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TECHNICAL ARTICLE

Industrial Application of Thixomet Image Analyzer for Quantitative Description of Steel and Alloy's Microstructure

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Abstract Numerous examples of practical use in industry of the Thixomet image analyzer for quantitative description of the microstructure of steels and alloys have been described. More than 200 image analyzers have been installed at Universities, R&D labs, and plants during the twenty years of the Thixomet's history. Sixty-five Thixomet plug-ins cover all Russian and most of International standards. The techniques of quantitative description of non-metallic inclusions, grain size, and all kinds of microstructural inhomogeneity in modern pipeline steels (such as microstructure banding, general anisotropy, blocks of a bainite with lath morphology, centerline segregation) have been developed, realized, and put into practice as a motorized hardware–software complex, Thixomet SmartDrive, at many enterprises. It was established that structural inhomogeneity evaluated by volume fraction and a blocks length of a bainite with lath morphology elongated along the rolling direction adequately described the mechanical properties of pipeline steels. The name of Thixomet was taken because of Greek "Thixis" (touch) and "Metrisi" (measurement), so an idea is "One Touch Measurement." Measurement operations from capturing an image up to a generation of quantitative description of a structure as a report are actually performed by several evident actions that confirm the name of the product. Thixomet has succeeded in international interlaboratory Round Robin Test Programs and performed with the good results across worldwide Image Analysis systems. This was a cause to get an official metrological certificate from Russian State Metrological Committee which certified Thixomet as a measurement tool.

Keywords Steel and alloys · Microstructure · Image analyzer · Microstructural inhomogeneity · Pipeline steels · Structure–property relationships

Quantification of Semi-Solid Materials (SSM) Structure

In 1996, the very first Thixomet image analyzer unit was installed at the Alumax Technical Center (Golden, CO) for the quantification of semi-solid materials structure based on the A356 alloy. Along with an evaluation of porosity distribution, eutectic content (continuous and occluded) and silicon content across the billet's diameter, a special grain shape factor was calculated in studies of anodized specimens examined in polarized light [1]. An identification and integration of dendrite fragments, revealed in a planar image, into one structurally isolated object was possible as a result of this measurement (Fig. 1). So, this shape factor of thixotropic grains can be evaluated taking into account the structural connections between its fragments. In this approach, a total specific length of interfacial boundaries is a function of the number of identified structurally isolated objects in 3D and not to the overall number of grains revealed in the 2D field of view. The grains are grouped based on the principle of their identical crystallographic orientation fixed by the common color of

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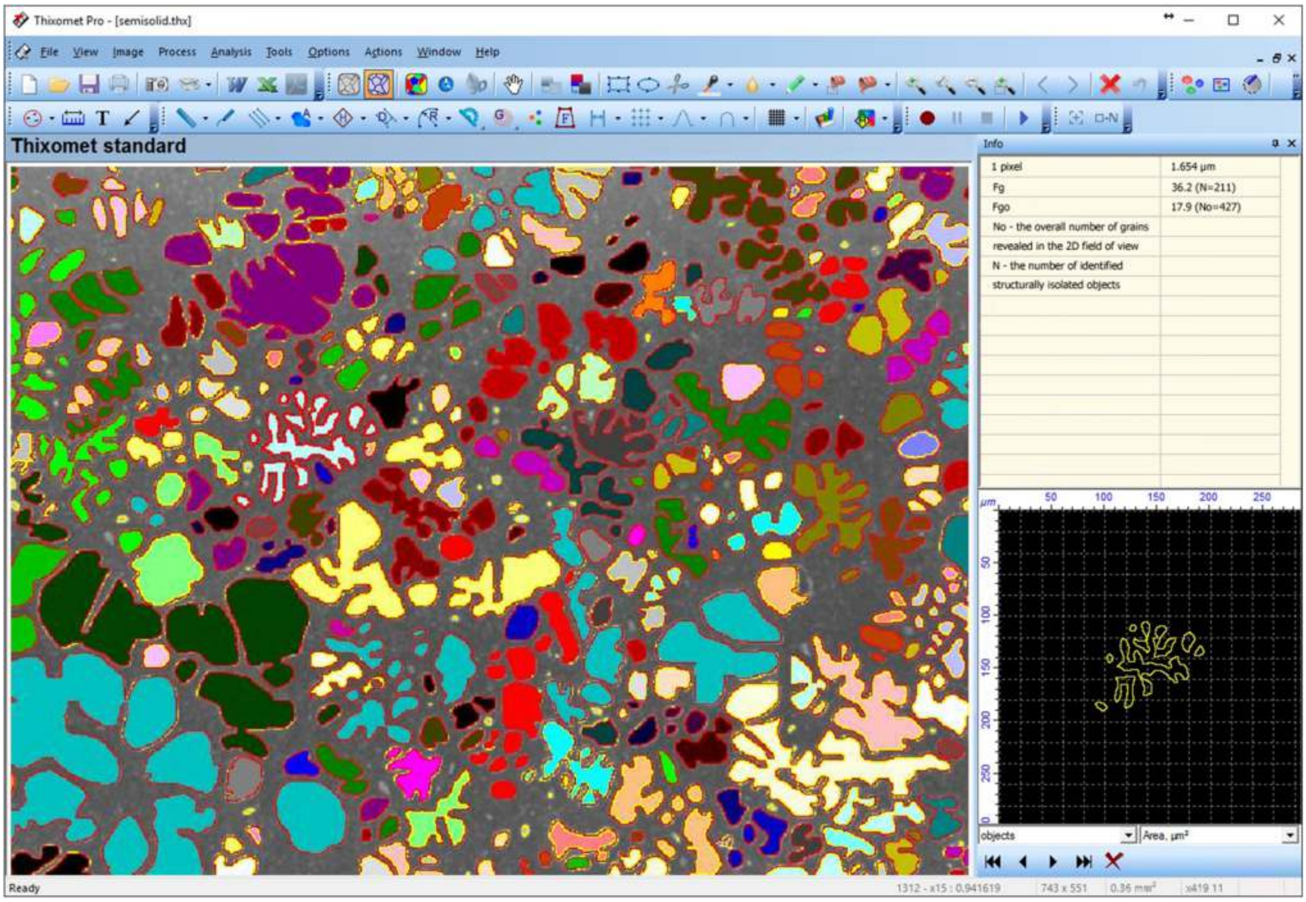

