

# The Effect of Size Stabilization of Carbon Steels Austenite

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**Abstract** – The researches into morphology and faulted substructure of martensite of carbon steels which undergone static (furnace) and dynamic (high-current electron beam) thermal treatment have been conducted using the methods of diffractive electron microscopy. It has been shown that a rapid quenching from a liquid state accompanied by the formation of grain ensemble of submicron-size (0.1–0.5  $\mu\text{m}$ ) leads to first – reduction of size of martensite – phase crystals, second – the fixation of surface martensite effect, third – the formation of one  $\sim 0.3 \mu\text{m}$ -size martensite crystal (grain – martensite crystal) in grains (subgrains).

## 1. Introduction

The effect of martensitic phase morphology alteration as well as martensitic transformation suppression by way of the formation of nano- and submicrocrystalline structure of metals (including steels) has been revealed when researching into a structure-and-phase state of powder materials [1–5]. For cast materials, this effect has not been observed due to the difficulties in obtaining nanocrystalline structure in big volumes.

The present paper gives the analysis of the results obtained in the research into the influence of the size of an individual grain of austenitic phase (size range is from 0.3 to 65  $\mu\text{m}$ ) upon the structure-and-phase state of a quenched steel. Steel grain structure developed as a result of static thermal treatment (austenization, micron-size grains), and electron-beam treatment (submicron-size grains).

## 2. Materials and Methods

For research material, the commercial carbon steel (Fe–0,45C, Fe–0,7C, Fe–0,98C) and alloyed steel (Fe–0,35C–1Cr–3Ni–0,5Mo–0,2V) pretreated by homogenizing annealing with subsequent quenching (860 or 940  $^{\circ}\text{C}$ , 2.5 hours) to produce more homogeneous (for ferritic steels) state regarding phase and structure. Electron-beam treatment conditions are given on Table 1. At every fixed value of electron pulse duration  $\tau$  electron energy  $U$  and electron beam density  $E_S$  corresponded to the melting of surface layer of the material. The depth of the melted layer varied within micrometer unit range. A structure-and-phase state of the surface layer has been studied to the depth of 200–300 nm which is certainly quenched

from a liquid state. Foils were prepared using thin plates (0.2 mm) cut of massive samples parallel to the illuminated surface.

Table 1. Material and treatment conditions

Steel	$U$ , keV	$E_S$ , J/cm <sup>2</sup>	$\tau$ , $\mu\text{s}$	$N$
Fe-0,98C (U10A)	350	48	0,04	10
Fe-0,45C (steel 45)	7–25	6	10	1
Fe-0,7C (U7A)	130–170	80–120	15–40	1

## 3. Results

Study of the steel quenched from 860 and 940  $^{\circ}\text{C}$  allowed us to analyze the influence of the real grain size of original austenite upon the mechanism of martensite nucleation. The study was carried out as follows. The number of packet and plates ( $N$ ) located in grains of a certain size class and the number of packet and plates ( $n$ ) located in grain volume, that is, visually not bounded up with grain boundaries, were defined. Then, the interrelation between grain size, and  $N$  and  $n$  parameters was determined. The conducted analysis revealed that there is a directly proportional dependence between the number of packet and plates and grain size for both austenization temperatures (Fig. 1). Spread of points on the graphs is probably due to the peculiarities of the internal structure and the state of grain boundaries being analyzed which exert influence upon martensite crystals nucleation. Approximating the data of dependence to coordinate origin we arrive to the conclusion that steel quenched from austenization temperature of 860  $^{\circ}\text{C}$  should be accompanied by preservation of original austenite grains not exceeding 4  $\mu\text{m}$  in size, and in case of quenching from 940  $^{\circ}\text{C}$  this effect is possible in grains less than 2.5  $\mu\text{m}$  in size.

It is considered that the main force driving the process of martensite transformation is a dislocation arrangement of the original austenite grain [1, 6]. However, the analysis of the results we have obtained indicates that the nucleation of martensite phase crystals in small grains (6–9  $\mu\text{m}$ ) is observed predominantly at the grain boundary, and in grains of a bigger size – at the boundaries and inside grains – at the boundaries of previously nucleated crystals. Hence, the size of original austenite grain affects not only the

number of sites of original austenite grain nucleation, but the mechanism of nucleation as well. In small grains (6–9  $\mu\text{m}$ ), an exclusively heterogeneous nucleation of martensite at grain boundaries is observed. In grains of a bigger size, martensite crystals nucleation is registered inside grains as well, most likely, at the boundaries of previously nucleated crystals.

