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Changes in the geometry of grain boundaries during recovery/continuous recrystallization in α-Fe

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Samples of α-Fe (Armco) have been deformed by 50% and subsequently annealed at 400 °C which is considerably below the conventional recrystallization temperature. The geometry of the grain boundaries has been studied through measurements of the grain boundary traces revealed on longitudinal cross-sections of the specimens. Two etching conditions were employed, one of which revealed networks containing only grain boundaries (designated GB in the further text) whilst the other resulted in delineating the 2-dimensional regions characterized by the presence of sulfur (designated S-GB). Changes in the geometry of these two types of grain boundaries as a result of plastic deformation and annealing are discussed in terms of *in-situ* high angle grain boundary formation and annihilation.

1. Introduction

Plastic deformation changes the geometry, microstructure and, thus, the properties of materials. In the case of polycrystals deformed at a low temperature, the major changes are an increase in the density of lattice defects and the rearrangement of grain boundaries. Upon annealing, the deformed microstructure tends to release stored energy and to equilibrate its microstructure. The microstructural changes during annealing have been recently studied in α-Fe using transmission electron, TEM, and light microscopy [1, 2]. A quantitative analysis has been carried out of the changes in the geometry of grain boundaries in material annealed at 400 °C, which is considerably below the conventional recrystallization temperature. Some characteristic stages in the variation of the grain boundary geometry as a function of annealing time have been observed in these studies. Initially the boundaries restore their near-equilibrium geometry by decreasing their elongation. This is accompanied by an increase in the total density of grain boundaries. This rearrangement has been suggested to be partly due to a rapid subgrain growth which is known to produce larger subgrains with relatively higher angle subboundaries of misorientation angle exceeding 10° [3, 4]. Also, a clear disproportion between the amount of changes in the geometry of the high angle grain boundaries and the degree of coarsening of the subgrains has been observed. This observed difference suggested that other mechanisms for the high angle grain boundaries rearrangement must operate in the studied material. The present paper reports results of further studies aimed at an explanation of the nature of the grain boundary rearrangement observed in α-Fe.

2. Experimental procedure

The studies have been performed on α-Fe (Armco) with an initial grain size of approximately 30 μm. The chemical composition of the material is given in reference [1]. Specimens in the form of rods were compressed at room temperature to give a total reduction in thickness of 50%. The deformed material was isothermally annealed at 400 °C for 0.5 h.

The geometry of the grain boundaries has been studied through measurements of grain boundary traces that were revealed on longitudinal cross-sections of the specimens. Two etching conditions were employed, one of which revealed networks containing only grain boundaries (designated GB in the further text) whilst the other resulted in delineating the 2-dimensional regions characterized by the presence of sulfur (designated S-GB).

Measurements of boundary geometry were carried out using an image analyser with specially developed software. A number of geometrical parameters of the individual sections revealed on cross-sections of the microstructures have been measured. These parameters are listed in Table I together with their

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TABLE I Geometrical parameters used to define properties of the boundaries

Parameter	dimension	interpretation
A	μm^2	section area of a nucleus/grain
d_{eq}	μm	circle equivalent diameter
p	μm	section perimeter
d_{max}	μm	section maximum chord
d_{max}/d_{eq}		shape factor which increases with the degree of the section elongation
p/d_{eq}		shape factor which increases with the degree of the section perimeter non-convexiness
$(S_v)_a$	μm^{-1}	surface area of a-type boundaries in unit volume

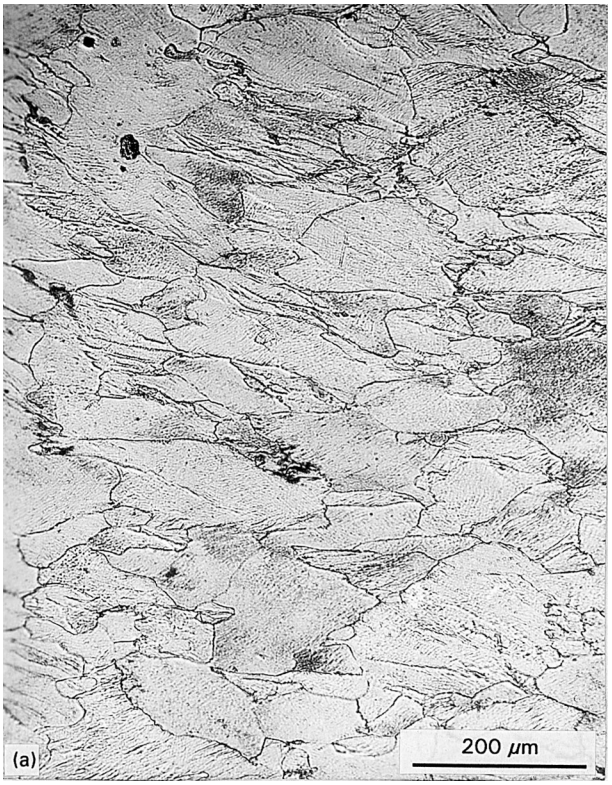
interpretation. The experimental distribution functions were described in terms of the mean values, E(), standard deviation, SD(), and CV() – coefficient of variations (CV(X) = SD(X)/E(X)).