Fig. 1 Thixomet screenshot. Alpha-solid solution grains are colored according to similar crystallographic orientation of the grains

these grains. Thixomet software also performs a total quantitative description of 2D SSM structure, as well as direct visualization and measurement of 3D structure by processing data obtained using serial sections [1].

Metallurgical Quality Estimation of Nickel-Based Superalloy Charge Billets

Since 1997 the Thixomet has been used by many Russian turbine blade plants for quality estimation of nickel-based superalloy charge billets. The total impurity rate is calculated with regard to the contribution of each impurity type: oxide films, slag globules, nitride clusters [2]. Their recognition is based on a grayscale level of the impurities, their morphology, and a character of mutual arrangement (Fig. 2). Oxide film elongation is calculated as a continuous linear feature; in this case, the breaks inside films are connected. The area occupied by nitride clusters or slag globules is estimated by an area of the figure, which is formed by an external boundary of impurity clusters. After this automatic analysis, the user can edit results by assigning “NC clustering rate” for nitride clusters or “SG clustering rate” for slag globules (Fig. 2). It means that all inclusions around each of them with distance equal to “NC(SG) clustering rate” will be incorporated into the cluster. The rate for nitride clusters is 10 times higher than for slag globules and oxide films in the same dimensional group, since nitrides and their clusters are more difficult to remove from melts than slag globules or oxide films, which are easily assimilated by the refractory lining of a crucible during remelting or by a ceramic filter during casting. The decision to use a charge billet in production of turbine blades is based upon the metallurgical quality evaluation results. Billets with an impurity rate above a defined critical value are withdrawn from production.

Thixomet Today

More than 200 units have been installed at Universities, R&D labs and plants during 20 years of the Thixomet’s history. Thixomet plug-ins cover all Russian and most International standards on non-metallic inclusions: ASTM E1245, ASTM E2283, ASTM E45, DIN 50602, EN 10247, ISO 4967, SEP 1572, GOST P51685, GOST 801, GOST 1778; grain size:

