RESEARCH OF PHASE TRANSFORMATION PRODUCTS IN HIGH STRENGTH STEELS BY ELECTRON BACKSCATTER DIFFRACTION (EBSD)

E.A. Yashina, A.A. Zisman, D.Sc., S.N. Petrov, Ph.D.
CRISM "Prometey", St. Petersburg, 191015, Shpalernaya str., 49, npk3@crism.ru

Abstract: In the present work, in order to evaluate the metastable structural state of high-strength steel immediately after the phase transformation as well as during the following tempering, the SEM-based EBSD is employed. The lattice orientation gradient in the alpha-phase of steel is mapped by a standard GAM function (“grain average misorientation”) that makes it possible to identify various types of martensite and bainite distinguishable in the dislocation density and phase stresses. The GAM sensitivity to the crystal curvature not only allows one to tell between different types of alpha-phase in the non-equilibrium quenched state but also reveals their change during tempering.

KEYWORDS: NON-EQUILIBRIUM QUENCHED STRUCTURES, HIGH-STRENGTH STEEL, MARTENSITE, BAINITE, EBSD, LATTICE ORIENTATION GRADIENT, GAM

1. Introduction

Complex bainite-martensitic structure of high-strength cold-resistant steels, which is a result of phase transformation in quenching, provides a unique combination of properties. Analysis of it remains an actual task since some species of alpha-phase, slightly differing in morphological characteristics, vary considerably in dislocations density and hence their contribution to the strength of material. This applies both to non-equilibrium as-quenched state and the structure evolution during subsequent tempering. Thus, studies of the final structure require new means of objective estimation. Low resolution of optical microscopy does not allow solving this problem, and transmission electron microscopy due to its locality does not provide evaluation on statistically representative volumes of steel.

In recent years the possibilities of panoramic crystallographic analysis based on scanning electron microscopy (SEM) dramatically increased in practical metallography owing to application of electron backscatter diffraction (EBSD) method. It provides a resolution of about 100 nm and an angular accuracy of about 0.1° to map the local crystal orientations on conventional sections [1]. However, in order to distinguish between the types of alpha-phase with different density of defects in the lattice and degree of its elastic distortion, some alternative functions of EBSD data are used [2, 3].

2. Materials and research techniques

Under consideration is high strength low carbon steel 09CrNi4MoCu with complex bainite-martensitic structure after quenching. By varying the cooling rate of dilatometric samples (diameter 5 mm, length 10 mm) the required structures with different ratios of the structural components were formed. Samples from rolled plates were heated in a vacuum chamber of dilatometer DIL 805 with rate of 5 °C/s to a temperature of 950 °C and kept at this temperature for 300 s and then cooled with rates 1 and 30 °C/s.

Similarly, the sections from rolled steel 09CrNi4MoCu in quenched condition and after tempering at temperatures of 625 °C (8 h) and 660 °C (1 and 6 h) were examined.

The sections were prepared by standard metallographic procedures, and the fine paste of amorphous aluminum oxide was applied at the final stage to reduce surface hardening. Panoramic EBSD analysis was carried out using SEM QUANTA 200 3D with scanning step 0.10 microns, the software EDAX-TSL was applied to determine the local orientations.

The standard option of the software package TSL OIM Analysis, namely the GAM-function, was used to evaluate the lattice curvature in each crystal (laths or blocks) of the transformed structure. This function characterizes the average angle of the lattice rotation between adjacent measurement points within the area, separated from the environments by boundary with misorientation of not less than specified tolerance angle θt. In the present work the value θt=5° was used that is close to the smallest relative rotation angle between the possible variants of orientation relationships (OR) between alpha-phase and a parent austenite lattice [4, 5]. On the one hand, this excludes the contribution of boundaries between different martensite or bainite crystals to the GAM-value, i.e. reflects only their own curvature; on the other hand, each crystal is characterized as a whole, because low-angle boundaries (θ<θt) inside it are thus ignored.

3. Results and discussion

3.1. Dilatometric experiments

Analysis of dilatometric curves (Fig. 1) shows that at a cooling rate 30 °C/s transformation starts at temperature of approximately 400 °C, and the most part of it takes place below Ms (for steel 09CrNi4MoCu martensite start temperature Ms is ~ 390 °C); at a cooling rate 1 °C/s transformation starts in bainite region at 440 °C and finishes at approximately 340 °C.

Fig. 1 – Dilatometric curves of steel 09CrNi4MoCu cooled with rate 1 (a) and 30 °C/s (b)
Metallographic sections of steel 09CrNi4MoCu proved that at a cooling rate 1 °C/s microstructure consists of bainite and martensite (Fig. 2, a), whereas at a cooling rate 30 °C/s microstructure represents predominately martensite with a small part of bainite (Fig. 2, b).

Fig. 2 - Microstructure of steel 09CrNi4MoCu after cooling at a rate 1 (a) and 30 °C/s (b)

Fig. 3 represents lattice curvature (GAM) maps after cooling of steel at a rate 1 (a) and 30 °C/s (b). To image the lattice curvature, the color scale [3, 6] was used together with previous data on its relation with different types of α-phase after quenching. Typical lattice curvature magnitude lies in ranges 0.4-0.7° and 0.7-1.25° for bainite and martensite, respectively. With this scale it was identified that after quenching with cooling rate 1°C/s, steel 09CrNi4MoCu contains 75% of bainite and 23% of martensite, whereas at cooling rate 30 °C/s – microstructure consists of 78% martensite and 20% bainite.

