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Digital measurements of non-metallic inclusions in steel

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Abstract: The experience of many-year research has shown that optimizing the steel chemical composition and microstructural characteristics, as well as reducing its contamination with non-metallic inclusions (NMI), it is possible to significantly increase the corrosion resistance of oilfield pipeline steels and increase the time of their trouble-free operation. The influence of complex NMIs on the steel corrosion resistance is determined by both the chemical composition of NMIs and their quantitative ratios. Therefore, obtaining metal products of the required quality is possible only when using the "control by structure" principle. In the work, based on the analysis of brightness fields of images (on a sample scale) in 256 shades of gray, the authors proposed digital, metrologically supported procedures for measuring the NMI heterogeneity of low-carbon oilfield steels: eliminating the heterogeneity of field illumination, justifying the criteria for binarization and noise filtering. For low-carbon steels of various types of melting, the authors identified the key role of dispersed non-metallic inclusions ranging in size from 5–10 μm^2 to 2 nm^2 in the formation of the corrosion resistance of steels. This may explain why, in some cases, there is no interrelation between the corrosion rate and the fracture resistance of steels, the formation of which is determined by larger particles. When representing the NMI as a set of random points on the plane, the distribution of distances between the nearest ones is estimated based on Voronoi polyhedra statistics. The study shows that an increase in the kurtosis coefficient of distributions of polyhedra areas is accompanied by an increase in the corrosion rate of the steels under study. This indicates the negative impact of heterogeneity in the arrangement of dispersed NMIs on the corrosion resistance of steels.

Keywords: digital measurements of structures; quality management by structure; non-metallic inclusions in steel; corrosion resistance of oilfield pipes.

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INTRODUCTION

Producing high-quality steel is possible only when controlling the non-metallic inclusions (NMI) present in it, which are an inevitable product of the technological process [1; 2]. The size of inclusions in steel depends on their origin and can vary from 0.01 microns to 10 mm. Due to their small volume fraction, NMIs primarily affect the destruction processes, but each size group affects in its own way [1]. Large inclusions visible to an unaided eye contribute to the formation of areas of destruction or corrosion. Small inclusions (less than 1 mm) can lead to the formation of fatigue failure sites. Dispersed inclusions can affect the plastic properties, and susceptibility to the austenite grain growth [3]. The quality of the metal is determined both by the NMI volume fraction, and size and by their shape and heterogeneity of location. In turn, analysis of the NMI chemical and phase composition, makes it possible to diagnose their origin for further elimination of the negative impact. The works [4–6] reflect the peculiarities of the influence of the size, shape and type of inclusions, on the mechanical properties of structural steels. NMI contamination is one of the main reasons for the rapid failure of oil pipes [7]. The corrosion activity of complex Nis, present in modern steels, depends on their chemical and phase composition, the optimization of which can prevent the negative impact of NMIs on the corrosion resistance of steels, under service conditions of oilfield pipelines [8].

Due to the significant influence of NMIs on the properties of steel, various methods are used to analyze them. To study the chemical composition of single NMIs, scanning electron microscopy, with the possibility of performing

X-ray spectral microanalysis is used. To determine the size and shape of inclusions, and estimate the distance between them, the extreme value method, Spark-DAT, thermodynamic calculations, etc. are used [9–11]. However, the ranking of structures based on comparison of their images, obtained by the light microscopy methods with reference scales is the most common. The GOST 1778, 5639 and 3443 reference scales have an empirical nature; this indicator is complex, and difficult to formalize. The statistical nature of images of "score" structures is often not taken into account; therefore, there are difficulties when determining a one-to-one correspondence between GOST "scores" and the quantitative characteristics of the geometric elements of their images. Digitalization makes it possible to document quickly measurements of structures, which allows collecting measurement statistics, and formulating recommendations on product quality management based on it [12].

