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GRAIN-BOUNDARY ENGINEERIGN IN MAKING TUBES OF LOW-CARBON STEEL

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Introduction

In most cases, the technology of rolling metal products includes various types of hot deformation with its schedules being responsible for formation of grain-boundary and subgrain structures. Grain size *d* is the main measure in this process as it influences the majority of crystalline material properties in accordance with various variants of Hall-Petch equation [1, 2]. However the physical meaning of this equation consists sooner in the distance passed by dislocations from one grain boundary to another than in the grain size proper and in the force necessary to transfer glide from one grain to another. That is why differences in the atomic structures of the grain boundaries exert a material effect on the value of their specific surface energy and consequently on the polycrystalline material properties.

In recent decades, studies in the metal and alloy structures pay a particular attention to special low energy boundaries. Practical application of particular characteristics of special boundaries (SB) has become feasible due to the development of the concept of coincident site lattices and the studies in atomic structures and crystallographic parameters of SB [3-7].

An idea of grain-boundary engineering (GBE) of materials suggested by T. Vatanabe in 1984 [3] has come into existence.

Its essence consists in such control of temperature-deformation production processes which favors creation of a microstructure with a large number of special grain boundaries $\Sigma 3$ and their derivatives $\Sigma 3^n$ [5, 6]. Increase in the number of such boundaries results in improvement of a set of physical, mechanical and corrosion properties of metal products [3, 4] including tubes operated in conditions of high-temperature creep [8].

As the tube production process includes a cycle of thermal treatments and working, GBE can be fruitful and prospective for its realization in the tube industry.

This paper objective *is elucidation of effect of hot-deformation scheme upon the control of quantitative composition and structure of grain boundaries and properties of the low carbon steel tubes used in power generating systems.*

Material and procedures used in the study

As a study material, tubes of steel 20 produced by hot working using two technological schemes: rolling and extrusion were used. ∅219×16 mm and ∅219×25 mm tubes were hot-rolled in a tube rolling unit with a plug mill and ∅219×9 mm and ∅219×20 mm tubes were hot-extruded in a 55 MN extrusion unit.

Structural condition of the tube metal was examined using metallographic and electron microscopy methods. A special attention was paid to quantitative assessment of special grain boundaries in ferrite.

For a more detailed identification of the grain boundary structure in steel by metallographic method, metallographic sections were subjected to special electropolishing. Etching was done in two steps: first in a 1% HNO_3 solution in ethanol with an addition of detergent (for a more clear identification of low-angle and special boundaries which etch much more weaker than ones of the general type) and then a deep etching was applied in a 4% HNO_3 solution in ethanol.

Special boundaries were identified by the features described in [9, 10] allowing identification of special boundaries at a probability about 95%:

- sharp knees at acute and oblique angles (facets) *always* indicate that the boundary in question belongs to special boundaries;
- boundaries entering with their one or both ends into triple junctions with the formation of opposed angles close to 180° are special boundaries (Σ=3).

SB's were counted by linear-intercept method. The relative quantity of special boundaries λ_{spec} was determined as a ratio of specific length of special boundaries ($\Sigma l_{i(spec)}$) to the total length of all boundaries in the specimen unit area ($\Sigma l_{i(total)}$ + $\Sigma l_{i(spec)}$):

$$\lambda_{spec} = \frac{\Sigma l_{i(spec)}}{\Sigma l_{i(total)} + \Sigma l_{i(spec)}}$$

To find Σl_i, length l_i of each intersected boundary was measured with no account of its curvature and a total facet length was estimated in a case of special boundaries. It ensured higher measurement accuracy at their small numbers. If counting is confined by just determination of the number of intersection points, the number of measurements should be increased considerably.

Besides, in order to study grain-boundary structures in boiler tubes of steel 20, electron raster microscopy was applied with analysis of pictures of electron backscatter diffraction (EBSD) based on Zeiss EVO-50 scanning electron microscope completed with Oxford Instruments detector of reflected electrons. Diffraction patterns were obtained at an accelerating voltage of electron source 20 keV and probe current 1 nA. The specimen was tilted to the initial electron beam at an angle of 70°. To index diffraction lines, integration was used by three diffraction patterns of Kikuchi lines. The data of grain orientation were processed using HKL Channel 5 software package.

Results obtained in the study of the grain boundary structure

Figure 2 shows microstructure of hot-rolled and hot-extruded boiler tubes made of steel 20. Some special boundaries are marked with arrows. With an application of linear-intercept method, the following quantitative data of SB in the structure of the tubes under the study were obtained: 4.5-12.5% in hot-rolled and 9.2-14.5% in hot-extruded tubes.

