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RECRYSTALLIZATION OF FERRITE IN SPHEROIDITE OF Fe-0.67% C STEEL

REKRYSTALIZACJA FERRYTU W SFEROIDYCIE STALI Fe-0.67% C

In this paper the study of ferrite recrystallization in Fe-0.67% C carbon steel with microstructure of the coarse spheroidite was performed. The purpose of the investigations was to determine the recrystallization mechanism and the sites of recrystallization nucleation.

It has been shown indirectly that in the investigated steel after deformation, ε , greater than $\varepsilon = 9\%$, recrystallization of the matrix (ferrite) proceeds discontinuously and recrystallization nucleates in the deformation zones around large particles.

Keywords: ferrite recrystallization, texture, coarse spheroidite

W pracy przeprowadzono badania rekrystalizacji ferrytu w węglowej stali Fe-0.67% C o mikrostrukturze grubego sferoidytu, których celem było określenie mechanizmu i miejsc zarodkowania rekrystalizacji po odkształceniu plastycznym na zimno. Wykazano pośrednio, że w badanej stali po odkształceniu ε większym od $\varepsilon = 9\%$ zachodzi nieciągła rekrystalizacja osnowy (ferryt), a zarodki rekrystalizacji powstają w strefach deformacji wokół dużych cząstek.

1. Introduction

Recrystallization in metals and metal alloys can proceed discontinuously (creation and motion of recrystallization fronts) or continuously (in situ). Recrystallization in situ is often observed during annealing after very high degrees of deformation or small deformations, in materials with a high content of the second phase particles [1].

Recrystallization mechanism in two-phase alloys is in principle not different from the recrystallization mechanism in single-phase alloys. However, the presence of the second phase particles affects significantly the process kinetics. The influence of the second phase particles on the recrystallization process may be formulated as follows [1, 2]:

1. the stored energy and driving force for recrystallization may be increased,
2. large particles can be the sites of easy nucleation for recrystallization,
3. particles, particularly arranged close together, can cause a distinct effect of "immobilization" of grain boundaries (the so called Zener drag effect).

The effects described in 1) and 2) accelerate recrystallization, while the occurrence of the case 3) inhibits the

process course. It comes out that the recrystallization course, especially its kinetics, grain size and texture after recrystallization depends on which of those effects is dominant. Because the particle size as well as volume fraction of dispersed phase in alloy depends on a chemical composition and heat treatment, the second phase particles can be used to control the microstructure and texture in industrial alloys [1-4].

There are few data about the effect of plastic deformation degree on the recrystallized grain size in two-phase alloys [2, 5]. Recrystallization of ferrite in steels with dispersed carbide phase has been the subject of the paper [6]. In cold-rolled (50%) hypo-eutectoid carbon steels with spheroidal particles of Fe_3C , the microstructure of recrystallized (700°C) ferrite (matrix) depends on the content and dispersion degree of cementite. For lower carbon contents ($\%C < 0.3$) cementite particles are located inside the recrystallized grains of coarse-grained ferrite, while in steels with higher carbon content (0.6% C) in the microstructure of recrystallized steel cementite particles are at grain (subgrain) boundaries of fine-grained ferrite. The differences found in the microstructure result from the way of the ferrite recrystallization course. For the lower contents of carbon

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a discontinuous recrystallization has been found, whereas for the higher ones, the in situ recrystallization has been indicated.

The metallographic research of recrystallization of a cold plastic deformed non-alloy Fe – 0.67 %C carbon steel with the microstructure of a coarse spheroidite showed that when the deformation degree increases, ferrite grain size decreases [7]. If the deformation is lower than 20%, there are cementite particles inside the recrystallized grains of relatively coarse-grained ferrite. In case of larger deformations (>20%) the cementite particles occur almost exclusively at the grain boundaries of recrystallized fine-grained ferrite. There were no significant differences found in the morphology of matrix microstructure (ferrite) of recrystallized samples depending on cementite dispersion in initial steel microstructure (before deformation).

In [7, 8] it was shown that for a given deformation level the grain size of recrystallized matrix (ferrite) depends on the cementite dispersion degree before deformation. In case of deformations up to 15%, the lower dispersion degree of cementite corresponds to the smaller grains of recrystallized matrix. In case of deformations exceeding 15% it is in opposite. With increasing deformation decreases the size of the particles that are potential sites of recrystallization nucleation (the deformation zones). For small deformations, the number of the particles, around which there can be nuclei of recrystallization, is smaller for steel of higher cementite dispersion degree; consequently the grain of recrystallized matrix is larger. It indicates (indirectly) that nucleation occurs during recrystallization in deformation zones around the largest particles. The conclusion is confirmed by the morphology of microstructure of recrystallized steel after small deformation (~9%), in which large particles are at the grain boundaries of the matrix.

