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Microstructural Quantification for Pipeline Steel Structure-Property Relationships

Standards currently used to assess the quality of steel plate for pipelines are out of date and cannot be used for this purpose. The motorized hardware-software complex "Thixomet SmartDrive" has been developed and installed in dozens of enterprises and companies to provide an objective quantitative estimation of all types of structural inhomogeneity in modern pipeline steels, such as microstructural banding, general anisotropy, blocks of bainite with lath morphology and centerline segregation.

All developed standards are either unique and have no analogues (general anisotropy and blocks of bainite with lath morphology), or are compatible but exceed in efficiency and objectiveness of quality estimation: current Russian (GOST 5640-68) and foreign (ASTM 1268, GB/T 13298) standards. Our work has demonstrated that structural heterogeneity evaluated by the volume fraction and size of elongated areas of bainite blocks with lath morphology parallel to the rolling direction will adequately describe mechanical properties obtained from tensile and impact tests of pipeline steels with yield strength from 485 up to 625 N/mm².

The "structure – properties" relationships determined can be used as a background for developing a quality estimation system that includes acceptance tests for steel products between suppliers and consumers of pipeline plate, and also for the improvement of production technology.

Keywords: pipeline steels; microstructure; mechanical properties; structural inhomogeneity; quality control of steel.

At the present time, the mechanical properties are the main parameters for conducting acceptance tests of plate for pipeline production. Tensile strength, impact toughness and drop weight tear tests determined at both room temperature and lower temperatures can be mentioned among these parameters. At the same time, the structure is taken into account optionally, by grain size according to the GOST 5369 and by banding according to the GOST 5640. Metallurgical quality is estimated by content of non-metallic inclusions according to the GOST 1778-70. It should be noted that all of these standards were developed in the 1960s and 1970s and therefore reflect the state of pipeline making technology of that period. However, at the present stage of production technology for pipeline steels neither the degree of banding nor content of non-metallic inclusions can be considered as a limiting factor determining their usability. Banding is now relevant only for low-strength ferrite-pearlite steels. Implementation of accelerated cooling after finish rolling results in formation of ferrite-bainite or pure bainite structures, rather than the former ferrite-pearlite banded

structure: therefore structural banding is practically absent in such steels, while the structural dispersion is characterized not only by the ferrite grain size (which cannot be practically measured in ferrite-bainite steels), but by the dispersion and morphology of the structural constituents while taking all components of this structure into account. Effective ladle treatment decreases the content of inclusions below the second grade (according to the GOST 1778-70), or down to parts per thousand in vol. % (according to the ASTM E 1245). Such a low content of non-metallic inclusions has no negative effect on steel properties. On the contrary, dispersed and uniformly distributed non-metallic inclusions form the base of so-called "oxide metallurgy" developed by Nippon Steel [1]. Acicular ferrite, providing the best combination of steel strength and toughness, nucleates at such inclusions as on substrates. Therefore, the set of structural characteristics used at present time for acceptance tests for pipeline steels is now out of date and cannot be applied to define the usability of plate for this application.

Moreover, mechanical properties determined, even with full-thickness specimens, cannot fully characterize steel usability. The results of these tests depends on the location of sampling; and, therefore, on features of plate structure that are inherited from a continuous cast slab, taking into account the influence of the technology of controlled rolling. As soon as up-to-date technology cannot provide an isotropic structure, equal along all three directions, the deviation of values in mechanical tests can be substantial. This means that the results of acceptance tests cannot be used for generalization of experience and establishment of significant "structure - property" relationships because it is not clear what microstructure can be identified with these properties, because the properties are determined on one kind of specimens and the structure on another. We can testify that only the microstructure located directly under the fracture surface and quantified along a representative square in this area can be used for defining "structure – property" relationships.

It is well-known, that a steel's structure defines its properties. Therefore, if we quantify the structure correctly using up-to-date methods of quantitative metallography, then the structure (unlike mechanical properties) can characterize plate usability more objectively. A large "structure – property" database is certainly required for such structural predictions.

The question about the expediency of using structure as the final defining parameter for estimating plate usability is no longer a theme for discussion. It is known [2] that a new heavy plate mill has been put into practice at the Xiangtan Iron and Steel works. This mill can produce approximately 2 million tons of heavy plates with width up to 4.8 m. In addition to the

conventional dimension range, the two-stand rolling mill can manufacture high-strength plates of X80 grade. Siemens has designed and supplied all main mill components, including roughing and finishing stands with control and measuring devices and automation systems. This mill is the first one to be equipped with a so-called “monitor of microstructure” system, initially developed by Siemens for hot rolling mills. This system to monitor microstructure predicts mechanical properties of the plate metal and even will cancel corresponding tests. Unfortunately, this publication [2] doesn't contain the details of such a control system.

Based on the above-mentioned, we believe that the following problems solved in this work are:

1. Techniques are developed for quantitative description of all kinds of structural inhomogeneity in modern pipeline steels;
2. “Structure – property” relationships for most of the mechanical properties included in the range of acceptance tests are revealed for steels with different strength categories.

A. Development of the technique for evaluation of microstructural banding

Most Russian microstructural standards employ visual comparison methods using standard charts for evaluation of the microstructure of steels and alloys. Analogue foreign standards often contain, along with a qualitative description of the structure (i.e., using standard charts), a quantitative description of microstructure parameters, obtained via stereological measurements. The standards GOST 5640-68 and ASTM E 1268 can be considered as the examples of such standards. It should be noted that at the present time Russia lags behind leading countries in usage of quantitative metallography for the description of the microstructure of materials. This situation occurred in spite of the fact that stereology as a science has been fully developed for the first time in the world by S. A. Saltykov more than 50 years ago [3]. The necessity for the fastest national implementation of quantitative metallography methods can be substantiated by at least two reasons. Firstly, evaluation of structure quality based upon stereological measurements is certainly more objective and exact in comparison with the visual method using standard charts. Secondly, at the present time, during integration of Russia into the global economics, it is necessary to overcome the problems of compatibility between national and foreign standards for the cases of mutual supply of metal products and conducting acceptance tests.

