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Effect of ausforming and cooling condition on the orientation relationship in martensite and bainite of low carbon steels

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A method to determine the orientation relationship (OR) in bainitic and martensitic transformations has been employed that does not use reconstruction of prior austenite grains. It is shown that the method keeps accuracy regardless of orientation non-uniformity induced inside austenite grains by hot deformation. The revealed OR behavior for bainite formed under continuous cooling agrees with its temperature dependence previously found in case of isothermal transformation. Ausforming effect on the OR detected in martensite is presumably due to deformation induced subgrains separated by low-angle dislocation boundaries.

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1. Introduction

Mechanical properties of high strength low alloy steels depend on crystallographic characteristics of the microstructure, in particular misorientations between its crystalline elements [\[1,2\].](#page--1-0) These misorientations are predetermined in turn by an orientation relationship (OR) between the final α and parent γ phase as well as by certain pairing variants of the same OR [\[3,4\].](#page--1-0) Correspondingly, to find the OR depending on the cooling rate or isothermal holding temperature [\[4,5\]](#page--1-0), ausforming [\[6\]](#page--1-0) and chemical composition [\[7\]](#page--1-0) is a problem of vital importance.

Formerly, to determine OR, the diffraction analysis with the transmission electron microscopy (TEM) was applied to selected areas containing islands of retained austenite [\[2,8\],](#page--1-0) however the latter is hardly perceptible in most low carbon steels. Later on, such a limitation has been overcome by means of electron backscatter diffraction (EBSD) [\[4,](#page--1-0) [6,7,9](#page--1-0)–11]. Usually the application of EBSD technique for OR determination includes the reconstruction of prior austenite grains [\[7,10](#page--1-0)–14], though this may result in an essential error when crystal orientations within them are inhomogeneous because of ausforming. In order to reduce inaccuracy due to the orientation gradient inside austenite grains, it has recently been proposed [\[15\]](#page--1-0) to derive the OR from interfacial misorientations near a triple line of α phase crystals transformed from the same prior grain. However, such a method allowing only for limited data on several triple junctions can hardly provide a result significant statistically. Moreover, alternative (representative) methods free of the above-mentioned limitation become necessary insofar as actual ORs always vary around some virtual "centers" [\[16\]](#page--1-0) rather than coincide with the latter.

Determination of OR in the present work does not involve any reconstruction of austenite structure undergoing the γ - α transformation, but is based only on interfacial misorientations in the transformed state. Recently this approach has been employed to determine OR in the bainite of rolled microalloyed steel [\[17\].](#page--1-0) Notice also that it apparently is very close to the method suggested by Gomes and Kestens [\[18\]](#page--1-0). Such an approach filters off influence of orientation gradients in deformed γ grains, whereas account for all the revealed boundaries notably increases significance of the derived OR. Dependence of the latter on the deformation substructure of γ grains and on the cooling rate is analyzed in martensite and bainite of high strength low carbon steels.

2. Experimental

The two considered steels further denoted by letters M and B have, respectively, compositions $0.084C - 0.33Si - 0.35Mn - 1.1(Cr +$ Mo)–5.2(Ni + Cu) and 0.090C–0.27Si–0.56Mn–0.83(Cr + Mo)– $2.65(Ni + Cu)$ (wt.%). Thermal treatments were conducted using a Bahr DIL-805 dilatometer. Mean size of the former austenite grains, dγ, was determined by the thermal etching in vacuum [\[19\].](#page--1-0)

Specimens \varnothing 5 × 10 mm of martensite (M) steel were reheated at rate 40 °C/s to 1100 °C, hold for 100 s ($d_2 \approx 80$ µm) or 300 s $(d_γ ≈ 160 μm)$ and then cooled at 40 °C/s to 800 °C. At this temperature specimens were compressed to 30 or 50% reduction (true strains of 0.36 or 0.69) at strain rate 1 s⁻¹ and finally cooled at 30 °C/s in argon to room

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Table 1 Description of specimen treatment.

Specimen	d, µm		$0 - i$ 'C/s	Specimen	d, µm	c	0 ₀ 'C/s	Specimen	t. um	ິ	\circ \mathbf{V} 'C/s
Mf ₀ Mf30	80 80	0.36	30 30	Mc0 Mc30	160 160	0.36	30 30	B ₅ B50	47 47		50
Mf50	80	0.69	30	Mc50	160	0.69	30	B50a	47	0.36	50

temperature. The deformation-free processing of the bainite (B) steel included reheating at 5 °C/s to 1100 °C, holding at this temperature for 1 min and then cooling at 5 or 50 °C/s. To study the effect of ausforming, the specimens of steel B were cooled from 1100 °C at 5 °C/s to 850 °C, then deformed to strain 0.36 at strain rate of 1 s^{-1} and finally cooled in argon at 50 °C/s. Details of all treatments and denotation of specimens are given in Table 1.

The temperature of martensite transformation start determined for steel M by dilatometer measurements is about 400 °C [\[20\]](#page--1-0), that agrees with calculation according to Andrews empirical formula [\[21\]](#page--1-0). The hot deformation of austenite reduces M_s by no more than 10 °C. In steel B bainite is formed at all treatments used [\[20,22\].](#page--1-0) Under cooling at the rate of 5 °C/s the transformation was developing within the wide temperature range from about 590 °C to 410 °C. Under accelerated cooling without ausforming the transformation begun at about 440 °C and then rapidly completed at 400 °C. In the latter case [\[20\]](#page--1-0) we get mainly a low-temperature bainite because the undercooling and respective fraction of athermal martensite are small since $M_s \approx 430$ °C for steel B [\[21,23\].](#page--1-0) The hot deformation of austenite preceding the accelerated cooling leads to a significant increase of the temperatures, at which bainite transformation occurs: it develops now within the range from \sim 500 °C to 400 °C.

