FORMATION OF NON-UNIFORM GRAIN STRUCTURE OF STEEL IN THE PROCESS OF HEAT TREATMENT AND METHOD OF EVALUATION OF MICROSTRUCTURE WITH SIGNIFICANTLY NON-UNIFORM GRAIN

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ABSTRACT

This paper presents a study on the issues regarding the steel microstructure with significantly non-uniform grain, which is obtained as a result of different heat treatment. A method of evaluation of steel non-uniform grain microstructure is proposed. Examples of the method implementation are presented.

Keywords: microstructure, inherently fine grain steel, inherently coarse grain steel, non-uniform grain, recrystallization, cross section area.

INTRODUCTION

The microstructure is one of the main parameters of steel and it determines to a large extent the complex of its physical and chemical, mechanical and operational characteristics. Today there are a lot of microstructure evaluation methods developed, but 'direct' examination of steel grain structure with determination of grain size, shape and pattern is still up-to-date and the most popular research method.

For carbon and structural steel (inherently fine grain steel) with insignificant non-uniform grain a direct evaluation of grain size is applied. Mainly the average size dav is considered. As optional values maximum dmax and minimum dmin grain sizes can be taken into consideration.

When inherently coarse grain steel microstructure is analyzed, minimum, maximum and average grain sizes are considered. Also non-uniform grain ratio Knug which is defined as ratio dmax / dav is analyzed This value is of particular interest because for some steel the non-uniform grain structure has a critical meaning, in particular, for the steel with specific physical properties (electrical steel).

When the influence of new treatment modes, which are different from conventional modes or conventional heat treatment processes and are used for certain types of steel, is studied and also when recrystallization kinetics is analyzed, i.e. fast change of structural parameters in the treatment process, the method of design of the curve of test sample surface areas occupied by grains of various size can be used. This method is most demonstrative for secondary recrystallization and also for collective recrystallization, development of which happens very fast within short time periods, i.e. for cases of non-uniform grain occurrence.

EXPERIMENTAL

Occurrence of such conditions is typical when inherently fine grain steel is severely overheated or when inherently coarse grain steel is heat treated with high heating speeds. The mechanism of non-uniform grain occurrence in both cases is the same (Fig. 1).

When inherently fine grain steel is overheated at temperature higher than certain level, reported to the system free energy overflow reaching certain limit tend to be compensated by means of grain boundary energy reduction, i.e. by reduction of grain boundary length via growth of some grains and absorption of the others. In this case temperature limit of recrystallization development is determined by grain boundaries stability and depends on inclusion morphology, size and shape, stability of existing grain crystalline pattern imperfections (dislocation walls, compound crystals boundaries, Cottrell clouds, foreign atoms), initial grain size, etc.

For coarse grain steel the parameter of heating temperature limit excess (overheating) is not a condition when significantly non-uniform grain occurs. In this steel under standard process treatment collective recrystallization with coarse grain formation takes place anyway. In that case the condition of abnormal growth of some grains along with temperature rise is defined

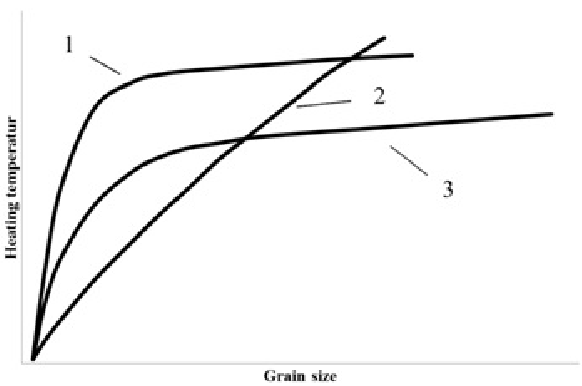

Fig. 1. Recrystallization kinetics. (1 - inherently fine grain steel, 2 - inherently coarse grain steel, 3 - inherently coarse grain steel under fast heating).

by parameters of the current heat treatment rather than chemical composition. One of these parameters is fast heating when recrystallization annealing is performed. Fast heating is here understood as heating with the speed of 100^{o}C per second and higher. This value is much higher than the one during strip heating when the strip is inside the heated furnace (heating is performed by convection and radiation). In that case significant free energy overflow received within short time is not compensated by grain boundary energy reduction, which happens as a consequence of grain growth during collective recrystallization. Thus condition No.1 is realized and abnormal growth of some grains occurs - significantly non-uniform grain structure appears. However, in some cases secondary recrystallization is not the thing to be considered, because the mechanism of selective growth of grains with specific crystallographic orientation is not implemented. Here dynamic collective recrystallization can be considered and in this context reaching of high temperatures similar to temperatures for fine grain steel is not required: the process takes place with lower temperatures. Nevertheless despite all differences the process of microstructure development in both cases (for fine grain and coarse grain steel) can be considered as identical, because the basis is the system effort to fast reduction of free energy overflow received in heating process.

