Modification of the Structure And Phase State of a Ferrite-Cementite Composition by an Electron Beam

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Abstract – The phase composition and defect substructure formed in carbon steel subjected to a microsecond high-current e-beam have been studied by thin-foil electron diffraction microscopy. It has been shown that the mechanism and the degree of globular cementite dissolution, the phase composition and the morphology of the appearing structure depend on the distance from a melt spot and are determined by the temperature field gradient. As the electron beam approach the "spot" (an increase in steel temperature), the evolution of the material volume containing cementite particles is accompanied by the formation of structures which alternate in a regular manner.

1. Introduction

The eminently flexible sources of dynamic thermal treatment of materials (as compared to the most-used laser sources) are intense pulsed electron beams (PEB's) [1]. The main advantage of this method of treatment leis in the possibility to widely vary (by varying the electron energy) the depth of electron penetration into a material and thus the dynamics of thermal fields and the voltage (stress) wave parameters. The possibility of widely varying the energy density, the pulse duration, and the electron energy in combination with near-complete electron absorption and with bulk energy liberation make the PEB method unique and highly efficient both for studies of the physics of nonequilibrium structural-phase states of a solid and for purposeful modification of the structure and properties of metal materials in order to improve their service characteristics [1, 2].

The objective of this work was to assess the regularities of structural and phase transformations occurring on dissolution of globular cementite in carbon steel treated by an intense electron beam.

2. Materials and Experimental Procedure

The material to be examined was carbon steel (0.7 weight. % C, 0.3 weight.% Mn, 0.2 weight.% Si, 0.2 weight.% Cr) required for much of industry. Before placing into the working chamber, the specimens in the form of cylinders of diameter 15 mm and height 7 mm were grinded, electrolytically polished, and cleaned from organic impurities in an ultrasonic bath. Electron-beam treatment of the specimen was realized on an accelerator with a plasma electron source based on a cold-cathode low-pressure arc discharge [3]. The mode of electron beam treatment, the parameters of the beam, and the results of calculations are considered in detail elsewhere [4]. The phase composition and the defect substructure of the steel were investigated by the method of thin-foil electron diffraction microscopy. The objects under study (thin foils) were obtained by electrolytic thinning of plates cut from the treated specimens by the electroerosion method. We analyzed the phase composition and the state of the defect substructure of a heat-affected zone in a nearsurface layer of thickness ~0.2–0.5 μ m, including the specimen surface.

3. Results and Discussion

In the initial state carbon steel is a α -iron-based polycrystalline aggregate whose volume and grain boundaries are occupied by globular cementite particles of submicron sizes (mainly 0.3–0.7 µm).

High-rate melting and crystallization result in a hardened structure in the spot of e-beam exposure. This structure consists of martensite crystals (~65% of the material volume), islands of the residual austenite and small amount of the initial cementite. Beyond the spot of irradiation (the heat-affected zone) the globular cementite particles are conserved to a greater extent. This allowed tracing the evolution of the structuralphase state of material volumes containing the given particles at different stages of their dissolution. It was assumed that the kinetics of dissolution of globular cementite particles in the heat-affected zone and associated structural-phase states of the material (the state of a material in the immediate vicinity of a cementite globule) are determined in the main by the distance to the melt spot boundary.

The initial stages of transformation of globular cementite particles under the action of the heat transferred to the treated material by the electron beam are found to occur at a distance of $\sim 10-12$ mm from the melt spot boundary. These transformations are in the formation of a defect layer with a thickness of tens of nanometers in a particle along the interface with the matrix. On a dark-field image the substructure of this layer manifests itself as a spotted contrast (Fig. 1,a).



Fig. 1. Electron image of the structure formed in the heat-affected zone due to transformation of the globular cementite particle; a, c – dark fields obtained in the reflexes [011] Fe₃C (a) and [110] α-Fe+[102] Fe₃C+ [125] Fe₂C (c); b, d – electron-diffraction patterns; the dark field reflex are indicated by the arrow

The microdiffraction pattern taken from the particle has a point structure (Fig. 1,b). Comparison of these facts allows the assumption that the spotted contrast of the sublayer formed in the cementite particle is governed by its defect structure, namely, by the presence of a dislocation substructure. In so doing, the cementite globule is still a single-crystal formation, yet a great number of bend extinction contours is observed in its volume that is indicative of elastic distortions of the crystalline lattice. These contours, as a rule, originate and terminate at the "particle – α -matrix" interface. It is apparent that the stress fields induced by the difference in the coefficients of thermal expansion of the cementite particle and α -matrix are responsible for the appearance of these extinction contours as well as for the defect sublayer.

Indexing of the selected area diffraction pattern taken deep in the cementite has made it possible to reveal weakly pronounced additional reflexes, along with cementite reflexes, which (presumably) belong to iron carbide of composition Fe_2C . One would expect that these particles are arranged along the "carbide-matrix" interface and result from dissolution of the cementite globule on fast heating and cooling in the course of e-beam treatment.