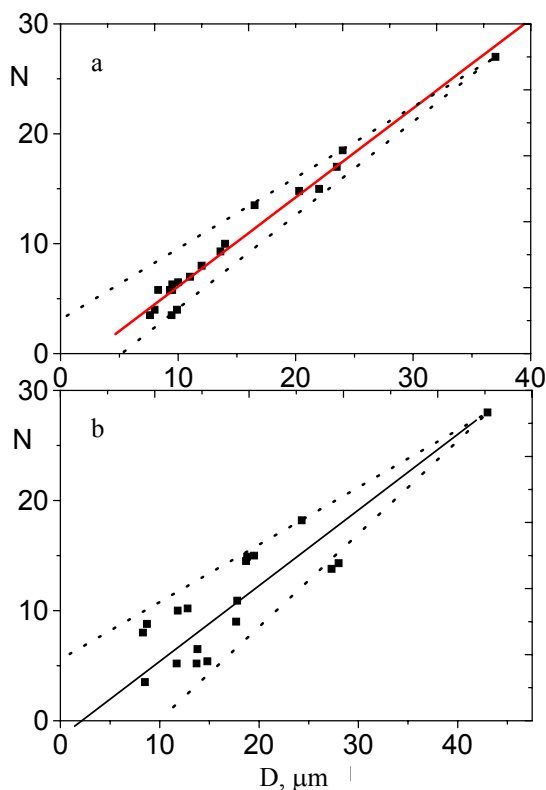


Fig. 1. Number of packets ( $N$ ) settled in grain vs size of grain of initial austenite ( $D$ ) for temperature of austinization 850 (a) and 940 °C (b)

The research into the structure of particular grains allowed us to reveal correlation dependence between grain size and grain area share taken by one packet. When defining the area of a grain ( $S_3$ ), the latter was approximated to a circle; in case of packet and plates, the approximation to an ellipsoid of revolution was done. As it follows from the results given in Fig. 2, area share falling to one packet increases along with the decrease in grain size, and at the limit, for small-size grains ( $D \rightarrow 0$ ) the situation when a grain may be occupied by one packet is real. It should be noted that the results we predicted using the method of approximation were experimentally confirmed in [7] when researching into the structure of laser treated carbon steel. In [7] it was shown that the volume of small ( $\sim 5 \mu\text{m}$ ) grains was often occupied by one pack. We obtained similar results (described below) when studied the martensite structure of steels treated by high-current electron beam (HCEB), that is, heated up and cooled down at super high speeds.

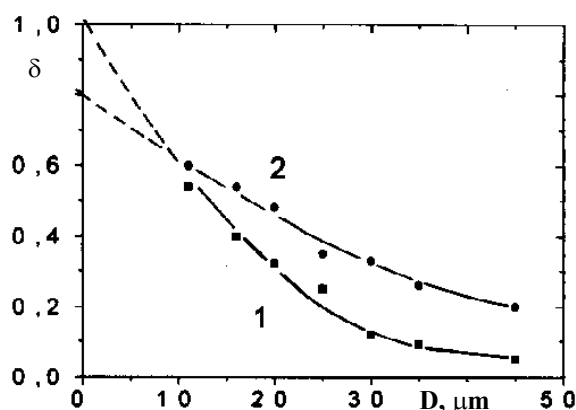


Fig. 2. Fraction of grain area of initial austenite occupied by packet ( $\delta = S_p/S_g$ ) vs size of grain. 1 –  $T = 860 \text{ }^\circ\text{C}$ , 2 –  $T = 940 \text{ }^\circ\text{C}$

The role of size effect in development of martensitic structure in grains of submicron-size range was analyzed in more detail using HCEB as an instrument for obtaining submicron-size grains.

Dynamic recrystallization developing during electron-beam treatment leads to the formation of ensemble of crystallites the size of which varies within the range from tenths of micrometers to micrometers. In accordance with substructure state (and characteristic size) crystallites were divided into two groups.

The first group (0.2–0.6  $\mu\text{m}$ ) included crystallites having net/grid dislocation subarrangement scalar density of dislocation of which is  $\sim 10^{11} \text{ cm}^{-2}$ . Crystallites are monophasic and have volume central tetragonal (BCT) (steel U10A) and volume central cubic (BCC) (steel 45) crystal lattice. The type of dislocation subarrangement, the value of scalar density, and tetragonal crystal lattice distortion allowed to assume that crystallites formed as a result of martensitic  $\gamma \rightarrow \alpha$  transformation.