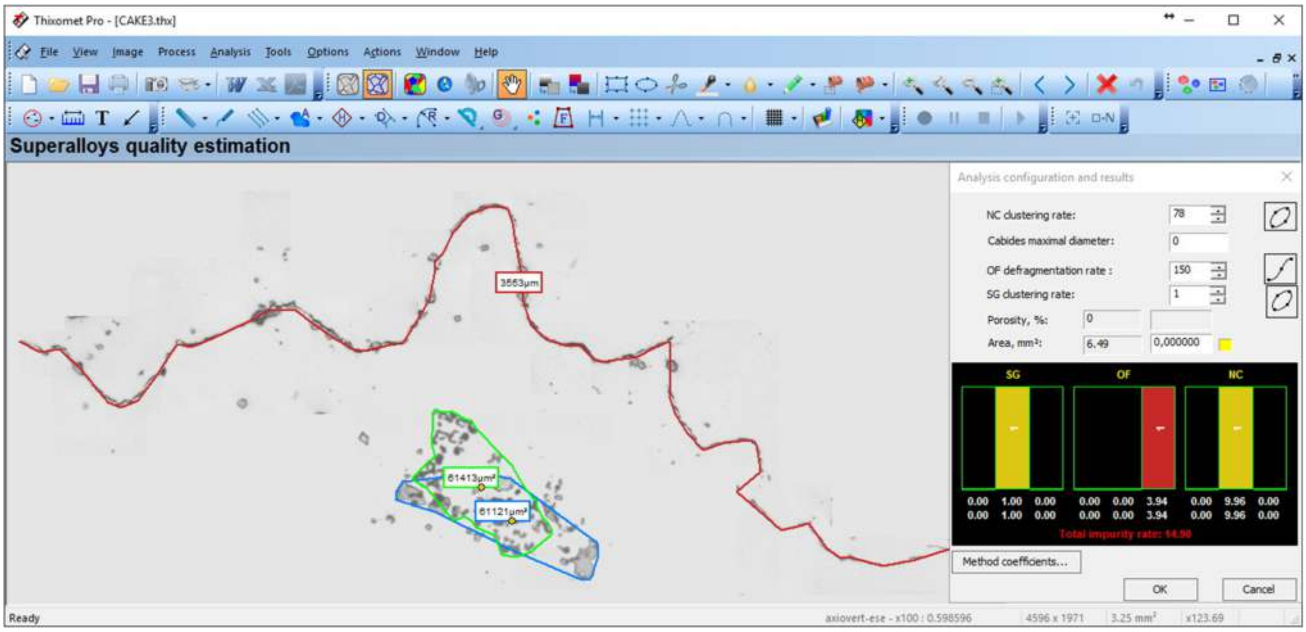

Fig. 2 Thixomet screenshot. The recognition of oxide films, slag globules, and nitride clusters is based on the grayscale level of the impurities, their morphology, and a character of mutual arrangement