The aim of this work is the development of the technique of quantitative evaluation of microstructural banding for low-alloyed pipeline steels (meeting the requirements of GOST 5640-68) using automatic image analysis.

The technique is intended for evaluation of microstructural banding in plates and coiled sheet of pipeline steels of ferrite-bainite class and steels with three, or more, structural constituents using corresponding charts. This chart is built based on the principle of increasing the number of bands of

the second phase, taking into account their continuity and degree of ferrite grain elongation. The assigned grade is based on stereological parameters that have been founded using a directed secants method with the assistance of an automatic image analyzer [3,4].

To simplify the description of the procedure of image analysis, we shall introduce the term “second phase”, meaning bainite itself and other products of austenite decomposition that are different in appearance compared to the ferrite matrix.

Banding describes the segregation of structural constituents (ferrite and second phase) in rolled steel, more exactly, - the degree to which these structural constituents separate into distinctly expressed layers that have been formed via stretching of micro-segregation areas along the rolling direction. Forming of a banded structure is affected by the metallurgical inheritance of conditions in the slab, namely segregation of impurities across the slab cross section, as well as such technological parameters as the hot deformation final temperature, reduction ratio during hot deformation, etc.

Microstructural banding is caused by dendritic segregation and depends on steel composition (especially on carbon), on solidification rate and on the procedure for the subsequent hot deformation of the steel. Segregation, as a base for microstructural banding, is restricted by size of the dendritic cell. Therefore, it is characterized by a rather uniform distribution inside the plate volume and can be evaluated by stereological methods. On the contrary, segregation bands which are formed as a result of zonal segregation, are inherited from axial chemical slab inhomogeneity and are located at the center of the plate, therefore they cannot be evaluated by stereological methods.

It should be noted that comparisons between standard chart images and those of the specimen are conducted at 100 X magnification, according to GOST 5640-68, while analysis of elongated ferrite grains requires 500X magnification. Such an analysis is possible only for subsequent examination of a sample at two magnifications. In classic metallography, it is impossible to observe the same wide field of view provided by 100 X magnification at 500X. Therefore, classic metallographic investigations are always a compromise between examining area and resolution. But, if we apply modern methods of quantitative metallography using the Thixomet® image analyzer, we can simultaneously evaluate both banding and ferrite grains elongation making measurements only at 500X magnification, while the required area corresponding to the size of the field viewed at 100X can be subsequently “stitched” together electronically from adjacent fields of view. When a motorized microscope stage is moving to the next field of view, the previous field is stitched precisely, “pixel to pixel” to the field that was captured just before. This way a high resolution “panoramic image” of any desired large area can be created.

Calculation of these parameters is based on the method of directed secants:

$$1. \frac{\bar{N}_{L\perp}}{\bar{N}_{L\parallel}}$$

where $\bar{N}_{L\parallel}$ is average number of particles of the second phase, crossed by secants that are parallel to the rolling direction, on

the unit of secant length; $\bar{N}_{L\perp}$ is average number of particles of the second phase, crossed by secants that are perpendicular to the rolling direction, on the unit of secant length.

$$2. \frac{\sigma_{NL\parallel}}{\sigma_{NL\perp}},$$

where $\sigma_{NL\parallel}$ is standard deviation of number of particles of the second phase, crossed by secants that are parallel to the rolling direction; $\sigma_{NL\perp}$ is standard deviation of number of particles of the second phase, crossed by secants that are perpendicular to the rolling direction.

$$3. \frac{\sigma_{VL\parallel}}{\sigma_{VL\perp}},$$

where $\sigma_{VL\parallel}$ is standard deviation of the fraction of second phase on secants that are parallel to the rolling direction; $\sigma_{VL\perp}$ is standard deviation of the fraction of second phase on secants that are perpendicular to the rolling direction.

Let's analyze workability of these parameters for three structures, two of which (Fig. 1, a and b) were taken from the book by S. A. Saltykov [3] and the third structure (Fig. 1,c) was synthesized from the first one (Fig. 1,a). The synthesized structure was obtained by redistribution of the second phase in the ferrite matrix in such a way, that the banding of the initial structure is decreased significantly, while the anisotropy of the separate particles of the second phase remained the same.

The $\bar{N}_{L\perp} / \bar{N}_{L\parallel}$ ratio defined as the anisotropy ratio in ASTM standard E 1268 only describes the elongation of individual microstructural constituents (ferrite or second phase) along the rolling direction and doesn't reflect either the structure orientation in general, or incorporation of single structural constituents into bands.

Orientation can be expressed by some general directivity of the structure along the rolling direction without its separation to distinct layers. But in order to describe general directivity, we need to use the $\sigma_{VL\parallel} / \sigma_{VL\perp}$ ratio that will be analyzed further.

Let's show that $\bar{N}_{L\perp} / \bar{N}_{L\parallel}$ ratio can be used for evaluation of the particles elongation of the second phase along the rolling direction and that it doesn't reflect the character of the relative position of the structural constituents; therefore, it cannot describe structural banding. For this purpose we shall compare the structures on the pictures 1,a and 1,c that are characterized by equal values: $\bar{N}_{L\perp} / \bar{N}_{L\parallel} = 3.30$. At the same

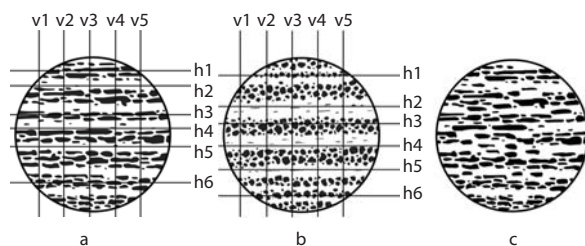


Fig. 1. Synthesized structures for analysis of stereological ratios for banding evaluation by the method of directed secants