The next stage of transformation of the material is accompanied by the formation of some intermediate layer in the α -phase volume along the interface with the carbide (Fig. 1,c). This layer is separated from the matrix by a large-angle boundary (Fig. 1,c). This is likely to point to the polymorphous $\alpha \rightarrow \gamma \rightarrow \alpha$ transformation in the steel volume adjacent to the particle. A dark-filed image of this layer reveals its complex structure. Namely, the layer consists of quasi-isotropic α -phase subgrains (crystallites) with an average size of ~60 nm. The crystallites are misoriented as evidenced by the essentially different contrasts of their dark-field images (Fig. 1,c) as well as by splitting of the α -phase reflexes on the selected area diffraction pattern (Fig. 1,d). Using known relations [5], which allow an estimate of the azimuthal component β_{az} of the total misorientation angle of α -phase crystallites from the value of reflex splitting, it can be shown that $\beta_{az} \sim 5$ degrees. The α -phase crystallites on the dark-field image have a spotted contrast characteristic for the net dislocation substructure of carbon steel martensite crystals. Consequently, it can be assumed that the α phase crystallites result from the polymorphous $\alpha \rightarrow \gamma \rightarrow \alpha$ transformation by the martensitic mechanism.

Indexing of the selected area diffraction pattern taken from the material volume containing a cementite globule and an intermediate layer has revealed, among cementite and α -matrix reflexes, reflexes of an additional phase which show the best correlation with the Fe₂C crystalline lattice (Fig. 1,d). Analysis of the dark-field image has made it possible to reveal particles of this carbide. It has been found that they are arranged along the intermediate layer interface both from the side of the cementite globule and from that of the α -matrix (Fig. 1,c). The average size of particles of this carbide is ~12 nm.

As the spot of e-beam exposure is approached, the thickness increases and the phase composition of the intermediate layer becomes more complicated (there appear γ -phase reflexes), the morphology of the α -phase forming it changes (lenticular crystallites are found).

The next stage of transformation of the cementite globule is associated with a change of the mechanism of failure of the carbide (liquid-phase dissolution of the globule, along with solid-phase dissolution, is registered). A two-layer transition structure is formed around the particles (Fig. 2). The layer adjacent to the dissolving particle consists of round α -phase crystal-lites of average size ~40 nm. The layer adjacent to the

 α -matrix surrounding the particle is formed by martensite crystals and residual austenite islands. Besides, as in the previous cases, there are nanoparticles of the second phase in the transition layer volume which are most probably identified as cementite particles. Obviously the sublayer adjacent to the particle results from liquid-phase dissolution, i.e., from contact melting of the steel along the "carbide-matrix" inter-



Fig. 2. Electron image of the structure formed due to transformation of the globular cementite; a – light field; b – dark field obtained in coincident reflex $[002]\gamma$ -Fe+ $[110]\alpha$ -Fe + [112]Fe₃C; c – electron-diffraction patter; the dark field reflex is indicated by the arrow



Fig. 3. Electron image of the carbon steel structure formed beyond the irradiation spot due to transformation of globular cementite particles: a – light field; b – electron-diffraction pattern, dark filed reflexes are indicated by arrows; c, d – dark fields obtained in the reflexes $[002] \gamma$ -Fe (c) and [130] Fe₃C (d)

face; the sublayer located from the side of the matrix surrounding the globule is brought about by solidphase dissolution of the cementite. High-rate quenching led to suppression of the leveling carbon diffusion and assisted in the formation of α - and γ -Fe nanocrystallites.

As the melt spot is approached, the degree to which the cementite particles are dissolved increases. This causes breaking of the transition zone symmetry. Crystallographically directed regions, along with a spherical transition region, are formed around the dissolving particles. As follows form analysis of the corresponding selected area diffraction pattern, these regions have a two-phase structure and consist of martensite crystals and interlayers (islands) of the residual austenite.

The structure formed at the final stage of globular particle dissolution is shown in Fig. 3. Complete dissolution of particles and subsequent high-rate crystallization of the melt in this steel volume results in socalled lamellar eutectic which consists of alternating ferrite, austenite, cementite lamellae. A similar structure was revealed earlier during high-rate crystallization of an eutectic Fe-C alloy [6].

4. Conclusion

Thus, the analysis of the structural-phase transformations occurring in high-rate melting and recrystallization of steel containing globular cementite particles has shown that the mechanism, the extent of cementite dissolution, the phase composition and morphology of the structure formed therewith depend on the distance from the melt spot and is determined by the temperature field gradient. Dissolution of globular cementite particles is accompanied by the formation of a transition layer of complicated structure and phase composition. As the melt point is approached, the mechanism of solid-phase dissolution changes over to that of liquid-phase dissolution.

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