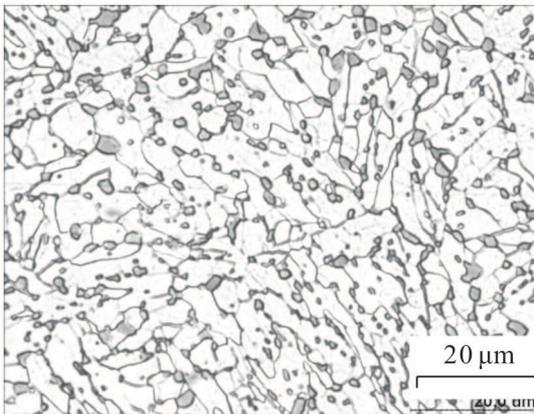


Fig. 1. Microstructure of Fe-0.67%C steel after preliminary heat treatment

In [7] the study of recrystallization concerns the microstructure of the state after recrystallization annealing in the scale of light microscope magnification (metallographic tests). To describe and analyze the microstructure there have been used the methods of qualitative and quantitative metallography (stereological measurement techniques). In this work the light microscope studies (LM), the X-ray tests (texture) and transmission electron-microscope studies (TEM) have been carried out, to determine the recrystallization mechanism and the sites of recrystallization nucleation in the coarse-grained spheroidite of Fe-0.67%C steel.

2. Material and heat treatment

The research has been carried out on a non-alloyed C70 steel, which the chemical composition is presented in Table 1.

TABLE 1
Chemical composition of Fe-0.67%C steel

| Element content [wt.%] | C | Mn | Si | P | S | Cr | Ni | Cu | Fe |
|------------------------|------|------|------|-------|-------|------|------|------|----------|
| | 0.67 | 0.27 | 0.20 | 0.011 | 0.016 | 0.07 | 0.04 | 0.07 | Ba-lance |

In order to receive an initial homogeneous microstructure of steel with coarse spheroidite, the following preliminary heat treatment has been performed: (i) homogenizing annealing (1200°C/5 h) in argon atmosphere; (ii) hardening (900°C/2 h) in the 5% water solution of NaOH; (iii) tempering in vacuum (700°C/150 h). It resulted in the formation of the microstructure with cementite particles at grain boundaries of in situ recrystallized fine-grained ferrite, whose grains are in general elongated and their configuration indicates a relation with microstructure of the former martensite (Fig. 1) [8-10].