Two variants of the formation of such a structure are possible. The first variant assumes that crystallites form within austenite temperature stability zone and represent grain (or subgrain) structure of a given iron polymorph. The subsequent cooling leads to martensitic  $\gamma \rightarrow \alpha$  transformation. At the same time one martensite crystal nucleates within the grain. The second variant assumes that steel crystallization leads to the formation of austenite with grain size of micrometer range. The subsequent quenching is accompanied by martensitic  $\gamma \rightarrow \alpha$  transformation with nucleation of a number of martensite crystals in austenite grain which are located perpendicular to the illuminated surface. These martensite crystals have regular geometric shape in a foil section and group in blocks of 6–8 crystals each, blocks average size is 1.5  $\mu\text{m}$ . Azimuthal component of full off-orientation angle –  $\Delta\alpha_{az}$  does not exceed  $\sim 1$  degree between crystals;  $\Delta\alpha_{az} \sim 5$ –6 degree between blocks. By the mode of arrangement the block is similar to martensite crystals

packet which forms when traditional methods of thermal treatment of steel are employed.

In crystallites (grains) of the second group ( $\sim 0.5$ – $1 \mu\text{m}$ ) a different structure is observed. In case of U10A steel, it is close to a classic one which forms in grains at traditional speeds of quenching. Namely, the crystals of plate-type martensite separated by the regions and interlayers of retained austenite are observed within the grain. The particles of a carbide phase of  $\text{Fe}_2\text{C}$  content are observed at the boundaries of martensite crystals and inside them on transformation twins as well. The difference from the traditional martensitic structure consists in high dispersibility of martensite crystals which dimensions are  $L \sim 300$ – $500 \text{ nm}$  and  $D \sim 60$ – $100 \text{ nm}$ . Such a small size of martensite crystals is conditioned by a small size of grains in which the crystals nucleate and by the method of structure formation connected with rapid steel quenching from a melt state. The latter fixes a non-equilibrium state of austenite and at the same time initiates the work of a great number of nucleuses of martensite crystals.

In U7A steel as a result of treatment by high-current electron beam, the grains of  $\sim 0.5 \mu\text{m}$  size the most of volume of which is taken by retained austenite were identified. In case of bigger sizes of original austenite grains ( $\sim 1 \mu\text{m}$ ), rapid quenching leads to almost complete martensite transformation. The structure consisting of the crystals of packet and plate-type martensite and interlayers of retained austenite develops in the volume of grain. Judging by a relative intensiveness of reflexes, volume share of the retained austenite is comparatively small. As has already been mentioned above, small size of martensite crystals deserves special attention: cross size of a packet-type martensite is  $\sim 60$ – $80 \text{ nm}$ , of a plate-type martensite  $\sim 150$ – $200 \text{ nm}$ , longitudinal size is  $300$ – $800 \text{ nm}$ . Earlier, the similar fact of size reduction of martensitic structure of steel when being quenched very rapidly was discussed in [7].

In steel 45, it turned to be impossible to identify retained austenite in significant quantities which was obviously due to a lower concentration of carbon in this material comparing to steels U7A and U10A. In grains of  $\sim 0.5$ – $0.6 \mu\text{m}$  size, as follows from the analysis of microphotographs presented in Fig. 3, the mixture of two morphologic modifications of ferrite is observed. Along ferrite grain boundaries,  $\alpha$ -phase crystals morphologically identical to martensite crystals are observed. The main volume of grain, at the same time, is monocrystal. We may assume that this structure developed in the following way. In the process of quenching, a partial  $\gamma \rightarrow \alpha$  transformation accompanied by the formation of two-phase structure ( $\alpha + \gamma$ ) occurred. The crystals of martensite nucleated in the area of grain boundaries (as it has already been noted, grain boundaries in most cases are preferable sites for martensite crystals nucleation), the rest volume of the

grain did not undergo martensite transformation. The subsequent preparation of the sample for the purpose of foil preparation for electron-microscopic analysis led to the alteration of field of elastic stress resulting from steel quenching, and as a consequence, to the additional  $\gamma \rightarrow \alpha$  martensite transformation retained austenite. We observed such transformations when researching into high-speed steel of S6-5-2 type treated by HCEB [8].