Smaller number of special boundaries in the structure of hot-rolled tubes can be explained by the fact that in the hot-rolling process realized by tension-compression scheme in several passes, degree of reduction in pass is comparatively low and dynamical recovery takes place in the deformation zone (between the rolls) partially or completely recovering the grain shape. As static recrystallization of austenite does not occur between passes at the initial rolling stage and reduction accumulates, dynamic recrystallization in the deformation zone becomes probable beginning from a definite total reduction value despite a comparatively small degree of reduction in pass [11, 13].

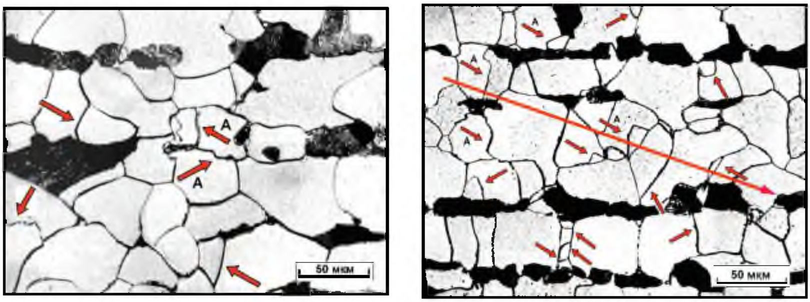
At a comparatively low deformation rate during hot rolling, alternation of declines (recrystallization softening) and rises (strain hardening) in flow stress occurs. Because these processes at different tube locations do not always coincide in time, a characteristic feature of the hot-rolled metal structure reveals itself in substructure heterogeneity, irregularity, serration of grain boundaries and appearance of colonies of new grains at the boundaries of initial ones. As a result, formation of special boundaries is hampered at a subsequent $\gamma \rightarrow \alpha$ transformation and their number in the final structure of hot-rolled tubes is small. They are indicated by arrows in Fig.1*a*.

All-sided compression of metal in hot extrusion ensures not only high plasticity of the worked material but makes it possible to realize large deformations in a working cycle. Due to a high degree and rate of strain in pressing carbon steels, dynamical recovery or even dynamical recrystallization occurs in the deformation zone and static recrystallization takes place after exit from the press. As a result, comparatively fine (No. 6-8) ferrite grains form in a subsequent austenite disintegration and hot-extruded tubes feature cross-sectional and longitudinal homogeneity of structure and mechanical properties [12, 13].

Study of grain-boundary structure in the ferrite component of extruded tubes has shown that special boundaries form between grains (Fig. 1*b*) and their share is considerably greater than that in hot-rolled tubes of analogous sizes (Fig. 1*a*).

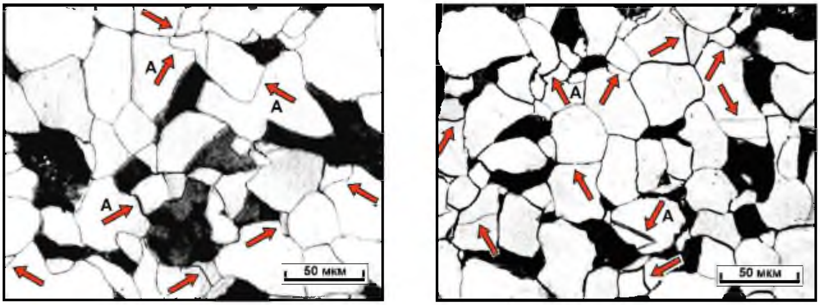
Increase in elongation from $\mu = 7$ to $\mu = 16$ during extrusion results in a growth of the share of special boundaries from 10.2% (Fig. 2*a*) to 16.5% (Fig. 2*b*) in the structure of hot-extruded tubes.

For the first time, the same specimens of hot-rolled and hot-extruded tubes were examined by scanning electron microscopy with analysis of electron backscatter diffraction (EBSD) patterns [14].



