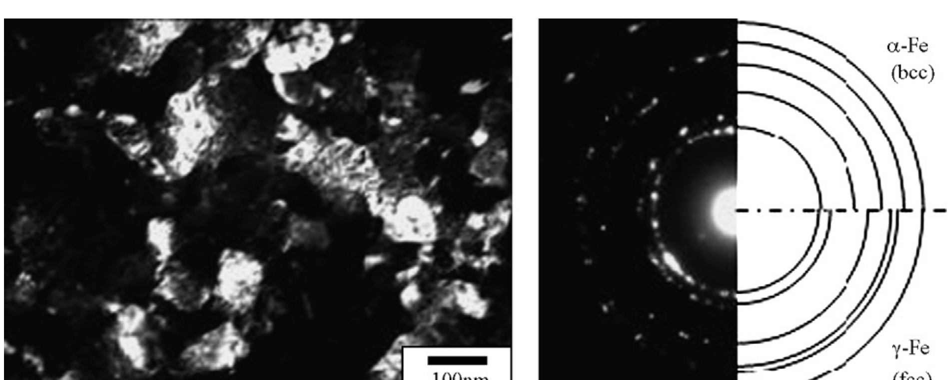

Fig. 3. TEM DF image and SAD pattern taken from the nanocrystalline region in the Fe–0.80%C pearlitic steel after ball drop deformation (dropped eight times from 1 m with 5 kg weight).

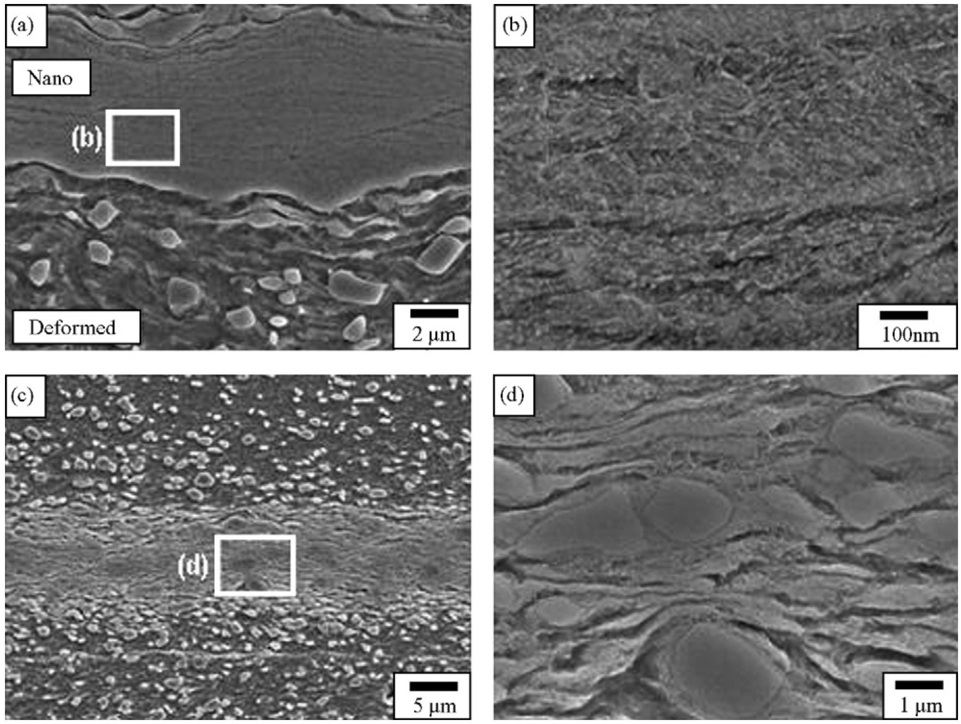

Fig. 4. SEM micrographs showing (a and b) cementite dissolution and (c and d) cementite particles surrounded by ferrite (Fe–0.80%C) by ball drop deformation (dropped 30 times from 1 m with 5 kg weight). (b and d) The enlarged micrographs of (a and c).

3.2. *Dissolution of cementite by laser heating*

Fig. 5(a) and (b) are the OM micrographs of the pearlitic and spheroidite steels heated by pulsed laser heating. In the OM micrographs, triangle shaped (cone in three dimension) white etching region are seen. These appearances are similar to that formed by ball drop deformation (Fig. 1). Fig. 5(c) shows the SEM micrograph of white etching region which appears as dark contrast under SEM. From the microstructure and hardness (HV 8.0–8.8 GPa) of the laser heated layer, it is clear that the structure of white etching layer is fresh martensite. Fig. 6 shows the SEM micrographs of the boundary between fresh martensite formed by laser heating and pearlite matrix. The boundary is sharp and clear. The enlarged SEM micrograph (Fig. 6(b)) indicates that in the re-austenitized region the cementite lamellae become thinner and split into small pieces. This indicates that during rapid heating and cooling the time in austenite state is long enough for cementite lamellae to start to dissolve but too short to reach complete dissolution. Such situation is more pronounced in the case of spheroidite steel. Fig. 7 shows the boundary between spheroidite matrix and re-austenitized region in the laser heated spheroidite steel. The volume fraction of re-austenitized region increases toward surface since the maximum temperature increased toward surface. The enlarged micrograph of the boundary (Fig. 7(b)) shows the cementite particles surrounded by re-austenitized region (now fresh martensite). This indicates that austenite phase starts to form at the cementite/ferrite interface and grows at the expense of cementite. As approaching the surface, the volume fraction of re-austenitized region increases and the size of cementite particles decreases.