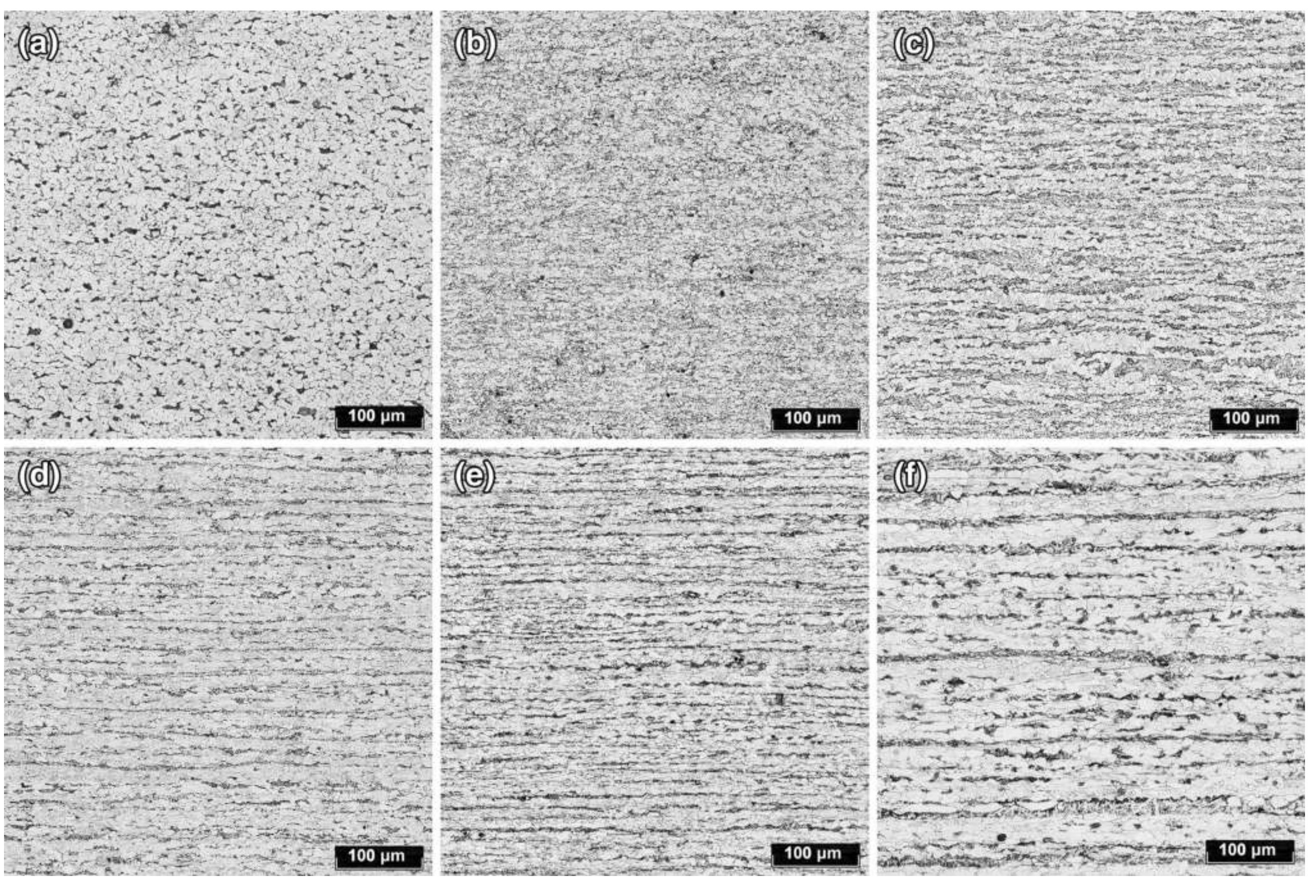

Fig. 3 New scales for ferrite–bainite microstructure. The banding coefficient (BR) is used to estimate a microstructure from randomly oriented up to banded according to new banding grades (BG): **a** BG = 0, BR = 1.2; **b** BG = 1, BR = 1.7; **c** BG = 2, BR = 2.5; **d** BG = 3, BR = 4.0; **e** BG = 4, BR = 4.8; **f** BG = 5, BR = 6.0