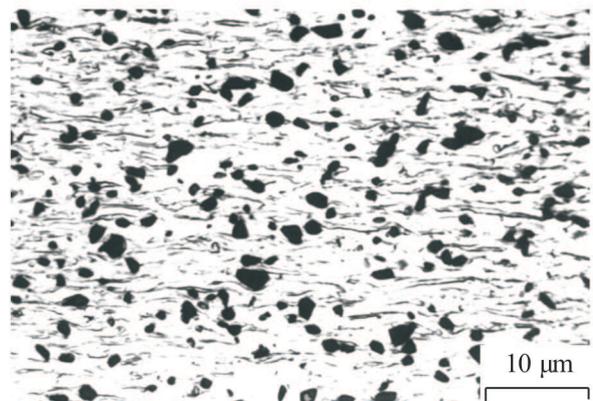


Fig. 2. Microstructure of Fe-0.67%C steel after 77% deformation

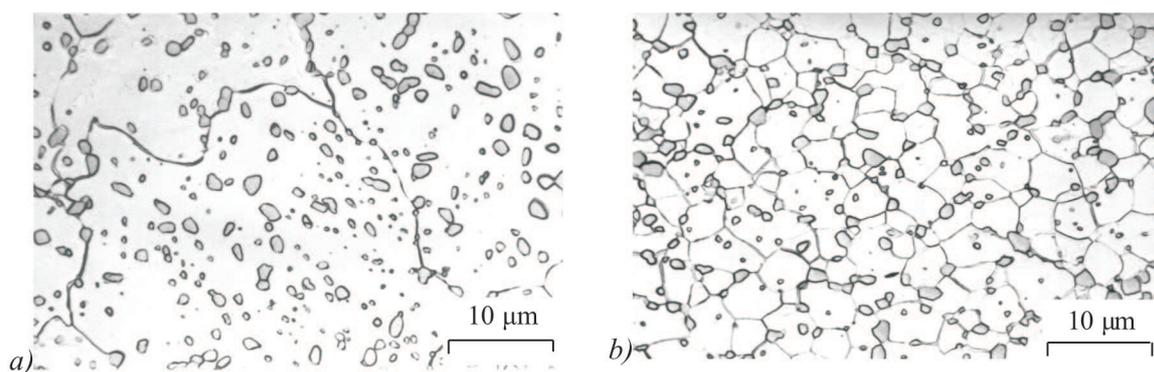


Fig. 3. Microstructures of Fe-0.67%C steel recrystallized (700°C, 5h), after deformation: a) 9%, b) 77%

The sections of the preliminary heat-treated bar have been cut to obtain the samples of dimensions 18×6×10 mm, which were cold-deformed by compression parallelly to the sample z axis (parallel to the 10 mm side) on a hydraulic press for 9% and 77% deformation (Fig. 2). The deformed samples were annealed in vacuum at 700°C and the time with duration from 3 minutes to 210 hours (Fig. 3a, b).

3. Light microscope

Description of the microstructure of the recrystallized matrix (ferrite) during subsequent annealing at 700°C was the aim of the LM studies. The specimens for LM investigations were double etched [7-10]: (i) in nital (for revealing the ferrite grain boundaries); (ii)

in boiling alkaline sodium picrate (for colouring the Fe₃C particles). Typical microstructures are shown in Fig. 3. After 9% deformation most of the cementite particles are inside grains of discontinuous recrystallized coarse-grained ferrite matrix (Fig. 3a). For 77% deformation the cementite particles are mainly at grain boundaries of fine-grained ferrite matrix (Fig. 3b). Qualitatively, the microstructure is similar to that for the preliminary heat treatment in Fig. 1. For quantitative description of the matrix grain size, the mean chord length, $\langle l \rangle$ was used. The relationship between the matrix grain size and annealing time for $\varepsilon = 9\%$ and $\varepsilon = 77\%$ deformation is shown in Fig. 4.

The matrix grain size after $\varepsilon = 9\%$ deformation is much larger than after $\varepsilon = 77\%$ deformation; the matrix grain size changes with annealing time are very small.

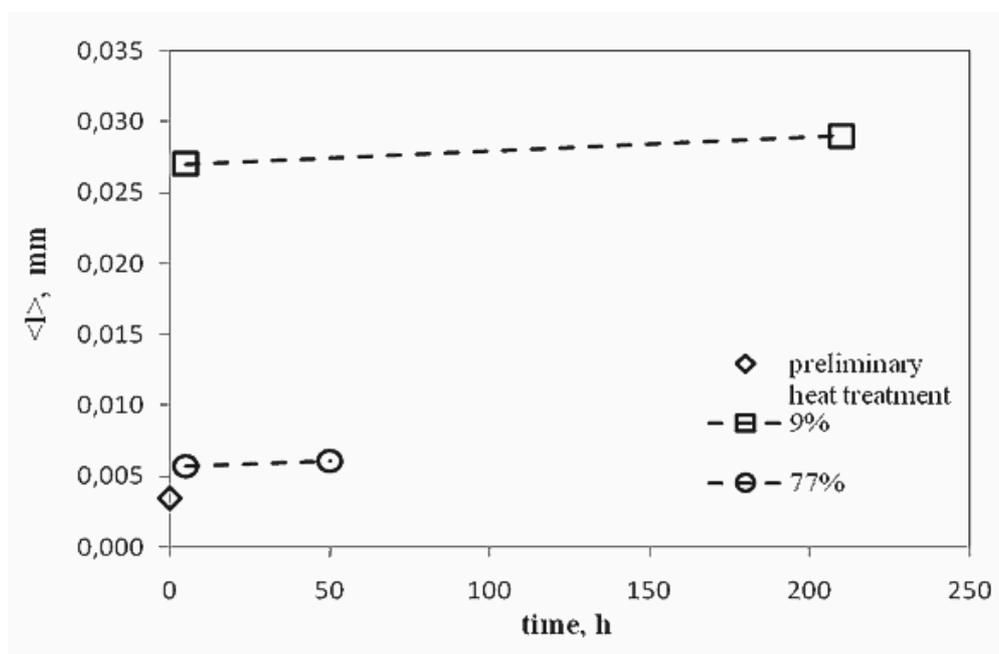


Fig. 4. Relationships between the mean grain chord length (of recrystallized matrix (700°C) after deformation 9 and 77%) on annealing time