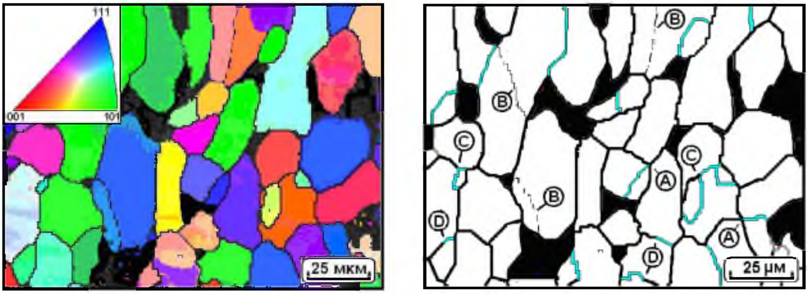
а *б*

Fig. 1. Microstructure in steel 20 tubes produced by two technologies: hot rolling *(a)*; hot extrusion *(b)*.



a *b*

Fig. 2. Microstructure in 219 mm diameter tubes extruded at different reductions: $\mu = 7$ *(a)*; $\mu = 16$ *(b)*.



a *b*

Fig. 3. Distribution of SB and pearlite colonies in steel 20 obtained by EBSD method: grain orientation map *(a)*; GB distribution scheme *(b)*.

An example result of such investigation is shown in Fig. 3*a* as a map of orientation images and Fig. 3*b* shows distribution of high-angle boundaries of a general type (black lines), pearlite colonies (black grains) and positions of special boundaries (double lines) with various indexes of reciprocal density of coincident sites Σ.

Special elements of a grain-boundary network, i.e. quadruple junctions (*A*) containing four high-angle boundaries of which one or more are special boundaries are also shown in Fig. 3*b*. Such configurations are stable and their presence is pinning surrounding boundary ensembles and restrains grain growth at high temperatures [6, 12].

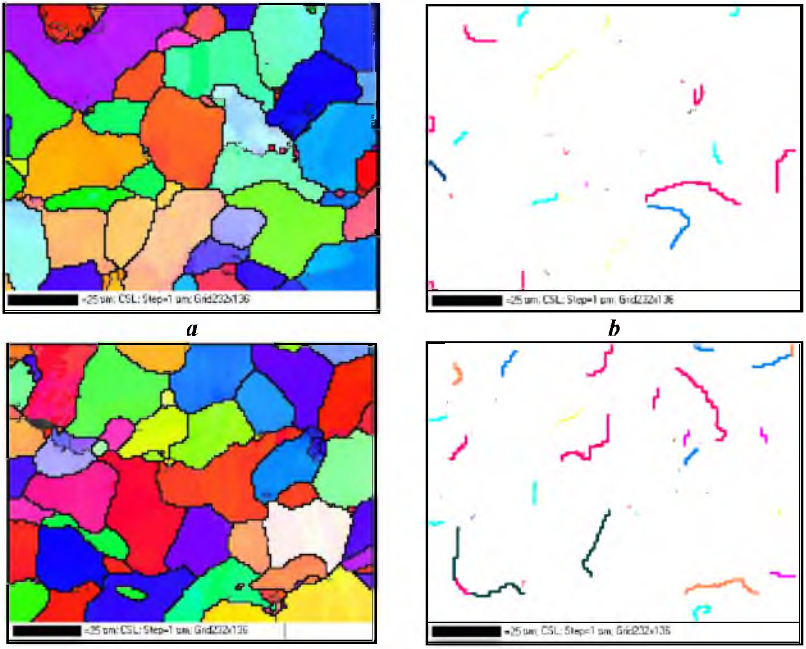
Some possible errors in functioning of the program processing the diffraction data obtained by EBSD method should be pointed out. For example, some low-angle boundaries (marked with symbols ***B***) are depicted with dotted lines which brings about certain ambiguity in the pattern of analysis. Symbols ***C*** in Fig. 3*b* indicate triple junctions in which two special boundaries and one random boundary of general type interact. Such combination contradicts the theoretical notion of interaction of three crystals along the junction. It is obvious that if two special boundaries Σm and Σn are contacting in a junction, than based on the matrix equations it can be proved that a special boundary with index $\Sigma(m\times n^{\pm 1})$ should be the third boundary in this junction. Note also the borderline cases (symbol ***D*** in Fig. 3*b*) when the program considers the same boundary a special boundary at one location and a general-type boundary at other location. This artefact results from an allowable range of special angle deviations from Brendon’s theoretical nominals [15]. Similar cases of doubt should be additionally assessed in an analytical way with an account for morphological characters to enhance validity of the obtained information.

Advantage of EBSD method is high speed of processing of electron diffraction patterns which enables analysis and assessment of large crystal arrays in a short time. However accuracy of determination of angles and axes of disalignment is not high at all times which is connected with both capabilities of EBSD analysis and sensitivity of this method to the quality of the specimen surface and software setting parameters [14].