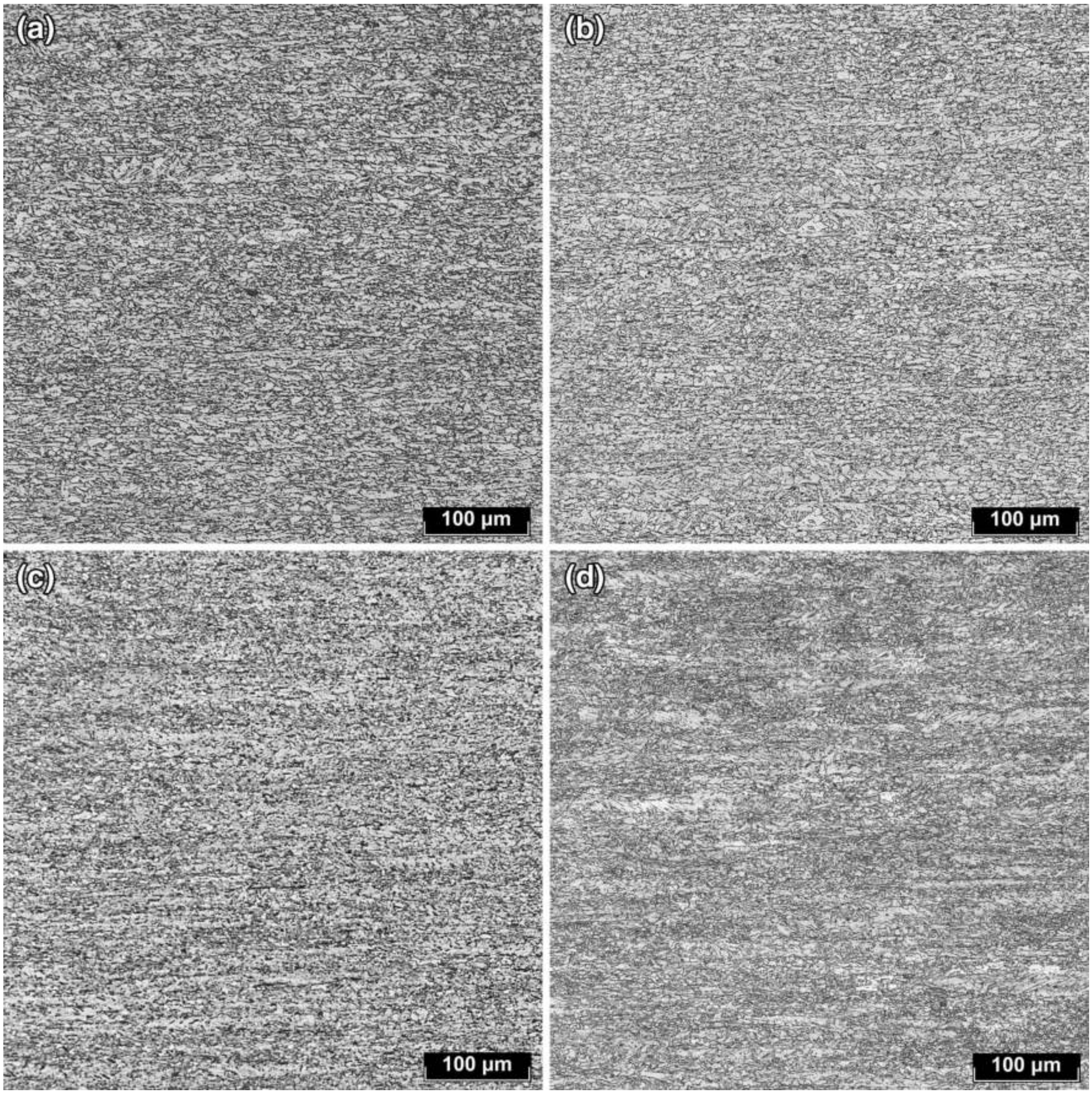

Fig. 4 Bainitic steel's microstructures with different values of anisotropy ratio: **a** AR = 0.3; **b** AR = 1; **c** AR = 1.6; **d** AR = 2.5

ASTM E1382, ASTM E112, DIN 50601, ISO 643, ISO 2624, GOST 5639; banding: ASTM E1268, GOST 5640; layer depth: ASTM E1077, ISO 3887, GOST 1763; and cast iron: ASTM A247, ASTM E2567, ISO 945, GOST 3443. It is just a list of 25 from available 65 Thixomet plug-ins.

Characterization of Modern Pipeline Steel's Microstructure

First, five Russian standards for total characterization of the structure of modern pipeline steels beginning from non-metallic inclusions and grain size and finishing by assessment of all kinds of microstructural inhomogeneity by optical microscopy were developed [3]. All these standards are based on panoramic images created by the Thixomet SmartDrive. Only the panoramic approach allows one to carry out precise measurements at a high resolution using sufficient area to obtain adequate assessment of structural quality. A motorized hardware–software complex, the Thixomet SmartDrive, was developed and installed at 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 [3–5].

Microstructural Banding

Banding is determined according to GOST 5640-68, a chart method based on the principle of increasing the number of bands of pearlite, taking into account their continuity and degree of ferrite grain elongation. The modern generation of pipeline steels has a ferrite–bainite microstructure with fine grains. We have developed a new set of scales to evaluate such microstructures. These new scales use the same principle as the existing scales for ferrite–pearlite structure: increasing number of bainite bands considering their continuity and an anisotropy of the ferrite grains (Fig. 3). It should be noted that comparison between standard chart images and those of a specimen were conducted at ×100, according to GOST 5640-68, while analysis of elongated ferrite grains required ×500. Such an analysis is possible only by sequential examination of a specimen at two magnifications. In classic metallography, it is impossible to observe the same wide field of view provided by ×100 at ×500. Therefore, classic metallographic investigations are always a compromise between an examination area and a resolution. But, if we apply modern methods of quantitative metallography using the Thixomet image analyzer, we can simultaneously evaluate both banding and ferrite grain elongation making measurements only at ×500, while the required area corresponding to the size of the field viewed at ×100 can be subsequently "stitched" together electronically from adjacent fields of view. When a motorized microscope stage moves to the next field of view, the previous field is stitched precisely, "pixel to pixel" to the field that has just been captured before. A high-resolution "panoramic image" of any desired large area can be created in such a way. The assigned grade is based on stereological parameters that have been created using a directed secants method with the assistance of an automatic image analyzer. The standard deviation of the fraction of a second phase on secants that are parallel to the rolling direction divided by the standard deviation of the fraction of a second phase on secants that are perpendicular to the rolling direction will unambiguously characterize structural banding [3]. This new banding coefficient (BR) can be used to estimate a