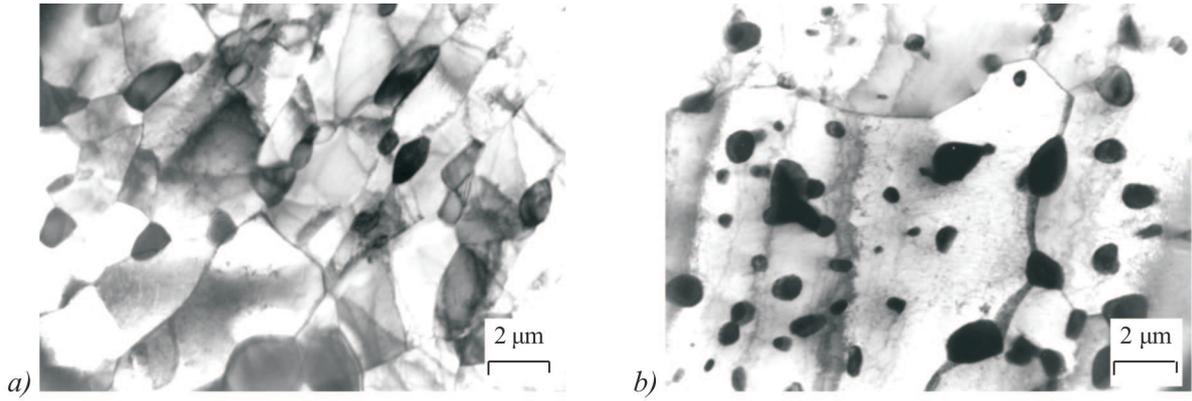


Fig. 5. Microstructures of Fe-0.67%C steel: a) after preliminary heat treatment; b) recrystallized (700°C, 5h) after 9% deformation

4. Transmission electron microscope

Microstructure of matrix (ferrite) and its changes during annealing (in the conditions of recrystallization) have been examined by using a technique of thin foils

in transmission electron microscope (TEM) JEM 200CX of the JEOL company.

In case of steel microstructure, after preliminary heat treatment the most of cementite particles is on the grains and subgrains boundaries of fine-grained matrix (ferrite) (Fig. 5a).

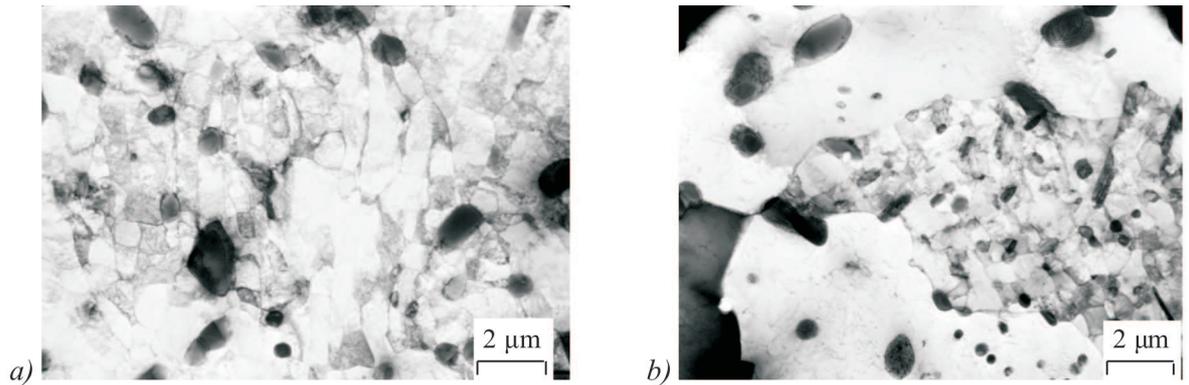


Fig. 6. Microstructures of Fe-0.67%C steel after 9% deformation and annealing at 700°C during: a) 3 minutes and b) 10 minutes