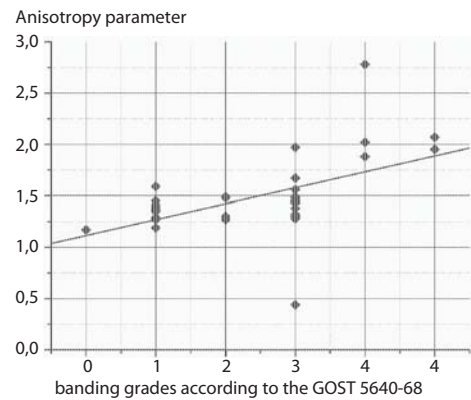


Fig. 2. Anisotropy parameter (see axis of ordinates) calculated for microstructure with different banding grades according to the standard GOST 5640-68 (see axis of abscissa)

time, they are absolutely different in their banding: the structure on the Fig. 1,c has a rather uniform distribution of the particles of the second phase, while the structure in the Fig. 1,a is characterized by substantial banding. On the contrary, the ratio for the structure shown on the Fig. 1,b is essentially lower than that shown on the Fig. 1,c (1.25 compared with 3.30), however, the structure on the Fig. 1,b has more distinct banding.

It was suggested that $\bar{N}_{L\perp} / \bar{N}_{L\parallel}$ ratio characterizes only the elongation of the second phase particles that is equal for the structures shown on the Fig. 1,a and 1,c and is essentially less for the structure from the Fig. 1,b (containing equiaxed second phase particles).

Indeed, as follows from ASTM E 1268: "The verbal description of the nature of the banding or alignment is qualitative and somewhat subjective". It is clear in connection with the above-presented analysis, that it's impossible to describe structure banding according to the standard GOST 5640-68, using for this purpose the $\bar{N}_{L\perp} / \bar{N}_{L\parallel}$ ratio, defined in ASTM E 1268 as the anisotropy ratio parameter (Fig. 2). "The values of the anisotropy index and the degree of orientation cannot be used to establish whether the microstructure is merely oriented parallel to the deformation direction or is actually banded. This difference requires pattern recognition techniques which are beyond the scope of this method". We have shown above that this statement of ASTM E 1268 is not true.

So, banding describes the character of the relative position of structural constituents (ferrite or second phase); or to be more precise, – the separation of all these structural constituents into distinct layers oriented along the rolling direction. Therefore, only criteria $\sigma_{NL\parallel} / \sigma_{NL\perp}$ and $\sigma_{VL\parallel} / \sigma_{VL\perp}$, characterizing deviations from mean value for the number of secant crosses and the fraction of the second phase in measurements along and across the rolling direction, respectively, can be considered as probable banding ratios.

Taking into account the analysis made in [4], it can be concluded that only the ratio $\sigma_{VL\parallel} / \sigma_{VL\perp}$, based on measurements of the fraction of the second phase on directed secants along and across the rolling direction, will unambiguously characterize structural banding without depending on the morphology of particles of the second phase. Indeed, this ratio

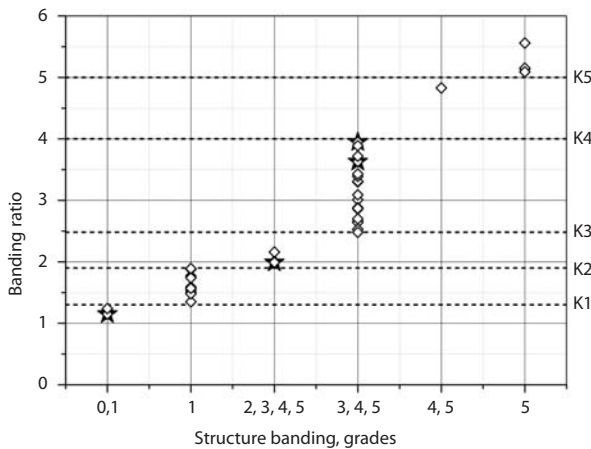


Fig. 3. The results of identification of real structures using banding ratio (see grades of structure banding on the axis of abscissa and banding ratio on the axis of ordinates)

decreases regularly from the 1st structure to the 3rd: 6.73; 4.32; 3.72 (Fig.1). If we check its use on photo reference standards from GOST 5640-68, we shall receive: $\sigma_{VL\parallel} / \sigma_{VL\perp} = 1.33; 1.62; 2.58; 2.82; 3.17$ and 4.25 respectively for the grades 0, 1, 2, 3, 4 and 5.

Therefore, the ratio $\sigma_{VL\parallel} / \sigma_{VL\perp}$ has been chosen reasonably as the only one from the other known ratios that adequately describes structural banding and does not depend on elongation features of single structural constituents along the rolling direction. Let's call it the "banding ratio". The technique for the calculation of anisotropy and banding ratios is based on fundamental stereological relations and realized with automatic image analyzers.

The results shown by image analysis for characterization of real microstructures using the above mentioned technique are shown in the Fig.3. After such a structure evaluation with the banding ratio, it is required to check the anisotropy of ferrite grain. Ultimate values of banding ratios, as well as anisotropy of ferrite grain and the second phase for determination of corresponding grades from zero to fifth are obtained using expert estimations method.

The developed technique is implemented with the Thixomet Image Analyzer and is used for evaluation of structural banding at many leading companies involved in pipeline production.

The total measuring procedure consists of the following steps:

- visual search for the area on a specimen with a maximum grade using the microscope at 100X magnification and then switch to the objective to produce the higher magnification;
- building a panoramic image of the sample area found during visual search;
- detection of the second phase and boundaries of ferrite grain;
- fine tuning of identification of boundaries of the second phase and ferrite grain;
- calculation of banding ratios and anisotropy ratios;
- definition of banding grade in accordance with the "decision diagram" [4].