Through digitalization, it became possible to provide mass measurements and obtain statistical estimates of the geometric parameters of structural elements, which was previously difficult to achieve due to the large amount of manual work. The obtained representative volume of measurement results, allows evaluating the influence of structural heterogeneity on the properties of steels, taking into account their different scales. An assessment of the variation in contamination of the 38KhN3MFA steel (from large forgings) with NMIs, showed that the scale of observation is of great importance, and affects the reproducibility of the measurement results of the NMI geometry [13].

Segmentation, i. e., dividing an image into its constituent areas or objects, is one of the important stages of quantitative analysis. Non-trivial image segmentation is one of the most challenging image processing problems. Most existing image segmentation algorithms are based on one of two basic properties of the luminance signal: discontinuity and uniformity. In the first case, the approach consists in the image partition based on sudden changes in the signal, such as luminance differences in the image. The second category of methods uses dividing the image into areas that are homogeneous in terms of pre-selected criteria. Threshold processing (binarization) is an example of such methods [14]. In the standards existing today (ASTM E45–18a and DIN EN 10247:2017-09), it is recommended to use the flicker method as a binarization method for quantitative assessment of NMIs, but it is manual, and as a consequence, subjective. In turn, most image analyzers have built-in global methods. The use of global binarization methods when processing panoramas of NMI images, can lead to the binary image distortion. The reasons for this are the small size, and number of objects to study, and the feature of automatic "stitching" of image panoramas, leading to illumination heterogeneity. Therefore, it remains relevant to develop physically justified binarization methods that allow identifying objectively the informative objects in images of structures.

The practice of developing digital procedures for measuring structures and fractures shows that considering the physical laws of the formation of the brightness field of their images, when identifying binarization and filtering criteria, and a reasonable choice of the measurement object representative volumes. Taking into account their statistical nature, can provide virtually the only trajectory for preparing images for their subsequent measurement. Consequently, one cannot expect significant differences from the results of measurements of the geometry of structural elements, obtained in different laboratories, on the same metallographic specimens [15]. It is obvious that digital metallography using image analyzers that have undergone interlaboratory round-robin comparisons, will significantly simplify acceptance tests, between the supplier and consumer of metal products [16].

The purpose of this study is to develop a methodology for quantitative analysis of non-metallic inclusions on a thin section scale, to identify patterns of their influence on the corrosion resistance of oilfield pipe metal.

METHODS

Sheets of five low-carbon steels of various melts were the objects of the study. The sheets intended for oilfield pipes were produced using standard technology. Table 1 presents the chemical composition of the steels under study.

The chemical composition of steels was determined using an OBLF QSN 750 automatic analyzer and complied with the requirements of regulatory documents. The microstructure was studied on longitudinal samples cut from sheets in the as-delivered condition. The cut samples were hot pressed into resin using a CitoPress-5 automatic electrohydraulic press. Grinding and polishing were carried out on an AutoMet 250 Buehler machine. For the structure metallographic examination, the authors used an Axio Observer D1m Carl Zeiss optical microscope.

To assess the steel contamination with NMIs in our study, panoramas were obtained on the scale of a metallographic specimen (the studied area for all samples was at least 170 mm^2). Image panoramas of non-metallic inclusions were obtained on unetched metallographic specimens, using Thixomet software at a magnification of ×50. The chemical composition of complex NMIs was determined using a JSM-6610LV scanning electron microscope at a magnification of ×500 (the analyzed area was at least 3.5 mm^2 on the sample scale). Nanosized precipitates of carbide (carbonitride) excess phases were studied by transmission electron microscopy using a JEM-200CX microscope.

Tensile tests to determine strength and plastic characteristics were carried out in accordance with GOST 1497-84. Electrochemical studies to determine corrosion characteristics and subsequent calculations were carried out in accordance with ASTM G3, G5, G59, G102, ISO 17475:2005, and GOST 9.912-89 standards.

Image panoramas were processed in prepared software using C#. The color image was converted to shades of gray (from 0 to 255) using the formula:

$$I=0.299 \cdot R+0.587 \cdot G+0.114 \cdot B,$$

where R, G and B are the intensity values of red, green and blue colors, respectively [17].