When the steel with significantly non-uniform grain is evaluated, not only standard microstructure parameters as d_{min}, d_{max} и d_{av} should be used. They are not going to provide detailed understanding of the process stage and its influence on formation of the final product end-use properties. It is particularly typical for uncompleted treatment process. As an example receipt of final grain size in isotropic (non-grain oriented) electrical steel can be considered. According to modern ideas regarding grain size influence on specific magnetic losses value - the bigger grain is the lower losses are. At that parameters dmax и Knug do not show what quantity of coarse grains fit into a unit of cross section area and thus what is their contribution in the end-use properties formation.

In GOST 5639-82 the methods of average grain area determination is specified. For non-uniform grain structure, which according to GOST is defined as a structure containing grains, which are different from the main (prevailing) size corresponding to a certain scale gauge more than one size and occupying an area more

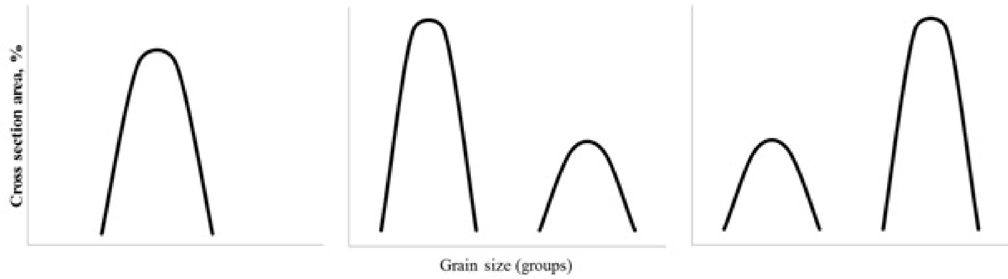

Fig. 2. Distribution of cross section surface area along the grains of different size during recrystallization (as per S.S. Gorelik). a) - collective recrystallization, b and c - secondary recrystallization.

than 10 % of the section, the evaluation of grain size by the method of chord length measurement is suggested. For each size group the following is determined: actual sizes, length of measured chords, quantity of chords for this size, total length of chords as per size and a size group and relative portion of chord length occupied by a size group.

In that case it is reasonable to add into analysis such a parameter as the sample cross section area occupied by grains of different size. As it is further shown, the method of measuring cross section area portions occupied by grains of different size is an efficient tool of study and analysis of structures with apparent non-uniform grain.

As a grain size both grain score and its value in absolute terms (μm, mm, m^{-3}, m^{-6}, nm, Å, etc.) can be taken. For convenience of the analysis of steel microstructure with significantly non-uniform grain size groups with certain multiple range can be used. For example, from 0 to 10 μm; from 10 to 20 μm, etc.

The methods of determination of grain area in cross section involves the following:

1. For each sample several microphotographs are taken under zooming sufficient for separate smallest grains detection and their size measurement.

2. Several areas with the amount of grains within the highlighted area (preferably, not less than 40) are chosen randomly on the microphotographs.

3. The number of i-size grains of a size group, which are inside a delineated figure, is calculated (ni).

4. Nominal area of the surface occupied by i-size grains is calculated.

$$S_{i\ nom}=N_i\ \pi\ (d_i/2)^2 \quad (1)$$

where di is the diameter of i-size grain (grain size of i-size group).

5. Percentage of cross section area occupied by i-size grains (size group) is calculated

$$S_i= S_{i\ \text{НОМ}}/\sum S_{i\ \text{НОМ}} \times 100\% \quad (2)$$

In order to minimize the measurement error it is recommended to perform several measurements for each sample with total amount of grains not less than 500. The final result can be defined as arithmetic average.

This method is used as demonstrative for evaluation of recrystallization passage mechanism. It is indicative that collective recrystallization diagram is a curve with one maximum while secondary recrystallization diagram - with two (Fig. 2). However, this information can be used not only as demonstrative but also while performing quantitative microstructure analysis.

According to the mentioned methods, the dynamics of recrystallization passage in cold rolled isotropic electrical steel of different alloying groups was evaluated under fast heating up to different temperatures. Heating speed amounted to more than 230^{o}C/s. Chemical composition of the steel is shown in Table 1.

At least 10 measurements from steel samples were taken for each heating temperature in all studied alloying groups. Measurement error was less than 5 %. Results are shown in Table 2.

Based on the results of the received data analysis fitted curves characterizing kinetics of recrystallization in the above conditions were constructed (Fig. 3).

RESULTS AND DISCUSSION

The analysis of the received data allows to determine intervals of recrystallization passage. For example, for the steel of all alloying groups (except for group 4) in a certain temperature interval cross section area distribution along the grains of certain size looks like a curve

Table 1. Chemical composition of the steel of studied heats, mass %.