It should be noted that the feature section area, A, and equivalent diameter, d_2, which define the size of the grain/cell sections, under some assumptions can be used to infer the volume distribution [5]. The parameters, d_{max} and p, can be used to describe the shape of the sections and in turn the shape of grain/cells via the shape factors d_{max}/d_2 and p/d_2. The first ratio describes the elongation of grain sections whilst the second is a sensitive measure of the boundary curvature of a grain section.

3. Results

Changes in the geometry of grain boundaries as a result of the anneal at 400 °C are illustrated in Fig. 1(a–d). In Fig. 1(a and b) the micrographs show characteristic behaviour of the grain boundary (GB) network in the material after 50% compression and after annealing at 400 °C for 0.5 h, respectively. Fig. 1(c and d) show sulfur-rich boundaries (S-GB) in the same specimens. Results of the measurements of geometrical features of the microstructures shown in Fig. 1(a–d) are given in Table II. It can be noted that the GB, and S-GB microstructures for the deformed material show considerable similarity. In particular, this is demonstrated by similar values of the shape factor, d_{max}/d_{eq}, which indicates elongation of the microstructures. Some minor changes in the surface density of the two types of surfaces, described by $(S_v)_{GB}$ and $(S_v)_{S\text{-}GB}$, can be explained by the limited sensitivity of the method.

After a 30 min annealing the GB grain boundaries have lost their preferential orientation along the direction perpendicular to the axis of compression as reflected by the value of $E(d_{max}/d_2)$. On the other hand the sulfur rich regions (S-GB) have undergone a significantly less intensive rearrangement and have retained a considerable anisotropy in their spatial orientation. In addition, the annealing is accompanied by a drop in the value of the shape factor of GB, $(d_{max}/d_{eq})_{GB}$ from 1.6 to 1.3, the latter of which is typical of annealed α-Fe. At the same time the shape factor for the S-GB microstructure, $(d_{max}/d_{eq})_{S\text{-}GB}$, remains almost unchanged. There are also measurable variations in the value of the other shape factor, p/d_{eq}, which is lower, and similar, for both the annealed microstructures.



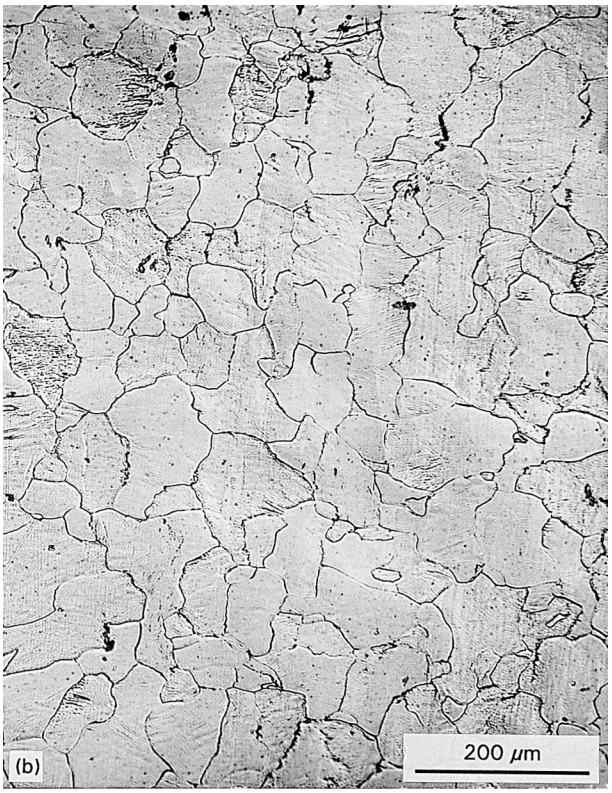


Figure 1 Geometry of grain boundary network, (a) and (b), and sulfur rich grain boundaries, (c) and (d), observed on the sections parallel to the axis of straining. Micrographs (a) and (c) show typical high angle grain boundary arrays in specimens plastically deformed by a 50% compression, (b) and (d) after a subsequent anneal at 400 °C for 0.5 h.