To process image panoramas, a complex algorithm was developed, which includes the elimination of illumination

Table 1. *Chemical composition of the steels under study*
Таблица 1. *Химический состав исследуемых сталей*

Steel	Element content, mass fraction, %										
	C	Mn	Si	S	P	Cr	Ni	Mo	V	Nb	N
1	0.052	0.466	0.150	0.002	0.005	0.054	0.091	0.009	0.099	0.002	0.022
2	0.054	0.653	0.214	0.001	0.005	0.067	0.104	0.009	0.044	0.024	0.007
3	0.049	1.090	0.248	0.001	0.005	0.244	0.076	0.095	0.002	0.033	0.008
4	0.049	0.920	0.230	0.001	0.004	0.651	0.071	0.008	0.004	0.040	0.007
5	0.044	0.635	0.258	0.001	0.003	0.538	0.070	0.009	0.044	0.025	0.007

heterogeneity of various natures and subsequent analysis of the brightness field to determine the binarization threshold. Illumination heterogeneity was eliminated using a method based on subtracting the 1st or 2nd degree surface from the original 3D distribution of image luminance intensities. The binarization algorithm included the construction of distribution histograms of intensities of the halftone image pixels $f(x,y)$. Dark objects were identified against a light background by determining the value of T threshold, which delimits the brightness distribution modes. Any point in the image with coordinates x_i and y_i, at which $f(x_i,y_i)>T$, was called the background, points with lower brightness were called the object. Geometric parameters were calculated according to the four-connection principle; boundary objects were not taken into account.

The authors calculated the following NMI parameters: density, volume fraction and average area, as well as the skewness and kurtosis coefficients of area distribution. To identify the “object – noise” threshold value, the authors relied on the parameters of the equipment used, and the nature of the object. For this purpose, in particular, the filtering threshold was varied, sequentially removing from the primary image the inclusions with an area of less than 5, 10, 25, 50, 75, and 100 μm^2, respectively. To assess the NMI location heterogeneity, the construction of Voronoi polyhedra [18] based on the method of perpendicular bisectors was proposed. After constructing the polyhedra and determining the nearest neighbors, the distances between the centers of objects (NMI), the areas of the polyhedra and the number of their nearest neighbors, as well as the kurtosis and skewness coefficients, of the distributions of the obtained values were calculated.

RESULTS

The variation of the threshold filter led to the following changes in the values of the NMI geometry parameters: a natural decrease in the density and volume fraction of particles, an increase in the average values of their area for the five steels under study (Fig. 1). It was identified that at high filter values, the difference in the density of the NMIs of the studied samples is leveled out. The difference in the density of large inclusions (from 100 μm^2 and above) for all samples was minimal – 29 pcs/mm^2, and for all inclusions (without filtering) – 218 pcs/mm^2.

The results of the NMI quantitative analysis were compared to the acceptance properties of steels. The results of tensile and corrosion resistance tests are given in Table 2.

The sheets studied corresponded to K52 and K55 strength classes, the scatter of strength and plastic parameters was insignificant. However, the corrosion resistance of the samples differed by a factor of 3. Fig. 2 shows the relationship between the NMI density, and the corrosion resistance of the steels under study, taking into account different levels of noise filtration.

Fig. 3 shows the dependences of Voronoi polyhedra parameters on the corrosion resistance values of the samples under study, from which it follows that these characteristics significantly correlate with each other.

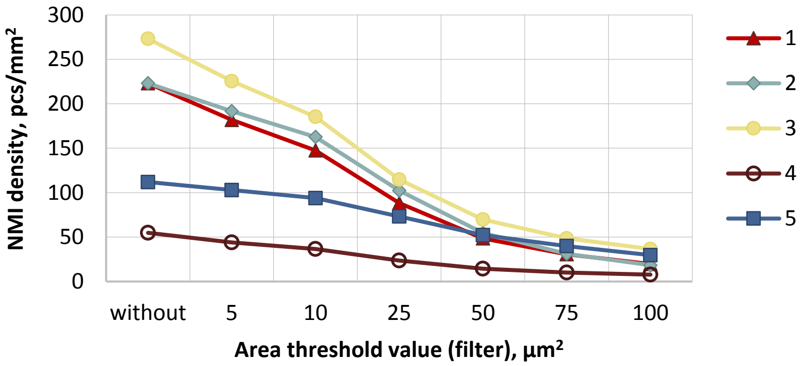
Steels 4 and 5 have the lowest density and more uniform distribution of NMIs, and as a consequence, the best corrosion resistance. In turn, for steels 1, 2 and 3, the statistics of Voronoi polyhedra do not differ, but for steels 2 and 3, a correlation with the NMI density was revealed. For steel 1, no significant relationship was found between the NMI density and corrosion resistance.