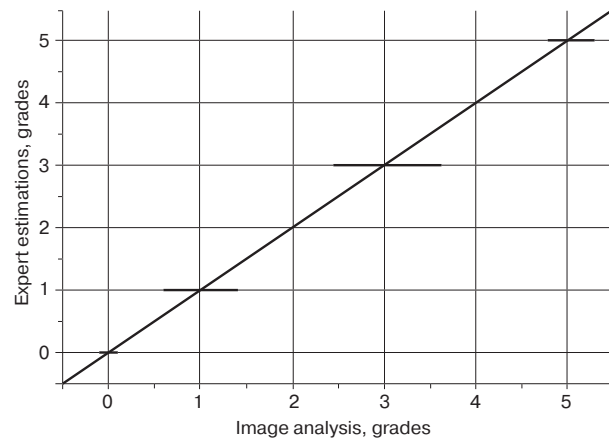


Fig. 4. The results of comparison of the values of banding grade, obtained using Thixomet Image Analyzer and expert estimations method (see image analysis in grades on the axis of abscissa and expert evaluation in grades on the axis of ordinates)

Afterwards, the 10X objective is used again, the microscope stage travels to the next area of a specimen with a maximum grade and the whole above-described cycle of measurements are repeated. As soon as the same values of the maximal grade are found in three areas of a specimen, characterized with maximal structure banding, conducting measurements will be finished and the banding grade of the investigated specimen, revealed in those three areas, will be established.

Search for the maximum grade which should be found at least three times on a specimen is conducted by the operator visually with 100X magnification. In order to identify correctly the second phase and boundaries of the ferrite grain, the measurements are made at magnifications 4-5 times higher than 100X, where the software provides building of the square or rectangular panorama with area not less than 0.5 mm² for one test cycle.

The results of comparison of the values of banding grade, obtained using expert estimations method and the Thixomet image analyzer, display their good convergence (Fig.4).

Therefore, it is shown that not one of the structural stereological parameters used in ASTM standard E 1268 and described by S. A. Saltykov, can correctly describe its microstructural banding, and that's why this standard describes banding only by the verbal description. In this paper, the technique of determination of microstructural banding in accordance with GOST 5640-68 standard has been developed and realized as Thixomet image analyzer plug-in. This technique operates using the grades of corresponding scale built on the principle of an increase of the number of bands of the second phase, taking into account the continuity and elongation degree of the ferrite phase.

The grade is defined on the basis of stereological parameters revealed by the method of directed secants in panoramic image investigations with the Thixomet image analyzer. Comparison of the results of the banding evaluation (executed with the aid of the image analyzer) with the results obtained by metallographic experts has shown their good

agreement. Usage of the image analyzer for the evaluation of the structural quality of low-alloyed pipe steels substantially increases the objectivity of grading of the microstructural banding.

B. Development of the method for evaluation of structure anisotropy

Modern pipeline steels are manufactured using thermomechanical treatment technology which includes accelerated cooling after finish rolling. In this way plate has an almost 100% bainitic structure, while microstructural banding is not present. However, anisotropy of the bainitic structure can be observed in such steels. The stereological methods that have been described above for the evaluation of ferrite-pearlite and ferrite-bainite microstructure are not applicable for the description of complicated bainitic morphologies. Thereby the method of evaluation of bainite anisotropy based on texture analysis of images has been developed for this case [5].

Texture analysis is conducted for the extraction of texture features that completely or partially characterize the examined image or its fragment. The task is to find such features that can unambiguously characterize microstructural anisotropy, i.e., the presence and intensity of a priority direction in the microstructure. This requires the building of a gray level co-occurrence matrix ($P_{d,\alpha}$); the constituents present a conditional probability $P_{d,\alpha}(i,j)$ of forming a pixel with gray level i at a distance d from the pixel with a gray level j . Features describing these matrices are calculated further. "Reciprocal" difference or homogeneity ($H_{d,\alpha}$) can be mentioned as one of these features [5]:

$$H_{d,\alpha} = \sum_i \sum_j \frac{P_{d,\alpha}(i,j)}{1+|i-j|} \quad (1)$$

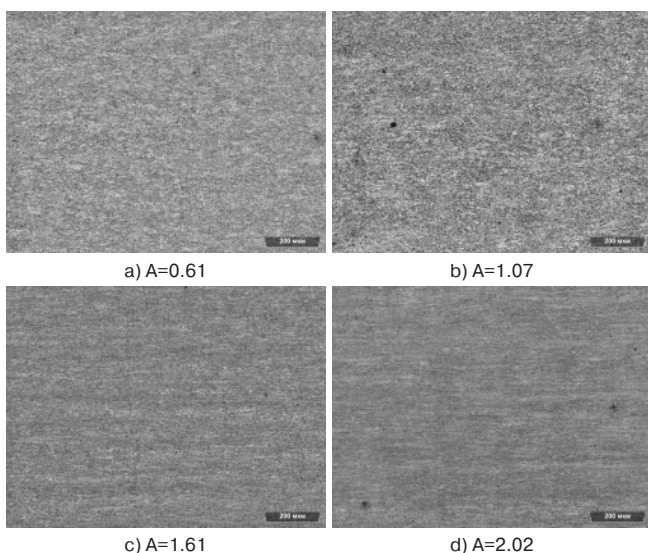


Fig. 5. Microstructure with different values of anisotropy ratio: a), b) – transversal direction; c), d) – longitudinal direction

This feature characterizes matrix scattering $P(i,j)$ relating to the main diagonal, that in its turn reflects the grain size or texture coarseness. Evidently, coarseness of texture for images of anisotropic structures is rather different in one direction, so homogeneity attributes differ respectively.

So, calculation of the anisotropy ratio (A) consists of the following operations: building of several matrices $P_{d,\alpha}$ for different directions α and calculations of homogeneity features on the base of each of them. The anisotropy ratio is worked out as standard deviation of $H_{d,\alpha}$ values calculated for different directions ($\sigma_{H_{d,\alpha}}$):

$$A = \sigma_{H_{d,\alpha}} \quad (2)$$

Using the methods of texture analysis, we have no need to select any constituent of microstructure therefore we can evaluate microstructure with a complicated morphology and increase evaluation objectivity. The ratio determined via this method characterizes unambiguously the general anisotropy of the microstructure of bainitic steels.