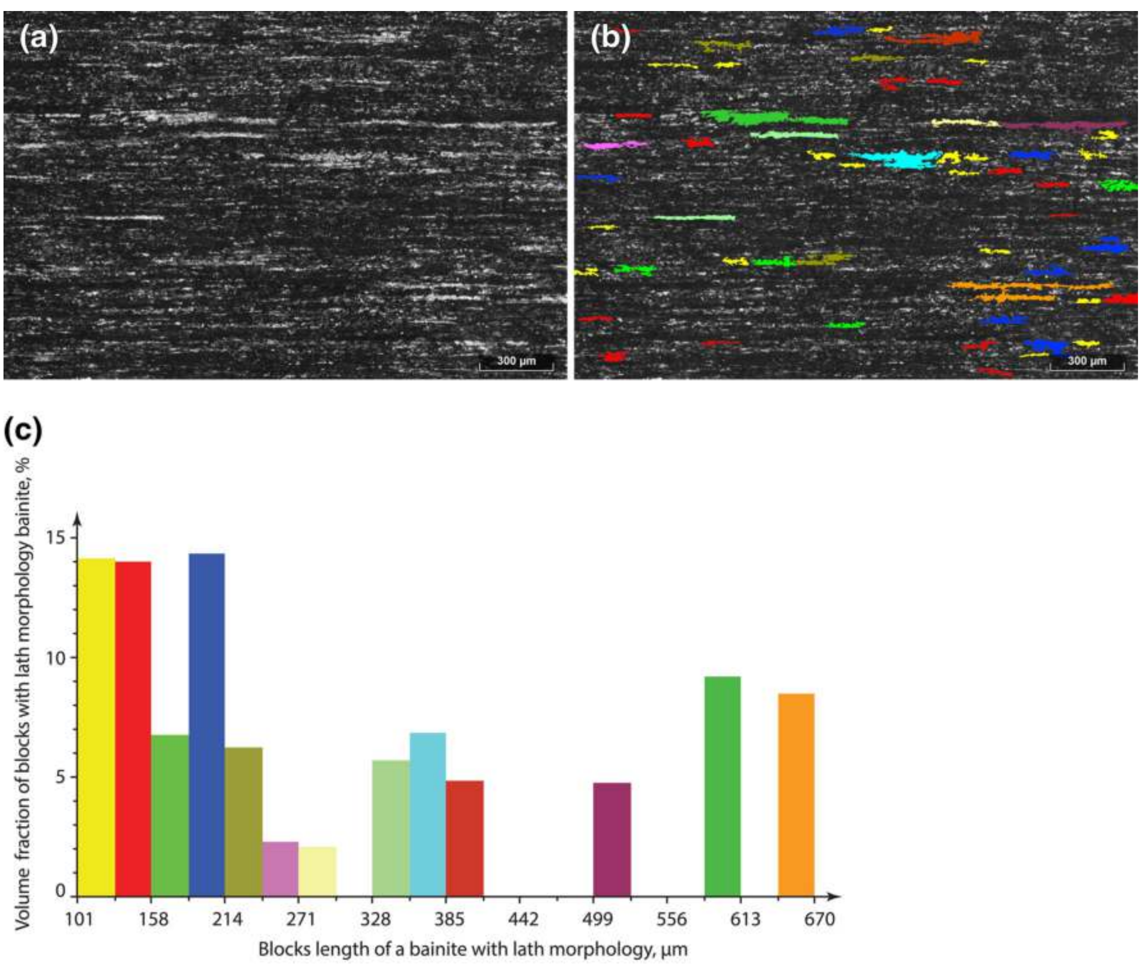

Fig. 5 Selection of the bainite blocks with lath morphology: **a** microstructure after tint etching in polarized light, ×100; **b** bainite blocks colored according to their size; **c** result of measurements

microstructure from randomly oriented up to banded according to GOST for ferrite–pearlite and new banding grades (BG) for ferrite–bainite microstructure. The banding grades are separated by critical values of the banding coefficient (Fig. 3).

Estimation of Bainitic Structure Anisotropy

Modern pipeline steels are manufactured using thermomechanical treatment technology which includes accelerated cooling after hot rolling producing plate with an almost 100% bainitic structure free from microstructural banding. However, anisotropy of bainitic structure can be observed in such steels (Fig. 4). The stereological methods that have been described above for the evaluation of ferrite–pearlite and ferrite–bainite microstructures are not applicable for a description of complicated bainitic morphologies. Hence, a method for an evaluation of bainite anisotropy based on texture analysis of images has been developed for this case [2]. Texture analysis is conducted for an extraction of quantitative attributes that characterize an examined image. The task is to find such attributes that can unambiguously characterize microstructural anisotropy, i.e., the presence and intensity of a priority direction in a microstructure. 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 the general anisotropy of a microstructure of bainitic steels. Images of microstructures with different values of anisotropy ratio (AR) are displayed in Fig. 4.