Table 3 presents the results of the quantitative assessment and chemical composition of NMI using scanning microscopy.

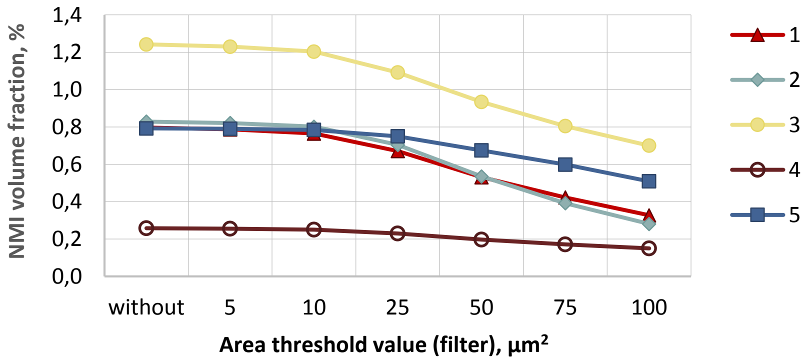
Fig. 4 shows images of nanosized particles obtained using transmission microscopy.

DISCUSSION

The change in the characteristics of steel contamination with inclusions with increasing filtration threshold (Fig. 1), is associated with the removal of small objects, the number of which prevails over large ones. Fig. 2 shows that the greatest strength of the relationship between corrosion resistance and NMI density (correlation coefficient is 0.95), is observed in the absence of their filtration. Increasing the filtration threshold and the accompanying screening out of small inclusions leads to a decrease in the correlation coefficient. This indicates that corrosion resistance is mainly affected by small inclusions (<10 μm^2). One should



a



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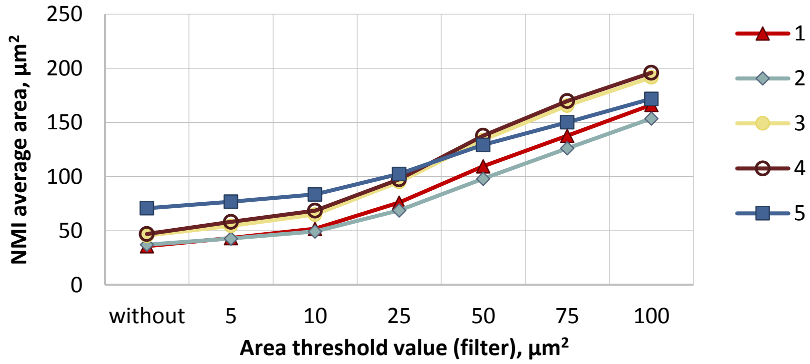


c

Fig. 1. *Statistical parameters of NMIs of the steels under study at different filtration levels:*
a *– density;* ***b*** *– volume fraction;* ***c*** *– average area*

Рис. 1. *Статистические параметры НВ изучаемых сталей при разных уровнях фильтрации:*
a *– плотность;* ***b*** *– объемная доля;* ***c*** *– средняя площадь*

Table 2. *Mechanical and corrosive properties of the steels under study*
Таблица 2. *Механические и коррозионные свойства исследуемых сталей*

Steel	Yield strength, MPa	Tensile strength, MPa	Ultimate elongation, %	Corrosion rate, mm/year
1	441.0	510.9	24.9	1.67
2	413.8	504.4	25.9	1.35
3	469.8	547.4	21.5	1.57
4	454.0	556.2	21.5	0.59
5	445.7	524.7	26.3	0.75