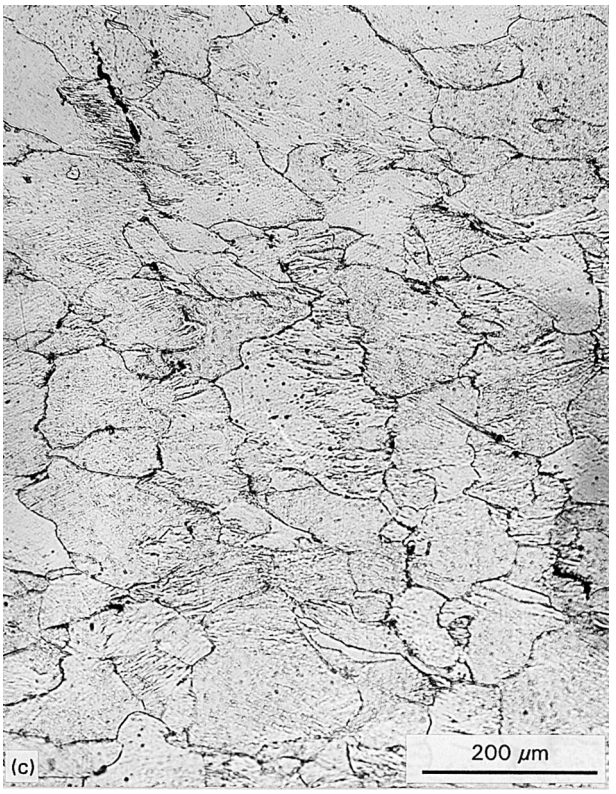


Figure 1 (Continued)

Taking into account the nature of the sulfur distribution in the material and the observed evolution of the microstructures, it can be concluded that before the annealing both S-GB and GB indicate the geometry of the pre-existing high angle grain boundaries in the deformed material. On the other hand in the annealed microstructures GB indicates positions of the newly formed grain boundaries whilst S-GB the positions of the pre-existing grain boundaries.

TABLE II Mean values, E(), standard deviations, SD(), and coefficients of variations, CV(), of the parameters characterizing geometrical features of the GB and S-GB microstructures in α-Fe

After a 50% compression		parameter	after annealing at 400 °C	
GB	S-GB		GB	S-GB
4.16	3.93	$E(p/d_{eq})$	3.89	3.84
1.58	1.54	$E(d_{max}/d_{eq})$	1.33	1.49
60.3	70.5	$E(d_{eq})$ (μm)	50.3	55.3
0.65	0.68	$CV(d_{eq})$	0.67	0.66
4093.3	5699.5	$E(A)$ (μm^2)	2878.7	3431.6
1.21	1.21	$CV(A)$	1.25	1.24

4. Discussion

It has been noted in previous papers that there are some characteristic stages in the variation of the grain boundary geometry as a function of annealing time at 400 °C [1, 2]. Initially, for annealing times in the range of 30 min, the boundaries restore their near-equilibrium geometry by decreasing their elongation. This is accompanied by an increase in the total density of the grain boundaries (i.e., a reduced grain size). This rearrangement has been suggested to be partly due to rapid subgrain growth which is known to produce larger sub-grains with relatively higher angle sub-boundaries with misorientation angles exceeding 10° [3, 4].

The two types of etching condition employed in the present study resulted in revealing boundary networks of significantly different geometries. These conditions delineate grain boundaries of different properties and origin. The grain boundaries shown in Fig. 1b are typical of a network formed as a result of annealing the plastically deformed material. On the other hand the sulfur-rich regions form a system of boundaries which depicts the position of the grain boundaries after the plastic deformation imposed and prior to the annealing. The geometrical features of these two grain boundary networks suggest that the general grain boundaries revealed in the annealed material have been formed independently of the pre-existing grain boundary network. If one takes into account the incompatibility between the two grain boundary networks and the subgrain microstructure revealed in the TEM studies of the material, a conclusion can be drawn that the observed process of the grain boundary rearrangement can be explained in terms of an *in-situ* (migration-free) disappearance of some grain boundary segments and the *in-situ* formation of new ones.

The observation of grain boundary rearrangements suggest that the role of grain boundaries in the process of recovery in cold-worked polycrystals can be explained in terms of *in-situ* high angle grain boundary formation and annihilation. These transformations of the grain boundaries take place as a result of their interactions with the sub-boundaries and do not require migration of high angle grain boundaries over distances comparable with the grain size of the material.

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