Images of microstructure with different values of anisotropy ratio are displayed on the Fig.5.

C. Development of evaluation technique of blocks of bainite with lath morphology

The results of the investigations revealed the blocks of bainite with lath morphology among different morphological forms elongated along the rolling direction. Exactly these bainitic blocks with lath morphology make the main contribution into general anisotropy and decrease essentially mechanical properties of the plate in transversal direction [6]. The technique of color etching with consequent analysis in polarized light [7] has been developed and covered by the RF patent; it allows to select definitely blocks of bainite with such a morphology and to measure their volumetric content and length of longitudinal inter-phase boundaries which determine mainly the mechanical properties for high strength pipeline steels. The above-mentioned technique includes the following steps:

1. Preparation of the polished section via the technique of sample preparation of the hot-rolled plate made of ferrite-bainite and bainite steels for quantitative metallographic investigations [8];

2. Color etching for obtaining optical effect under polarized light [7];

3. Image analysis of ferrite-bainite microstructure. Quantitative analysis of the structural constituents was carried out on motorized light microscope Axiovert 200 MAT, equipped with Thixomet Image Analyzer. The technique for automatic quantitative analysis of ferrite-bainite microstructure has been developed. Its idea can be expressed in such a way:

- 3.1. Building of panoramic image with required area at required magnification of the etched structure under polarized light (pictures 6,a and 6,b). Magnification from 100X to 500X are considered as the most objective; it should be taken

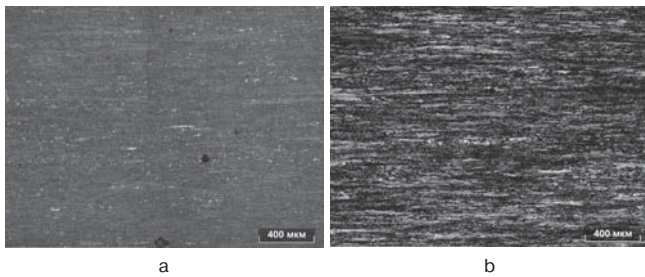


Fig. 6. Panoramic image of color etched structure in the bright field (a) and under polarized light (b). Original magnification: 100 X

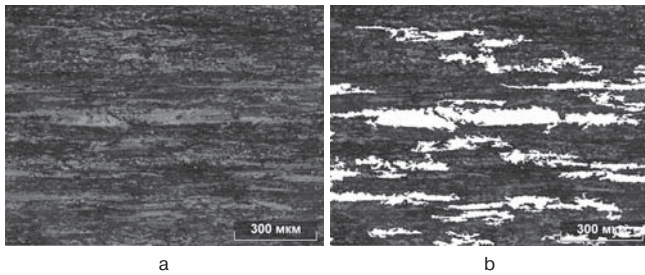


Fig. 7. Panoramic image of color etched structure in the bright field (a) and structure recognized by Thixomet Image Analyzer (b). Original magnification: 200X

into account that image contrast under polarized light decreases while magnification increases.

3.2. Carrying-out of quantitative analysis for blocks of bainite with lath morphology in the conditions of “automatic measurements” using corresponding module of Thixomet Image Analyzer (pictures 7,a and 7,b); these areas are revealed by the proposed color etching method.

3.3. After obtaining quantitative evaluation of structural constituents (in our case – bainitic areas with lath morphology), elongated in the rolling direction (Fig.7), we can estimate their effect on the properties of the plate.

Investigations have shown that the structure inhomogeneity and the length of longitudinal interphase boundaries have negative effect on the fracture behavior during drop weight tear tests (DWTT) and practically lead to slip cracks. In ferrite-pearlite steels, such boundaries are presented by the boundaries between ferrite and long pearlite colonies that are elongated along the rolling direction, while in bainitic steels these are boundaries between the elongated blocks of bainite with lath morphology and other structural constituents [6].

Slip cracks of samples after DWTT are practically absent if structure is rather uniform and is characterized by absence of elongated interphase boundaries (Fig.8). The larger is an anisotropy ratio and the longer are the interphase boundaries, the more intensive will be the slip cracks observed on fracture of samples after DWTT (Fig.9).

D. Development of centerline segregation evaluation technique

Most standards for quality estimation of mill products specify the investigation of samples taken from the quarter of plate thickness (not taking into account the central area). Usually the central area contains the rough marks of zonal

liquation that cannot be fragmented during rolling and that have negative influence on mechanical properties of plate metal. Centerline segregation is considered to be the roughest appearance of inhomogeneity and anisotropy in the pipeline steel.

Centerline segregation is evaluated via different techniques. E.g., Dillinger technique deals with visual evaluation of the total plate thickness including the central area. One of the six classes is determined after examination of the plate templates by the unaided eye. This technique does not deal with any detailed evaluation of the structure of centerline segregation during its microscopic examination on etched cross section, therefore, it takes into account neither the number and the size of bands inside it nor non-metallic inclusions that are decorating it.

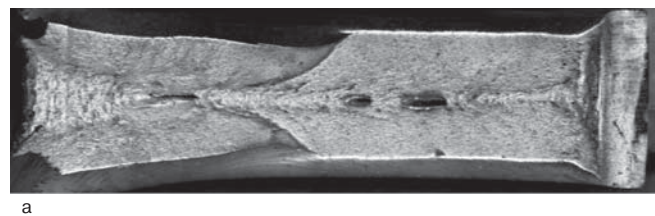


Fig. 8. Fracture surface of a sample after DWTT (a) and bainite microstructure corresponding with it (b). The magnification bar is 50 microns long

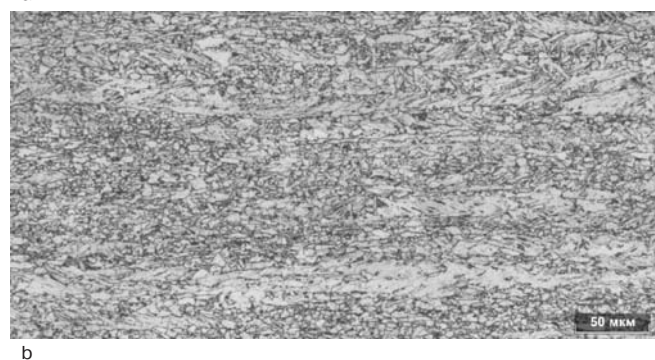
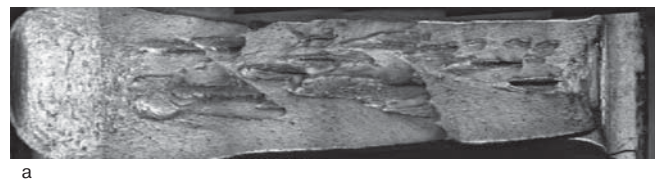


Fig. 9. Fracture surface of a sample after DWTT (a) and bainite microstructure corresponding with it (b). The magnification bar is 50 microns long.