EBSD analysis was carried out with SEM Quanta 3D FEG equipped with field emission gun (FEG), the accelerating voltage was 20 kV. Specimens were mechanically polished and then electropolished in $HClO₄ + CH₃COOH$ solution. The scanning step was 0.2 to 0.4 μ m and a EDAX Pegasus system was used to process the obtained data. The resulting microstructure images are shown in [Fig. 1.](#page--1-0)

3. Approach to determination of orientation relationship

A notable fraction of boundaries with misorientations about 5° or more separate crystals of α phase nucleated and grown within the same γ grain. The larger prior γ grains, the higher this fraction, whereas remaining boundaries coincide with those of γ grains. Owing to symmetry of cubic lattice, an OR predetermines certain set of 24 orientation variants, designated as $V_1,...V_{24}$, which α crystals formed within the same γ grain can have¹. Since inter-variant misorientations are predetermined by the OR, the latter can be restored from sufficient misorientation data. To solve this problem is to find the OR, which gives the minimum quadric deviation Δ of calculated from measured misorientations. The minimization is performed with an iterative procedure gradually diminishing Δ as follows.

Let us denote the misorientations measured at the boundaries (namely, misorientations for couples of points, between which the boundary has been identified) as M_i , $i = 1$, n, where n is the total amount of the misorientations. The inter-variant misorientations related to a fixed OR with Euler angles $(\varphi_1^{\text{OR}}, \varphi_2^{\text{OR}}, \varphi_2^{\text{OR}})$ are denoted as $W_j =$ $V_1V_j^{-1} = W_j(\varphi_1^{0R}, \varphi^{0R}, \varphi_2^{0R}), \ \ j = 2, 24.$ The deviation of a measured misorientation from its calculated counterpart is

$$
\delta_i\left(\varphi_1{}^{OR}, \varphi_2{}^{OR}, \varphi_2{}^{OR}\right) = \min_{j=2,24} \left(\delta_{ij}\left(M_i, W_j\right)\right),\tag{1}
$$

where δ_{ij} is angular difference between M_i and W_j . When searching OR by discrete steps in the orientation space, the procedure eventually stops at an optimal OR such that $\Delta(\varphi_1^{OR}, \varphi_2^{OR}, \varphi_2^{OR})$ is minimum.

In calculating Δ the following issues should be allowed for. First, even with lowest-angle boundaries ($\theta < 2^{\circ}$) neglected, there remains a lot of misorientations between lathes within the same block. Second, some portion of measured misorientations are due to boundaries between blocks originated in different γ grains, i.e., interfaces between the latter. Focusing on inter-variant boundaries between blocks, it is pertinent to exclude the above mentioned issues which could hinder the optimization procedure.² Therefore, the mean deviation is expressed as

$$
\Delta\left(\varphi_1^{\text{ OR}}, \varphi^{\text{ OR}}, \varphi_2^{\text{ OR}}\right) = \sqrt{\sum_{k=1}^m \left(\delta_k\right)^2 / m},\tag{2}
$$

where the summation includes only misorientations deviated from the inter-variant ones by no more than certain ε ($\delta_k = [\delta_i \le \varepsilon]$), and m is the total number of such misorientations. The selected tolerance ε should be a compromise between the significance of the misorientation data and necessity to exclude random boundaries from the analysis. As it was noted above, the present method is similar to that suggested by Gomes and Kestens [\[18\].](#page--1-0) The latter method is yet only briefly outlined in [\[18\]](#page--1-0), and thus one cannot judge the similarity and difference of the techniques.

In our calculations ε was chosen so that not less than 90% of actual boundaries have been included in the procedure. The latter was performed in two stages in order to determine the OR with a sufficiently high accuracy, while consuming the minimum time. The first optimization was done by searching OR with a coarse step (-1) ° starting with any well-known OR, e.g., the Kurdjumov–Sachs one. 3 Therefore, a region of the Euler space in the vicinity of the starting point was scanned. The values of Δ for a current point and its closest neighboring points in the Euler space were calculated at every step, and the point with the smallest Δ was taken as new current OR. If all neighboring points had the value of Δ bigger than the current one, then optimal OR was considered to be found to a first approximation. Thus determined OR, which is already close to the optimal one, can be then used as a starting point for the final refined optimization with a smaller step (-0.1°) . One can choose the ε value for either stage from the plots given in [Fig. 2,](#page--1-0) where the length fraction of boundaries close to the inter-variant ones is presented as a function of ε. According to these plots, tolerance values ε of 8° and 5°, respectively, appear to be appropriate for the first and second stages considered above.

4. Results and discussion

It turned out that the ORs determined for steel M on different regions of the same specimen show acceptable scattering only among regions of not less than 80 μm dimensions. Taking this into account, four areas of 80 \times 80 μ m² were analyzed on each specimen in order to

¹ The martensite or bainite crystals composed of slightly misoriented lathes are referred to as blocks [\[3\].](#page--1-0)

² Though some intra-block boundaries and former austenite boundaries anyway can get involved into consideration, usually their fraction is not so large to influence considerably a precision of the OR determination.

³ Sensitivity to the starting OR was not examined systematically. At the same time, taking the Greninger–Troiano OR instead the Kurdjumov–Sachs OR as the starting one does not change the resulting OR.

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