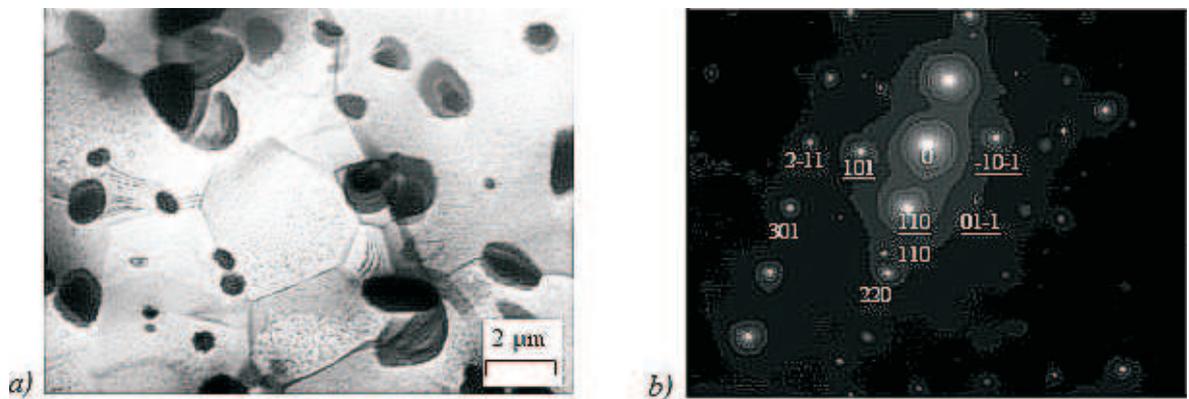


Fig. 7. Microstructures of Fe-0.67%C steel after 77% deformation and annealing during 3 minutes at 700°C (a), diffraction from grain boundary (two different axis $[111]\alpha_1$ i $[113]\alpha_2$) (b)

In the material 9% deformed and annealed at 700°C for 3 minutes no recrystallization process has been observed (Fig. 6a). After 10 minutes of annealing, next to the non-recrystallized areas, there are observed recrystallized areas (Fig. 6b). In those areas there are large cementite particles both inside the grains and at the grain boundaries. In the areas where small cementite particles existed, recrystallization process has not been observed. It indicates the easier nucleation of recrystallization on large particles, Fig. 5b. However, after 5 hours of annealing the smaller cementite particles are mostly detected inside the grains and subgrains of the recrystallized matrix (ferrite) (Fig. 5b) but not at the grain boundaries like in the case of the preliminary heat-treated steel (Fig. 5a), while large cementite particles have been observed at the grain boundaries of matrix.

Morphology of the microstructure of the sample 77% deformed and annealed at 700°C for 3 minutes (Fig. 7a) indicates discontinuous recrystallization. There are grains of a distinctly polygonal shape observed in the microstructure. From the analysis of diffraction it results (Fig. 7b) that grain boundaries in ferrite are of a high angle type.

5. X-ray

Research of texture concerned the preliminary heat treated, deformed and recrystallized state. The plane on which the texture has been examined was parallel to the deformation direction.

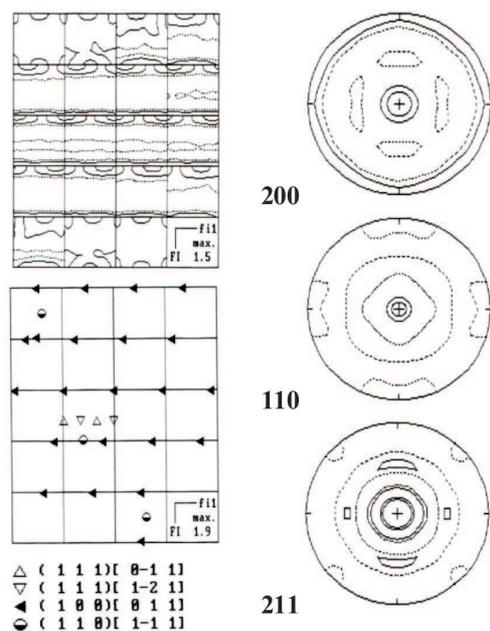


Fig. 8. Orientation Distribution Function with ideal orientations and pole figures of materials after pre-heating

Examined samples:

- after preliminary heat-treatment,
- after cold plastic deformation ($\epsilon = 9$, $\epsilon = 77\%$),
- annealed at 700°C during:
 - 210 hours after 9% deformation,
 - 50 hours after 77% deformation.