Meanwhile, it is well-known that just these parameters of centerline segregation are decisive in the formation of the mechanical and corrosion-resistant metal properties. GB/T 13298 technique assigns class for hot-rolled plate structure on the basis of research results of its central area at magnification 200X. Additionally, structure is evaluated at magnification 500X to assess the non-metallic inclusions that are decorating the band. Such inclusions or wide single band can be the basis for an assignment of additional 0.5 class penalty. Class 1 means the structure with slightly visible discontinuous bands in the field of view, class 2 is assigned in the cases when number of such bands is not more than 3, class 3 is for the structure with more than 3 bands, class 4 means 3 bands located close to each other and uniformly.

As it was mentioned above, an assignment of the structure class is made mainly in qualitative way by visual evaluation, therefore, such evaluations of centerline segregation are inexact and subjective.

In order to improve the objectivity the technique of quantitative evaluation of centerline segregation in plate has been developed. It applies the ratio of microstructural inhomogeneity (RMI) [9] that is calculated based on local specific excess of the microhardness in centerline segregation. RMI is well-defined segregation parameter for all main alloying elements and impurities in steel and, therefore, can be considered as an objective characteristic of centerline segregation. However, this technique can be used only for research works (due to its labour intensity), so we need to develop more simple and at the same time more objective evaluation method for routine control.

Reference standard of GB/T 13298 technique has been taken as the basis. The method of directed secants [4] was used

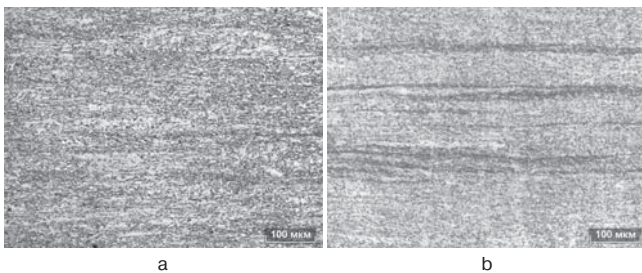


Fig. 10. Images of microstructure from the centerline segregation: 1 class (a), 4 class (b)

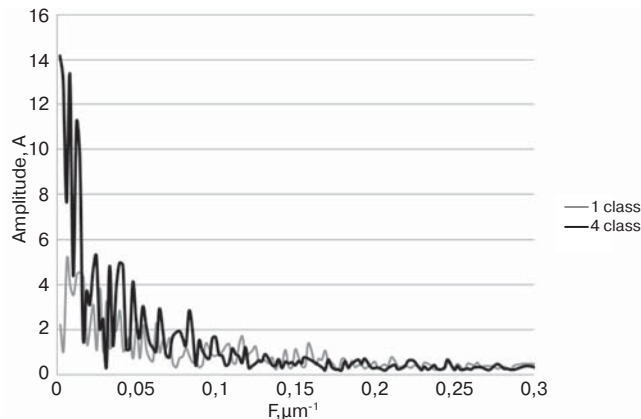


Fig. 11. Spectra of the $M(y)$ function for the images of centerline segregation of 1 and 4 classes

in an evaluation technique for microstructural banding of ferrite-pearlite and ferrite-bainite steels. This method requires a detection of the dark structural constituents (pearlite or carbides in bainite) according to the grayscale level. Modern pipeline steels are manufactured using the technology of thermomechanical processing with accelerated cooling systems at the end of finish rolling. In this case the plate is characterized by bainitic structure practically and the detection of any structural constituents including centerline segregation seems to be impossible due to complicated bainite morphology.

The image of centerline segregation is a periodic signal and for evaluation of its parameters we propose the spectral analysis by using Fourier transform.

In order to see the image of microstructure of the center area at magnification 200X, the average values of the grayscale level are calculated on the secants parallel to the rolling direction ($M(y)$). Deviations of these values reflect the presence of dark bands on the image. The spectrum of ($M(y)$) function is calculated using discrete Fourier transform.

Microstructure images of centerline segregation of 1 and 4 classes are presented in the Fig.10, correspondingly, while their spectra are shown in the Fig.11.

It was established via expert evaluation method that the amplitude of harmonics (A) in the frequency range between 0 and $0.05 \mu\text{m}^{-1}$ described the centerline segregation degree in accordance with standard charts in optimal way. Therefore, the sum of amplitudes of harmonics in this frequency range is calculated (L).

Equation used for class assignment can be expressed as follows:

$$B = -3,56 + 0,08 * L \quad (3)$$

Correspondence between the classes obtained using image analysis and the results of expert evaluation presented in the Fig.12 confirms adequacy of the developed technique. It is also important that the class assigned in such a way is objective because image analysis algorithm does not require operator participation.

Therefore, the package of methods for quantitative evaluation of structural inhomogeneity of pipeline steel has been developed. This package covers all known kinds of an inhomogeneity that are observed in light microscope.