Alloying group	C	Si	Mn	S	P	Al	Cr	Ni	Cu	Ti	N
0	0.034	0.081	0.17	0.007	0.26	0.055	0.03	0.01	0.02	0	0.004
1	0.029	0.68	0.18	0.007	0.13	0.231	0.04	0.02	0.05	0.008	0.005
2	0.035	1.272	0.19	0.006	0.017	0.372	0.05	0.03	0.06	0.004	0.007
2 with phosphorus	0.033	1.15	0.22	0.005	0.188	0.299	0.05	0.02	0.05	0.014	0.005
3	0.036	2.21	0.19	0.006	0.076	0.381	0.05	0.02	0.05	0.008	0.005
4	0.006	3.055	0.21	0.005	0.011	0.427	0.02	0.01	0.03	0.003	0.003

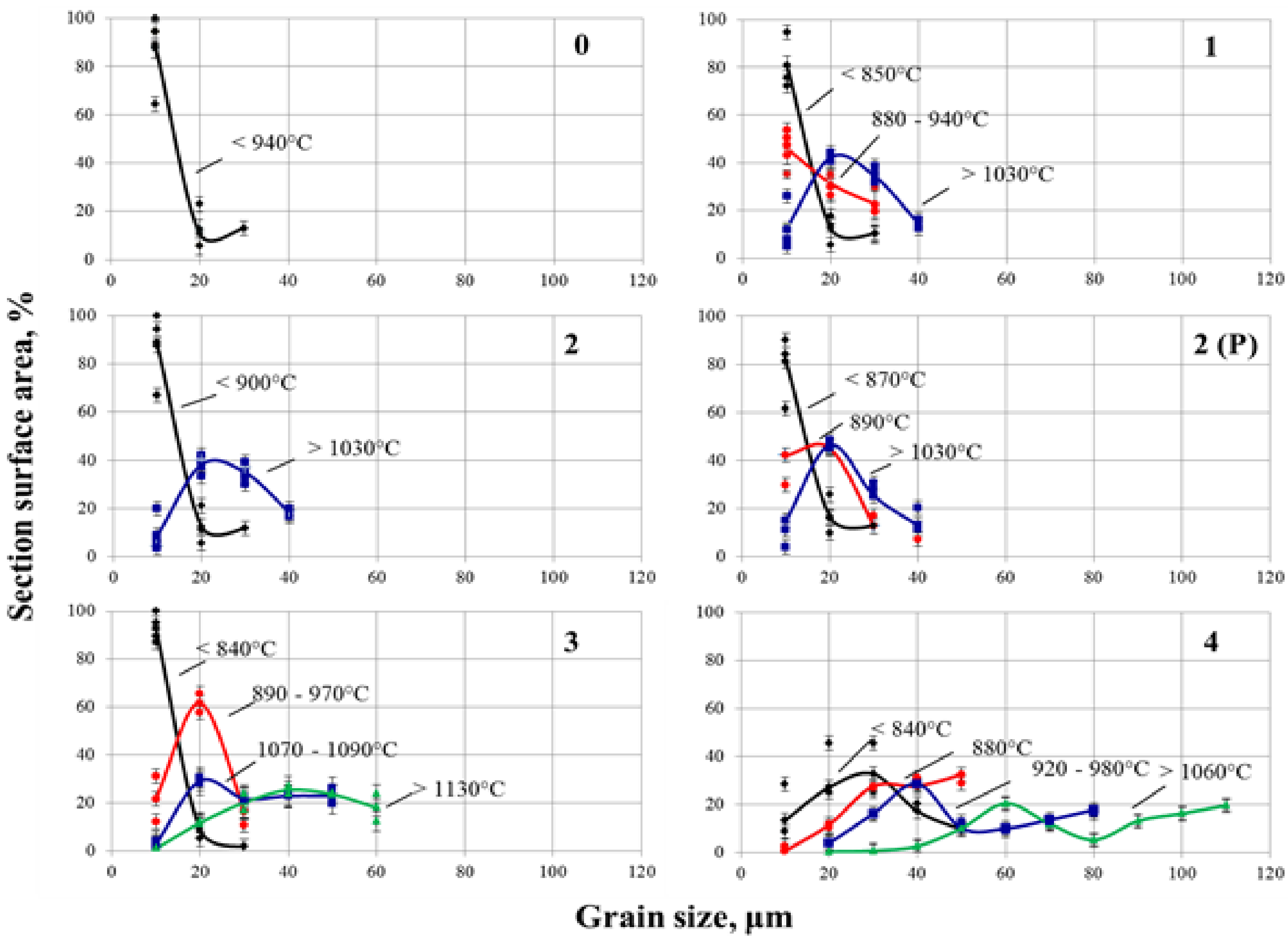

Fig. 3. Fitted curves of cross section surface area distribution along the grains of different size.

with a down extreme point. At that the left part characterizing section area occupied by fine grain is more significant than the right one. Such curve is typical for the beginning of collective recrystallization.