The results of X-ray examinations presented as an orientation distribution function (ODF) and pole figures showed differences in the texture of the particular material states (Fig. 8÷12).

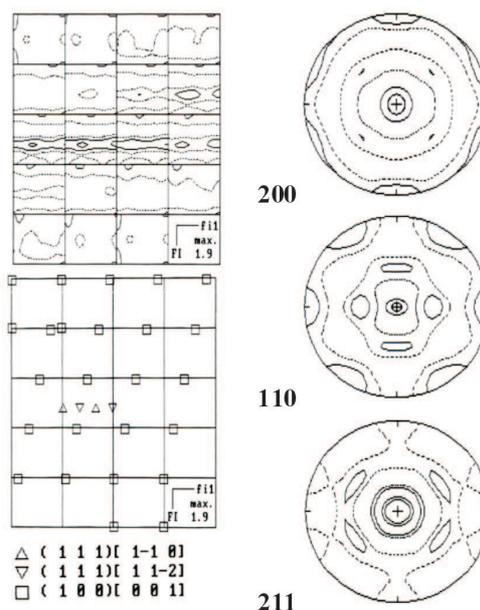


Fig. 9. Orientation Distribution Function with ideal orientations and pole figures of materials after 9% deformation

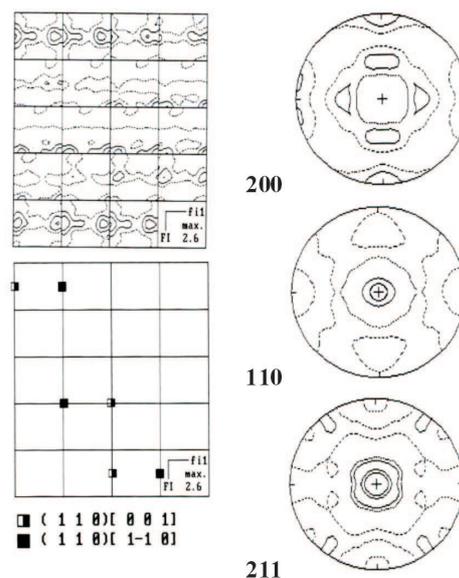


Fig. 10. Orientation Distribution Function with ideal orientations and pole figures of recrystallized materials after 9% deformation (700°C/210 h.)

Because changes of ferrite grain size with annealing time are very small (Fig. 4) the ferrite texture after long time of annealing can be considered as recrystallization texture.

In the texture of the preliminary heat-treated material occurs fibrous texture $\{111\} \langle uvw \rangle$ with main components $\{111\} \langle 110 \rangle$ and $\{111\} \langle 112 \rangle$ (fiber γ) (Fig. 8).

In the texture of the 9% deformed material are the same orientations as in the texture of the preliminary heat-treated material (Fig. 9).

In the texture of the 77% deformed material occurs the orientations: $\{112\} \langle 110 \rangle$, $\{110\} \langle 112 \rangle$ and the cubic orientation $\{100\} \langle 001 \rangle$, moreover one can distinguish a weaker orientation $\{013\} \langle 031 \rangle$ (Fig. 11).

After small deformation, the texture of ferrite is clearer, i.e. essentially, it can be described with two ideal orientations, while after large deformation there are many orientations in the texture (Fig. 9 and 11).

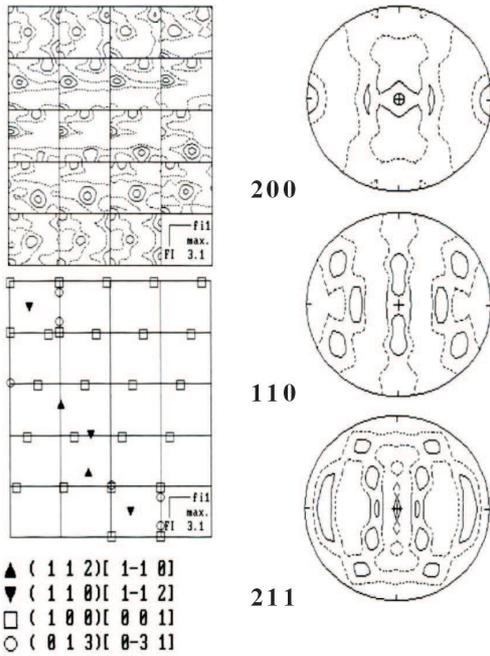


Fig. 11. Orientation Distribution Function with ideal orientations and pole figures of materials after 77% deformation