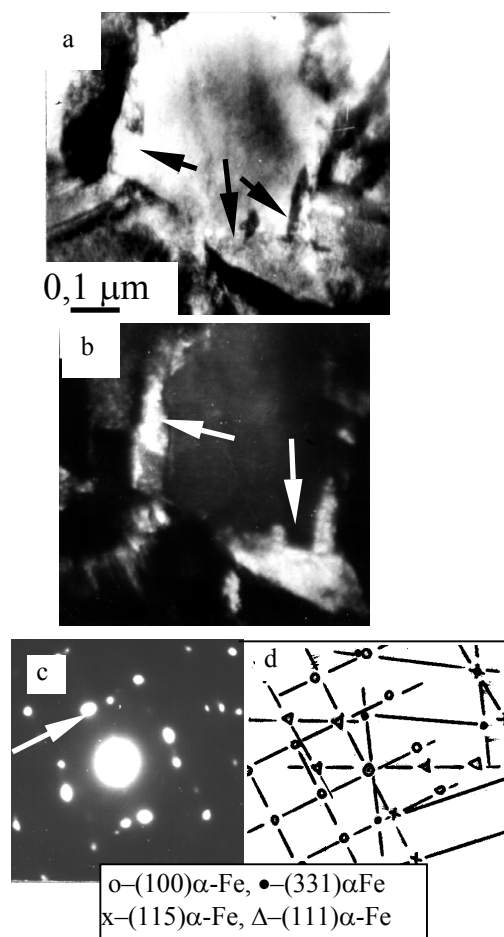


Fig. 3. TEM images microstructure of the near-surface layer of steel 45, irradiated with HCEB. a – bright-field images; b – dark-field images in reflection  $[110] \alpha\text{-Fe}$  (reflections are indicated with arrows); c – diffraction patterns; d – diagram of diffraction patterns. Arrows indicated (a) and (b) martensite crystals

In case of a bigger size of grains ( $\sim 1 \mu\text{m}$ ) in steel 45 (the same as in the cases considered above), martensitic structure consisting of crystals of a packet-type and plate-type martensite develops. Thus, it can be concluded from the results obtained, that a rapid hardening from a liquid state accompanied by the formation of submicron-size grain ensemble leads to first – size reduction of martensite phase crystals, which was earlier noted, second – the fixation of surface martensite effect, third – the formation of one  $\sim 0.3 \mu\text{m}$ -size martensite crystal (grain – martensite

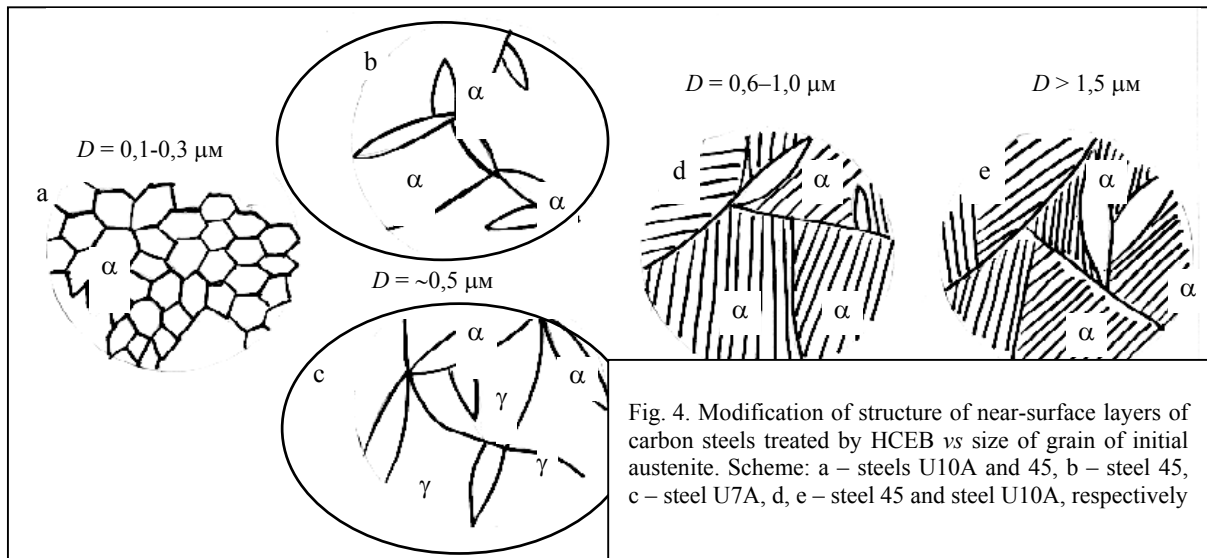


Fig. 4. Modification of structure of near-surface layers of carbon steels treated by HCEB vs size of grain of initial austenite. Scheme: a – steels U10A and 45, b – steel 45, c – steel U7A, d, e – steel 45 and steel U10A, respectively

crystal) in grains (subgrains). Fig. 4 represents a schematic evolution of martensite phase morphology in carbon steels quenched from a liquid state with regard to the size of austenite of submicron range.

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