When heating temperature is rising, the curve appearance changes and looks more standard with a top

Table 2. Change of grain cross section area depending on heating temperature.

Alloying group	Heating temperature, °C	Cross section area (%) as per grain size (μm)										
		10	20	30	40	50	60	70	80	90	100	110
0	720	100										
	740	94.3	5.7									
	780	94.3	5.7									
	810	94.3	5.7									
	860	87.5	12.5									
	900	87.5	12.5									
	940	64.3	22.9	12.9								
1	740	94.6	5.4									
	750	75.6	13.9	10.5								
	760	72.2	17.8	10								
	770	53.8	26.4	19.8								
	780	50.5	30.1	19.4								
	850	47.5	30	22.5								
	880	43.2	34.6	22.2								
	900	35.2	35.2	29.7								
	940	25.9	42.4	31.8								
	1030	11.8	44	31.6	12.6							
	1060	7.9	42.5	35.4	14.2							
	1150	4.9	40.5	38.4	16.2							
2	740	100										
	760	94.6	5.4									
	770	88.9	11.1									
	900	67.1	21.1	11.8								
	1030	20	33.3	30	16.7							
	1070	6.9	42	33.3	17.8							
	1080	4.1	40	37	18.8							
	1150	3.7	37.3	39.1	19.9							
2 with phosphorus	720	90.1	9.9									
	770	84	16									
	780	84	16									
	790	84	16									
	800	84	16									
	870	61.5	25.8	12.7								
	890	42.3	45.1	12.7								
	980	29.9	45.7	17.1	7.2							
	1010	11.2	48.4	28.7	11.7							
	1060	4.1	45.2	30.4	20.3							
3	740	100										
	750	95	5									
	800	89.2	10.8									
	840	87.2	11	1.8								
	890	31.3	57.8	10.8								
	970	12.3	65.6	22.1								
	1070	4.2	30.5	22.4	23.1	19.8						
	1090	1.4	28.1	21.4	22.9	26.2						
	1130	1	12.2	23.5	27.1	23.9	12.3					
	1210	0.9	11.3	16.9	23.7	23.4	23.8					
4	710	28.6	45.7	25.7								
	800	8.9	25.3	46.5	20.3							
	840	2.6	10.4	26.9	31.2	28.9						
	880	0.6	11.6	27.6	27.7	32.5						
	920	—	4.1	16.2	28.7	12.8	9.2	12.6	16.4			
	980	—	3.6	16.1	28.6	7.4	10.7	14.6	19			
	1060	—	0.3	0.7	2.6	10.1	20.4	11.9	5.2	13.1	16.2	19.6

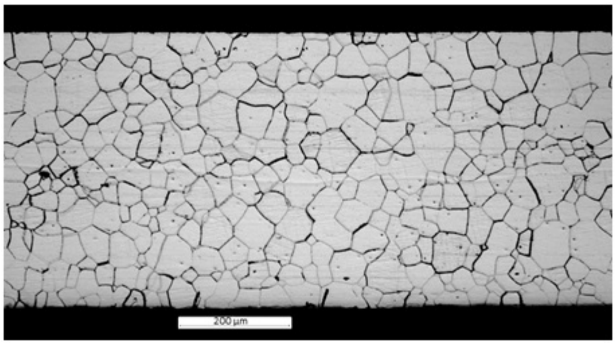

Fig. 4. Microstructure of the steel of alloying group No.4 under heating up to 1060°C.

extreme point and places going down. Such curve is typical for the intensive development of collective recrystallization (Fig. 3). In this regard it is possible to estimate maximum and minimum grain sizes and contribution of this or that size group into end-use properties.

For electrical steel with high content of alloying elements when heating temperature rises in these conditions, first, curve flattening takes place and then a tendency of the second maximum occurrence appears. However, microstructure analysis did not reveal any insular non-uniform grain structure typical for secondary recrystallization (Fig. 4). That is why in this particular case relating to the above mentioned heating conditions, we can speak about dynamic development of collective recrystallization. In this circumstances based on the curve rate (Fig. 3), it may be said that the main contribution into steel properties formation will be provided by 50 - 70 µm and 90 - 110 µm grains. The results received can be used for selection and adjustment of further heat treatment mode.

CONCLUSIONS

Determination of the cross section occupied by grains of different size is an efficient method of studying the steel microstructure with significantly non-uniform grain. This method supports conventional methods of microstructure evaluation, gives the possibility to evaluate kinetics of recrystallization passage, to predict end-use properties of the final product and also it can be used during elaboration of heat treatment modes for better consideration of factors influencing steel microstructure development during annealing.

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