Estimation of Blocks with Lath Morphology Bainite

The results of investigations revealed blocks of bainite with lath morphology among different morphological forms elongated along the rolling direction (Fig. 4d). These bainitic blocks with lath morphology are the main contributors to general anisotropy and decrease the essential mechanical properties of a plate in a transverse direction. The technique of tint etching by sodium metabisulfite with consequent analysis in polarized light has been developed and covered by a Russian Federation patent [6]; it promotes perfect selection of bainite blocks with lath morphology (Fig. 5a) and facilitates a measurement of their volumetric fraction and a length of longitudinal inter-phase (Fig. 5b, c). The grayscale image (Fig. 5a) was obtained after tint etching in polarized light without lambda plate. This image is easily binarized and quantified.

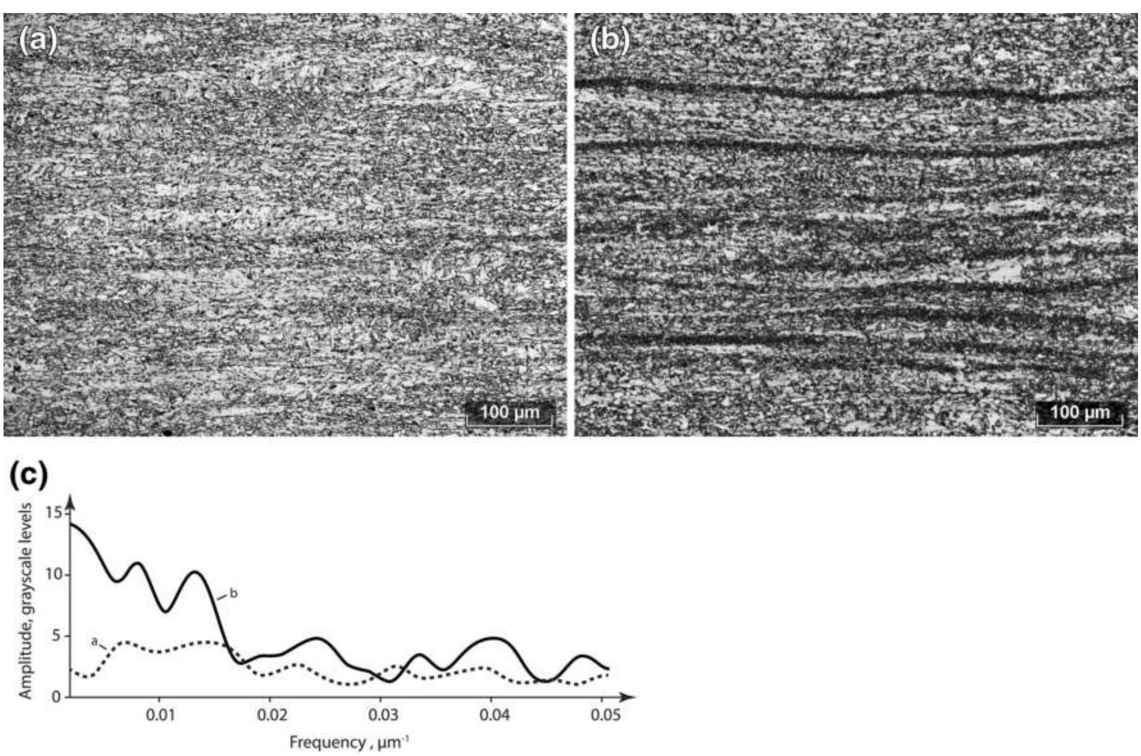

Fig. 6 Assessment of the centerline segregation zone: M(y) spectrums (**c**) for images of 1st (**a**) and 4th (**b**) grades