4. Discussion

To investigate the individual effect of deformation and thermal shocks on the dissolution of cementite, ball drop deformation and pulsed laser heating were carried out. During heavy deformation especially at high strain rate, it has been reported [9] that the deformed local layers experience rapid heating and quenching cycle. It is expected that pulsed laser heating pro-

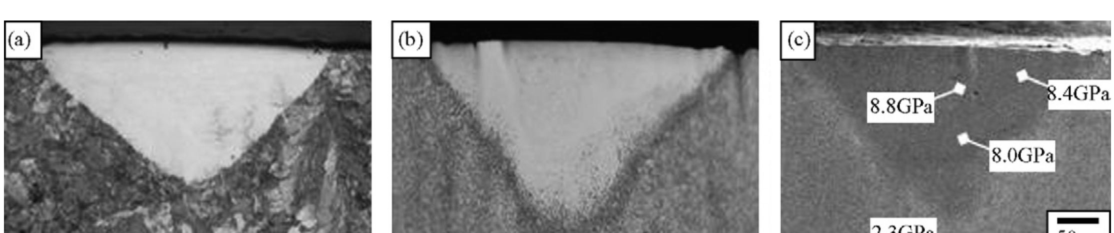

Fig. 5. OM micrographs of Fe–0.76%C steel with (a) pearlite and (b) spheroidite structure heated by pulsed laser (50 W). (c) SEM micrograph of heated Fe–0.76%C spheroidite steel showing the results of Vickers microhardness test.

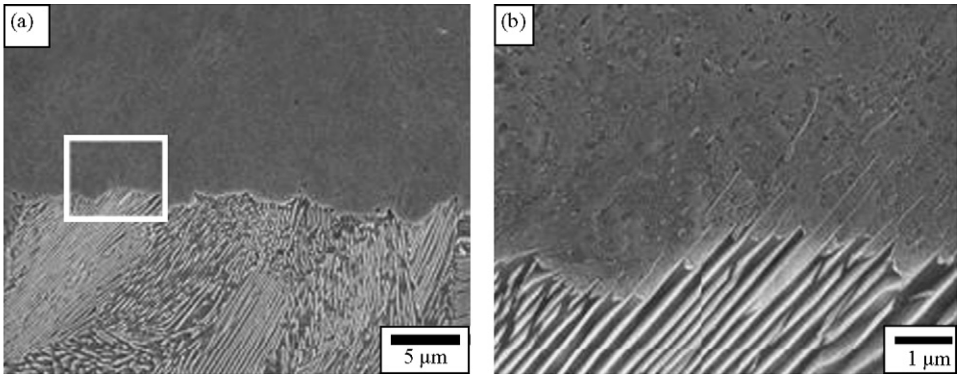

Fig. 6. SEM micrographs of the boundary between the fresh martensite produced by laser heating (150 W) and pearlite matrix in the Fe–0.76%C pearlitic steel after annealing at 373 K for 3.6 ks. (a) Low magnification and (b) high magnification of the boundary.

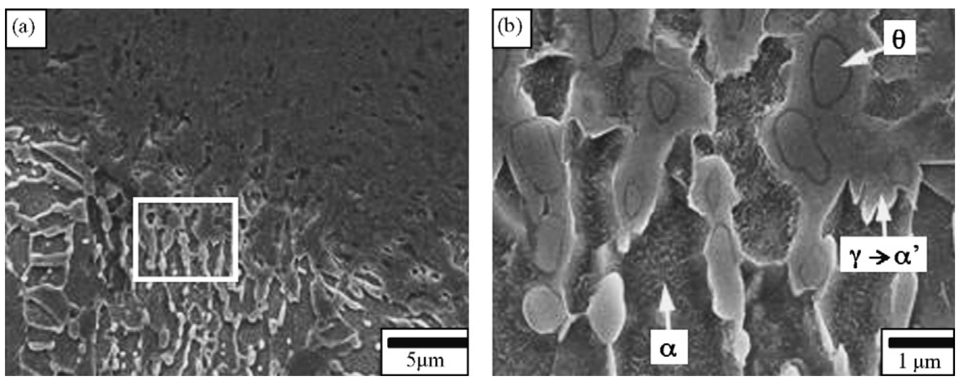

Fig. 7. SEM micrographs of (a) boundary between spheroidite matrix and re-austenitized region in the laser (150 W) heated Fe–0.76%C spheroidite steel after annealing at 373 K for 3.6 ks. (b) The enlarged image near the boundary marked in (a).

duces similar thermal cycles. From the experiments of laser heating, it became clear that rapid heating and cooling cycle can produce some microstructures also observed in heavy deformation. Of course there are clear differences between the laser heated samples and those subjected to heavy deformation. The white etching regions observed under OM are fresh martensite in the former and nanocrystalline structure in the latter although cementite is completely dissolved in both cases. This suggest that local temperature rise in heavily deformed specimens especially at high strain rate is large enough to reach above Ac_1 (depending on heating rate and higher than 1000 K in Fe–C system) and can produce fresh martensite during subsequent rapid cooling. Large hydrostatic pressure might assist the dissolution of cementite by decreasing Ac_1 temperature. The thermal effect on the dissolution of cementite in samples deformed by wire drawing, ball milling and high-pressure torsion is still an important subject to be examined.

5. Summary

The dissolution behaviors of cementite by deformation and rapid heating was studied using ball drop deformation and pulsed laser heating. In the experiments of ball drop deformation, ferrite grains become nanometer sized and a layer appears as featureless structure under SEM observation when cementite is completely dissolved. Sharp boundaries were observed between the featureless and deformed structure regions. The laser heating experiments revealed that rapid heating and quenching cycle produced some microstructures also obtained by ball drop deformation. The boundaries between fresh martensite and matrix were sharp. The microstructures with partially dissolved cementite observed at the boundaries were similar to those observed in specimens subjected to ball drop deformation.

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