An example of processing results obtained by EBSD method of studying hot-rolled and hot-extruded boiler tubes of steel 20 as a map of orientation images is shown in Figures 4*a, c* and patterns of distribution of special boundaries with various indices of reciprocal density of coincident sites Σ are shown in Fig. 4*b, d*.

Average specific number of special boundaries in determination by EBSD method was 12.5% in hot-rolled tubes and 23.5% in hot extruded tubes made of steel 20 at a high degree of deformation (elongation μ=16).

Comparison shows that the results of quantitative assessment of SB’s by metallographic and electron microscopy methods differ somewhat, however the determined fraction of special low-energy boundaries in extruded tubes was well larger in both cases (nearly twice as large) than in rolled tubes. Exactly this difference in the grain-boundary structure explains much higher level of long-term strength of hot-extruded boiler tubes as compared with hot-rolled ones [11, 13] that promotes enhancement of their reliability in operation at power generating units of thermal stations.



a *b*

c *d*

Fig. 4. Distribution and quantitative assessment of SBs by EBSD method in hot-rolled tubes *(a, b)* and in hot-extruded tubes *(c, d)* made of steel 20.

Resume

1. In accordance with the concept of coincident node lattices, special low-energy boundaries $\Sigma = 3$ were revealed in the grain-boundary structure of ferrite component of boiler tubes made of steel 20.
2. Temperature and deformation parameters of the production process have an influence on grain boundary structure in metal of boiler tubes made of steel 20: the number of special boundaries in hot-extruded tubes is twice as much than in hot-rolled tubes (23.5% against 12.5%).

References

1. Hall E.O. The deformation and aging of mild steel: iii. discussion of results. Proceedings of the Physical Society of London, – 1951. V. B64, – P. 747–753.
2. Petch N.J. The cleavage strength of polycrystals. J. Iron and Steel Institute. – 1953. – P. 25–28.
3. Watanabe T. An approach to grain-boundary design for strong and ductile polycrystals // Res. Mechanica – 1984. V. 11. – P 47-84.
4. Косевич В.М., Иевлев В.М., Палатник Л.С., Федоренко А.И. Структура межкристаллитных и межфазных границ. М.: Металлургия, 1980, – 256 с.
5. Сухомлин Г.Д. Взаимодействие границ отжига в ГЦК поликристаллах. // Физика металлов и металловедение. – 1982. Т.54, вып. 1. – С. 192-194.
6. Kopezky Ch.V., Andreeva A.V., Sukhomlin G.D. Multiple twinning and specific properties of $\Sigma=3^n$ boundaries in FCC crystals // Acta Metallurgica et materialia. – 1991. – V. 39, №7. – P. 1603-1615.
7. Randle V. 'Special' boundaries and grain boundary plane engineering // Scripta Materialia. – 2006. – V. 54, – P. 1011-1015.
8. Thaveeprungsriporn V., Was G., The role of coincidence-site-lattice boundaries in creep of Ni-16Cr-9-Fe at 36°C // Metall. Mater. Trans. – 1997, V. A, 28 –P. 2101-2112.
9. Bolshakov V.I., Sukhomlin G.D., Laukhin D.V., Beketov A.V., Dergach T.A., Kuksenko V.I. Special grains and multiple junctions in pre-eutectoid ferrite of low-carbon steels. // In: Collected Papers: Theory of Construction. Warsaw. – 2007. – pp. 72-79.
10. Сухомлин Г.Д. Специальные границы в феррите низкоуглеродистых сталей // Металлофизика и новейшие технологии. –2013. Т. 35, № 9 – С.1237-1249.
11. Опрышко Л.В. Структура и эксплуатационные свойства котельных горячепрессованных труб из непрерывнолитой заготовки // Теория и практика металлургии. – 2010. – №5-6. – С.124-126.
12. Горелик С.С. Рекристаллизация металлов и сплавов. М.: Металлургия, – 1978. – 567 с.
13. Опрышко Л.В., Головняк Т.В., Ветлянская А.Д. Жаропрочность углеродистых котельных горячепрессованных труб из непрерывнолитой заготовки // Металлургическая и горнорудная промышленность. – 2011.– №5.– С.49-51.
14. Schwartz A.J., Kumar M., Adams B.L., Field D. (Ed.). Electron Backscatter Diffraction in Materials Science: Springer. – 2010. 425 p.
15. Brendon D.G. The structure of high-angle grain boundaries // Acta Metall. – 1966, – V. 14, – P. 1479-1484.