Fig. 3 - Lattice curvature maps of steel 09CrNi4MoCu after cooling at a rate 1 (a) and 30 °C/s (b)

Detailed research of features of phase transformation products after cooling at rate 1 and 30 °C/s was carried out in terms of lattice curvature spectrum (Fig. 4).

Fig. 4 – Lattice curvature spectrum of steel structure after cooling at rate 1 (dashed line) and 30 °C/s (solid line)

One peak, corresponding to bainite-martensite structure is observed for cooling rate 1°C/s. Possibly, high start transformation temperature leads to formation of bainite and so-called ‘self-tempered’ martensite, both having close dislocation densities. It might be supposed that the specific shape of spectrum is due to overlapping of contributions of two structural components.

Raise of cooling rate to 30°C/s leads to increase of average lattice curvature from 0.62° up to 0.81°, and the separation of structural components by lattice curvature is observed. This splitting seems to correspond to severely distorted lath martensite, ‘self-tempered’ martensite and bainite.

Thus, panoramic EBSD analysis proves to be an efficient means for quantitative attestation of bainite-martensitic structures of high strength steels. This permits both to distinguish between structural α-phase species differing in lattice distortion and to study the structural features of phase transformation products in terms of lattice curvature spectrum.

Research of industrial probes

The developed method was verified on specimens cut from 09CrNi4MoCu steel plates after quenching in industrial conditions. Lattice curvature map of steel 09CrNi4MoCu after quenching (Fig. 5, a) reveals the prevalent fraction of martensite in structure. Tempering at 625°C leads to abrupt decrease of lattice curvature level (Fig. 5, b), so that the lattice curvature of tempered martensite turns to be close to that of bainite.
For detailed analysis, it should be taken into consideration that structural species of $\alpha$–phase differing in density of defects and degree of elastic distortion, and consequently in lattice curvature, change during tempering in different ways (Fig. 6, a). The quenched microstructure proves to have bimodal distribution with main martensite peak and relatively small peak due to insignificant fraction of bainite.

**Fig.5** - Lattice curvature maps of steel 09CrNi4MoCu after quenching (a) and tempering at 625°C (b)

During tempering, the significant change of spectrum shape is observed along with the expected total decrease of lattice curvature. If to ignore the weakest peculiarities, the martensite peak (in contrast with bainite) is split by the slow shift of its right part corresponding to the highest dislocation density. It may seem strange because the elastic energy, i.e. the driving force for structural evolution must be maximal. At the same time, from the kinetic point of view this split may be expected, since the excessive density of dislocations complicates their rearrangement (annihilation). Along with the influence of initial dislocation density, the observed effect may be explained by different width of martensite laths, resulting in different spectrum evolutions during tempering. Although the result of the competition of all above mentioned factors depending on tempering conditions is still hardly predictable, the example demonstrates efficiency of lattice curvature spectrum as a representative characteristic for structural states of $\alpha$–phase.

**Fig.6** – Lattice curvature spectrum after quenching (dashed line) and tempering at 625°C (solid line) of steel 09CrNi4MoCu

**Evolution of inter-variant misorientation spectrum in tempering**

Martensitic and bainitic microstructures are reliably recognized, according to [4, 5], by spectrum of inter-variant misorientations after quenching, however the research of tempering kinetics in a similar way has not been implemented yet. Seemingly, this approach was ignored due to the common assumption that the tempering effect involves only the reduction of dislocation density and carbon redistribution, whereas the packet-lath structure remains unchanging.

Orientation relationship (OR), determined in steel according to [6] is characterized by Euler angles ($27.89^\circ$ $8.81^\circ$ $18.25^\circ$) for rotation of elementary cubic cells during $\gamma$–$\alpha$ transformation and corresponds to inter-phase rotation angles of $1.59^\circ$ и $2.58^\circ$ for close-packed planes and directions, respectively. Fig. 7 shows variant pairing frequency diagrams after quenching (a), tempering at 625°C for 8 h (b), at 660°C for 1 h (c) and 6 h (d). After quenching (Fig. 7, a) the typical spectrum for martensitic structure was obtained [4, 5], however tempering led to an unexpectedly strong evolution. First of all, increase of variant pairing $V_2/V_1$ fraction and decrease of $V_4/V_1$ fraction should be mentioned. The longest tempering at 625°C resulted in the weakest effect comparing to tempering at 660°C for 1 h. Increase of tempering time up to 6 h at 660°C (Fig. 7, d) led to notable growth of boundaries, corresponding to variant pairing $V_2/V_1$.

**Fig. 7** - Variant pairing frequency diagrams after quenching (a), tempering at 625°C for 8 h (b), at 660°C for 1 h (c) and 6 h (d)
The revealed regularities indirectly evidence that tempering leads to the growth of martensitic crystals, which orientation corresponds to V2/V1 variant paring.

4. Conclusions

The present work shows that EBSD analysis is an efficient means for quantitative attestation of complex bainite-martensitic structures of high strength steels. It permits to distinguish between structural species of α–phase differing in lattice distortion and dislocation density. In particular, volume fractions and the distribution of martensite and bainite can be derived from lattice curvature maps. Rate of softening of different types of α–phase in tempering is also manifested by evolution of curvature spectrum. Thus, different resistance of bainite and martensite to tempering (softening) is revealed.

EBSD data processing enables to determine the inter-phase orientation relationship and to analyze variant pairing at the boundaries. On this base, the important tempering effect was discovered in steel under investigation – the increase of grain boundaries with V2/V1 variant pairing.

This work is financially supported by Board of Education and Science of Russian Federation, grant №14.579.21.0003, unique identifier RFMEFI57914X0003.

References