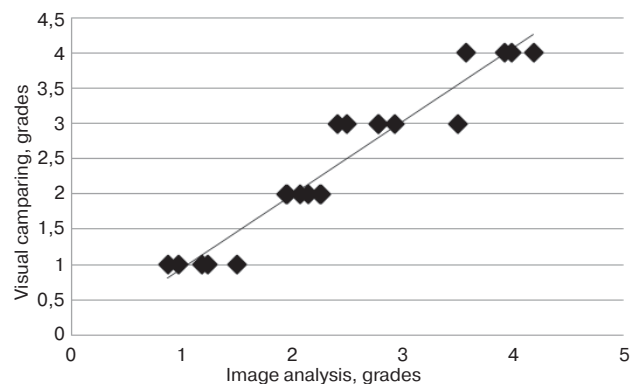


Fig. 12. The correlation results of class values of centerline segregation, obtained via visual comparing with standard charts (see the axis of ordinates) and via image analysis (see the axis of abscissa)

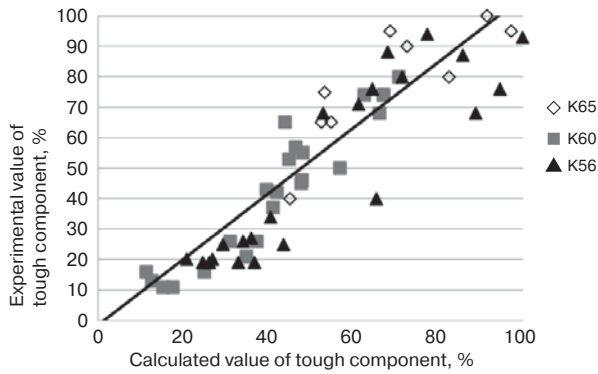


Fig. 13. Correlation of calculated (see the axis of abscissa) and experimental (see the axis of ordinates) values of a tough component in fracture of DWTT specimens

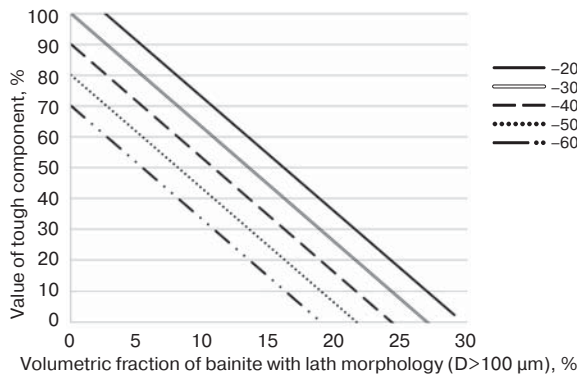


Fig. 14. Calculated values of tough component in fracture of DWTT specimens (see volumetric fraction of bainite with lath morphology (D > 100 μm) in the axis of abscissa and value of the fraction of tough component in the axis of ordinates)

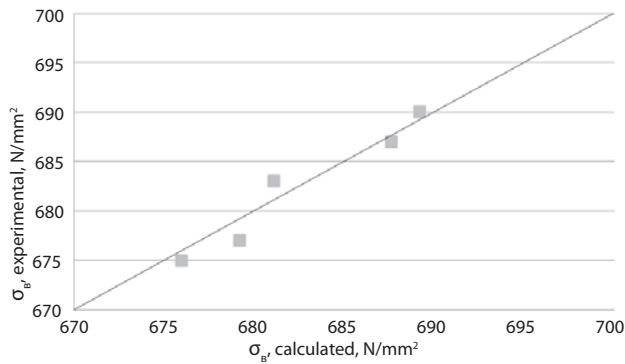


Fig. 15. Correlation of calculated (see the axis of abscissa) and experimental (see the axis of ordinates) values of tensile strength for steel with K65 strength category

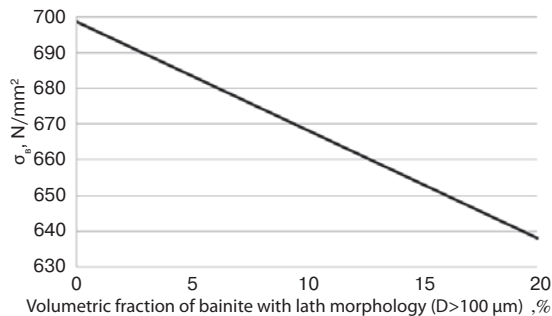


Fig. 16. Calculated values of a tensile strength for steel with K65 strength category (see the axis of ordinates, volumetric fraction of bainite with lath morphology, D > 100 μm, see on the axis of abscissa)

E. Influence of structural inhomogeneity on mechanical properties of pipeline steels

Investigations have been carried out on pipeline steel for following grades: K56 (X65), K60 (X70), K65 (X80) with ferrite-bainitic structure after controlled rolling. Investigated steels were characterized by different level of strength properties and impact energy that allowed us to reveal structure-properties relationships. All structure parameters that have been evaluated according to the above-described techniques, as well as the grain size and non-metallic inclusions, were involved in multi-dimension statistical analysis. However, all investigated properties were adequately described only by such parameters of steel inhomogeneity as the length and the number of blocks of bainite with lath morphology, the most weak constituent of pipeline steels structure at present time.

Examples of an influence of volumetric fraction of bainite with lath morphology with blocks having length more than 100 μm on mechanical properties of steel are presented below.

The regression equation (4) adequately describing experimental data with 0.9 correlation ratio has been obtained for three strength categories:

$$B = 129,68 + 0,99 \times T_{\text{test}} - 3,71 \times V_{B>100\mu\text{m}}, \quad (4)$$

where B is an amount of tough component in fracture during DWTT, %; T_{test} is the temperature of DWTT, °C; $V_{B>100\mu\text{m}}$ is volumetric fraction of bainite with lath morphology with blocks having length more than 100 μm, %

The graphs describing relationships between the fraction of tough component and volumetric fraction of bainite with lath morphology (D > 100 μm) at set temperature in the range from -20 to -60°C have been plotted according to the equation (4) (Fig.13 and Fig.14).