The texture after recrystallization annealing of material after 9% deformation is described by fiber $\{110\} \langle uvw \rangle$, in which the strongest orientations are $\{110\} \langle 001 \rangle$ (Goss texture) and $\{110\} \langle 110 \rangle$ (Fig. 10). Whereas in the recrystallization texture of the material after 77% deformation the dominant are the orientations which in a sample in deformed state were weak or were located in the scatter region of main components of the deformation texture ($\{013\} \langle 1031 \rangle$, $\{013\} \langle 100 \rangle$, $\{013\} \langle 131 \rangle$, and $\{013\} \langle 031 \rangle$). The ori-

entations which were strong in the deformed sample disappear (Fig. 12).

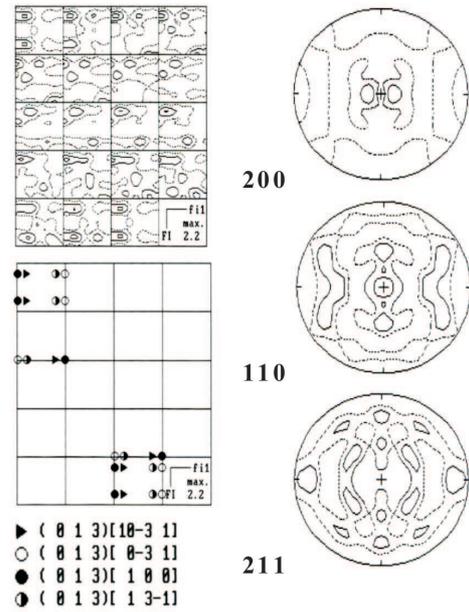


Fig. 12. Orientation Distribution Function with ideal orientations and pole figures of recrystallized materials after 77% deformation (700°C/ 50 h.)

6. Discussion

The X-ray examinations showed that the texture of recrystallized ferrite is different from the deformation texture either after small ($\epsilon = 9\%$) or high ($\epsilon = 77\%$) deformation. In recrystallization texture the components of deformation texture decayed totally or partially, and in their place new orientations appeared.

In case of the steel recrystallized after 9% deformation, morphology of the microstructure (Fig. 5b) and the ODF (Fig. 9 and 10) clearly showed a discontinuous progress of the recrystallization process, i.e.: through nucleation and motion of recrystallization fronts. The place of formation of recrystallization nuclei were probably the deformations zones around large Fe_3C particles.

In case of the steel recrystallized after 77% deformation the observations made with light microscope did not allow to determine whether the recrystallization was discontinuous or in situ, because microstructures after recrystallization annealing (Fig. 5a) and in a state before deformation, i.e. the preliminary heat-treated (Fig. 1) are similar in morphology.

Examination of the microstructure using TEM showed that in the sample 77% deformed and annealed at 700°C for 3 minutes the grain boundaries of ferrite are of a high angle type. It seems that the presence of high angle boundaries formed after such short time of an-

nealing exclude the in situ recrystallization. This may be confirmed by the results of the X-ray tests. They demonstrated the difference between the textures of ferrite in deformed and recrystallized state which point out that the in situ recrystallization does not occur.

7. Summary and conclusions

In the examined Fe-0.67% C steel, a discontinuous matrix recrystallization of a coarse, cold plastic deformed spheroidite occurs after deformation larger than 9%. In case of small deformations ($\sim 9\%$) the most of cementite particles are placed inside of the grains of a coarse-grained matrix. There are also large particles present at the grain boundaries (Fig. 5b).

It was demonstrated indirectly that the discontinuous recrystallization of the matrix after cold plastic deformation of a coarse spheroidite nucleates in the neighborhood of large particles. This is supported by:

- i the dependence of grain size of recrystallized matrix on deformation degree [7];
- ii the arrangement of particles in microstructure (after small deformation ($\sim 9\%$) big particles are at the grain boundaries of recrystallized matrix);
- iii the crystallographic texture of the recrystallized matrix after $\varepsilon \sim 9\%$ deformation (differences in the deformation and recrystallization textures) and,
- iv the TEM microstructure (the presence of the high angle grain boundaries formed after a short time of annealing).

Acknowledgements

The financial support from The Ministry of Science and Higher Education, contract No 3 T08A 038 27 is gratefully acknowledged

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