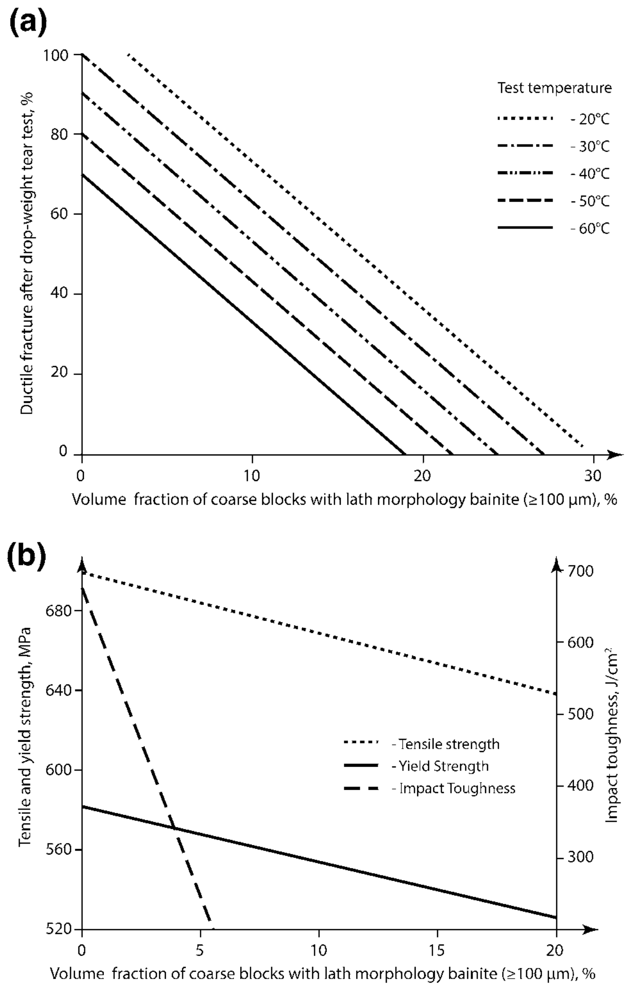

Fig. 7 Mechanical properties vs. volume fraction of coarse blocks with lath morphology bainite ($\geq$100 μm): **a** ductile fracture after a drop-weight tear test at temperature in the range from −20 to −60 °C; and, **b** impact toughness, tensile, and yield strength

Development of Centerline Segregation Evaluation Technique

The parameters of centerline segregation are decisive in the formation of mechanical and corrosion-resistant metal properties. The GB/T 13298 technique assigns classes for hot-rolled plate structure based on the research results of its central area at a magnification of $\times$200. Additionally, the structure is evaluated at $\times$500 magnification to assess the non-metallic inclusions that are decorating the band. Such inclusions or a wide single band can be a basis for an assignment of an additional 0.5 class penalty. Class 1 defines a structure with slightly visible discontinuous bands in a field of view; class 2 is assigned to the cases when a number of such bands is not more than 3; class 3 is for a structure with more than 3 bands; class 4 means 3 bands located close to each other uniformly. The image of centerline segregation is a periodic signal and for an evaluation of its parameters we propose a spectral analysis using the Fourier transform [3]. In order to see an image of a microstructure of center area at magnification $\times$200, the average values of grayscale level are calculated on the secants parallel to a rolling direction (M(y)). Deviations of these values reflect a presence of dark bands in an image. The spectrum of the (M(y)) function is calculated using the discrete Fourier transform, established via the expert evaluation method so that an amplitude of harmonics in the frequency range between 0 and 0.05 μm^{-1} describes the degree of centerline segregation in accordance with standard charts in an optimal way (Fig. 6).

Influence of Structural Inhomogeneity on Mechanical Properties of Pipeline Steels

Investigations have been carried out on pipeline steels of the following grades: X60, X70 and X80 with ferrite–bainitic structures after controlled rolling. Investigated steels were characterized by their different levels of strength properties and impact energies that allowed development of structure–property relationships. All structure parameters that have been evaluated according to the above-described techniques, as well as 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 a length and a number of blocks of a bainite with lath morphology. The regression equation adequately describing experimental data has been obtained and the graphs describing relationships between a fraction of tough component, tensile and yield strength, impact toughness versus volume fraction of a bainite with lath morphology ($D > 100$ μm) have been plotted (Fig. 7). Therefore, it is necessary to minimize the size of bainitic areas with lath morphology in order to increase mechanical properties of steels.

Conclusions

1. Twenty years' of experience with the development and industrial applications of the Thixomet Image Analyzer has been summarized. Examples of practical applications of the Thixomet for the purpose of technological improvement for commercial steel and alloys have been demonstrated with a focus on the unique advantages of techniques for quantitative structure estimation.
2. The techniques for quality estimation of aluminum alloys produced by semi-solid processing have been

developed and applied for rheocasting improvement. The estimation of metallurgical quality of nickel-based superalloys charge billets greatly reduced the rejection rate of turbine blades. The assessment of all kinds of microstructural inhomogeneities in modern pipeline steels revealed “a bottle neck” for such kind of steels structures and led to improvements in their production technology with this goal in mind.

3. The quantitative description of an alloys microstructure by means of the Thixomet Image Analyzer can play a critical role in the quality system for improved steel and alloys production. As soon as the “structure–properties” database will be created, the factory acceptance test results based on mechanical properties will be enhanced by the quantitative structure assessment.

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