The regression equation, adequately describing tensile strength with volumetric fraction of blocks of bainite with lath morphology (having size more than 100 μm), has been obtained for steel with K65 strength category and presented on Fig.15 and Fig.16:

$$\sigma_B = 698,44 - 3,01 \times V_{B>100\mu\text{m}} \quad (5)$$

The regression equation connecting the impact toughness (KCV) and the volumetric fraction of lath morphology bainite with blocks having length more than 100 μm (%) has been obtained for K56 steel and presented in Fig. 17 and Fig.18:

$$KCV (CVN) = 675,17 - 85,53 \times V_{B>100\mu\text{m}} \quad (6)$$

Therefore, it is necessary to minimize size for bainitic areas with lath morphology in order to increase the strength properties of examined pipeline steels. Additionally, it is possible to reveal ultimate size of such areas and their volumetric fraction in the structure for each steel strength category (Figs.14, 16 and 18).

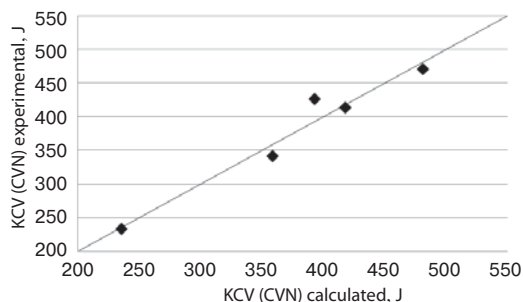


Fig. 17. Correlation of calculated (see the axis of abscissa) and experimental (see the axis of ordinates) values of impact toughness for steel with K56 strength category

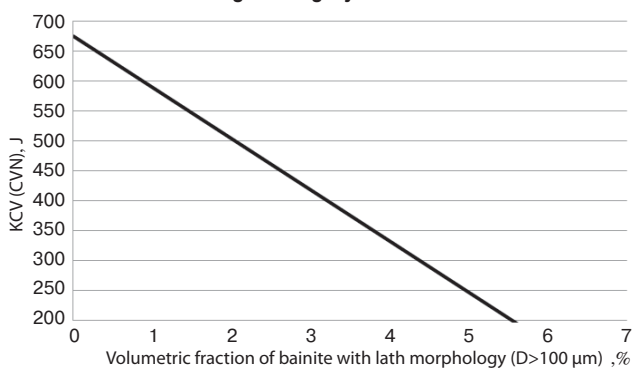


Fig. 18. Calculated values of impact toughness for steel with K56 strength category (see the axis of ordinates, volumetric fraction of bainite with lath morphology, $D > 100 \mu\text{m}$, see on the axis of abscissa)

Conclusions

1. The techniques of quantitative description of all kinds of structural inhomogeneity in modern pipeline steels (such as microstructure banding, general anisotropy, blocks of bainite with lath morphology, centerline segregation) have been developed, realized and put into practice as a motorized hardware-software complex "Thixomet Smart Drive" at dozens of enterprises.

2. It was established that the structural inhomogeneity evaluated by the volumetric fraction and the length of the areas of bainite with lath morphology elongated along the rolling direction adequately described the mechanical properties during tensile and impact bend tests of pipeline steels with yield strength from 485 up to 625 N/mm².

3. Quantitative criteria of an evaluation of structural anisotropy for ferrite-bainite steels are the important parameters for their quality evaluation during acceptance tests as well as for improvement of production technology of pipeline steel.

4. All developed structure evaluation techniques for ferrite-bainite steels generate the system of quantitative criteria of quality evaluation for pipeline steels after controlled rolling and are the basis for their quality control system.

REFERENCES

1. Shigeaki Ogibayashi. Advances in Technology of Oxide Metallurgy. Nippon Steel Technical Report. April 1994. No. 61. 76 p.
2. *Novosti Metallurgii po Stranam i Regionam. Kitai. Novyi tolstolistovoi stan kompanii Xoangtan* (Iron and Steel News of the World. China. New heavy plate mill of Xiangtan). *Chernye Metally - Ferrous metals*. 2010. № 12. p 10.
3. Saltykov S. A. Stereometricheskaya metallografiya (Stereometric metallography). Moscow. Metallurgiya. 1970. 376 p.
4. Kazakov A. A., Kiselev D. V., Andreeva S. V., Chigintsev L. V., Golovin S. V., Egorov V. A., Markov S. I. *Razrabotka metodiki kolichestvennoi otsenki mikrostrukturnoi poloschatosti nizkolegirovannykh trubnykh staley s pomoshchyu avtomaticheskogo analiza izobrazheniya* (Development of the technique for quantitative evaluation of microstructural banding for low-alloyed pipeline steels using automatic image analysis). *Chernye Metally - Ferrous metals*. 2007. № 7-8. p 31-37.
5. Pratt W. K. *Tsifrovaya obrabotka izobrazheniy* (Digital Image Processing). Moscow. Mir. 1982. Book 1. 312 p. (Translation from English: A. Willey – Interscience Publication. John Willey and Sons. 1978).
6. Malakhov N. V., Motovilina G. D., Khlusova E. I., Kazakov A. A. *Strukturnaya neodnorodnost i metody ee snizheniya dlya povysheniya kachestva konstruktsionnoi stali* (Structural inhomogeneity and methods for its lowering for quality improvement of structural steels). *Voprosy Materialovedeniya – Material Science Problems*. 2009. № 3 (59). p. 52-64.
7. RF Patent № 2449055. *Sposob issledovaniya struktury trubnykh staley* (The method of examination of pipeline steels structure).
8. Kazakov A. A., Kazakova E. I., Kiselev D. V., Motovilina G. D. *Razrabotka metodov otsenki mikrostrukturnoi neodnorodnosti trubnykh staley* (Development of the methods for evaluation of microstructural inhomogeneity of pipeline steels). *Chernye Metally – Ferrous metals*. 2009. № 12. p. 12-15.
9. Kazakov A. A., Chigintsev L. V., Kazakova E. I., Ryaboshuk S. V., Markov S. I. *Metodika otsenki likvatsionnoi polosy listovogo prokata* (The technique for evaluation of centerline segregation in rolled plate). *Chernye Metally - Ferrous metals*. 2009. № 12. p. 17-22.