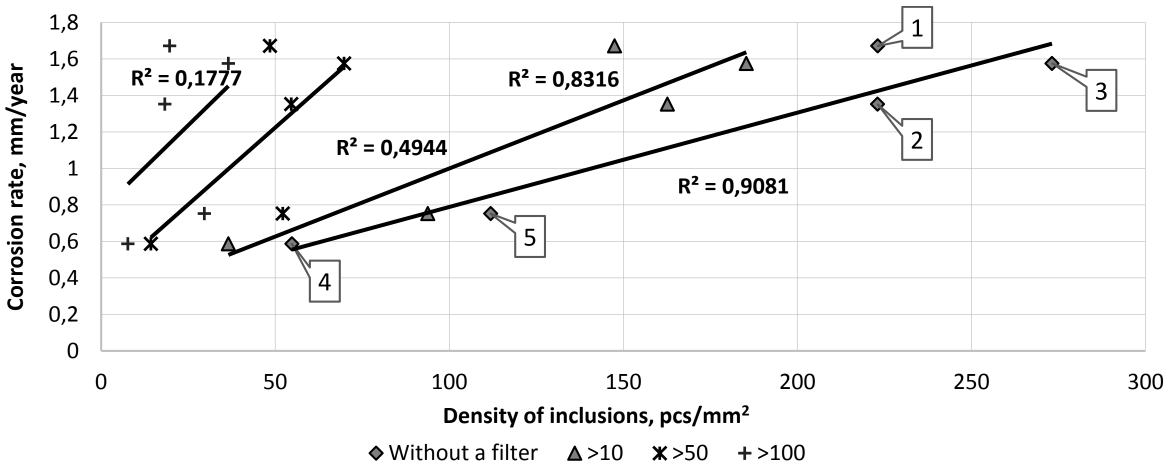


Fig. 2. *Correlation of NMI density and corrosion rate in the steels under study when varying the inclusion filtration level (by their area)*
Рис. 2. *Соотношение плотности НВ и скорости коррозии в исследуемых сталях при вариации уровня фильтрации включений (по величине их площади)*

consider this fact during "noise – object" filtering, since an incorrect choice of threshold can lead to the loss of a significant part of the information. Despite the fact that the volume fraction of small NMIs is low, they have a higher density compared to large ones. In this regard, there is an increased likelihood that small inclusions will form clusters, which, in turn, will contribute to a decrease in the corrosion resistance of steels.

It is unlikely that the absence of a significant relationship between the NMI density and corrosion resistance for steel 1 may be associated with the chemical composition of inclusions and the release of nanosized particles. Therefore, further analysis of the contamination of steels with NMIs was carried out using scanning and transmission microscopy. Microanalysis of inclusions using SEM showed that, unlike other steels under study, steel 1 is distinguished by a high density of complex NMIs based on aluminum-magnesium spinel and a high Al/(Mg+Ca) ratio. It is known [19] that with an increase in the proportion of the Al_2O_3 phase in a complex NMI, the tendency to the appearance of microfractures around the NMIs increases, which leads to a decrease in the corrosion resistance of steel. In turn, modifying steel with calcium allows giving a globular shape to such inclusions and thereby reduce their negative impact [20].

Transmission electron microscopy showed that steel 1 contains a large number of nanosized carbonitride particles. The belonging of particles to one type or another was assessed by the nature of the mutual arrangement of the particles, the presence of a regular orientation correspondence between the particles, and the ferrite matrix, and by the shape of reflections of the particles in the microdiffraction patterns. Nanoparticles were detected in almost all areas of the sample tested for their presence. Particles of all known types were observed, most of all particles were of the interphase/mixed type; their volume density was high (Fig. 4). Thus, the presence in steel 1 of nanoparticles of a different (than in steels 2–5) nature, determined the deviation from the found general patterns reflecting the relationship between the NMI density and corrosion resistance.

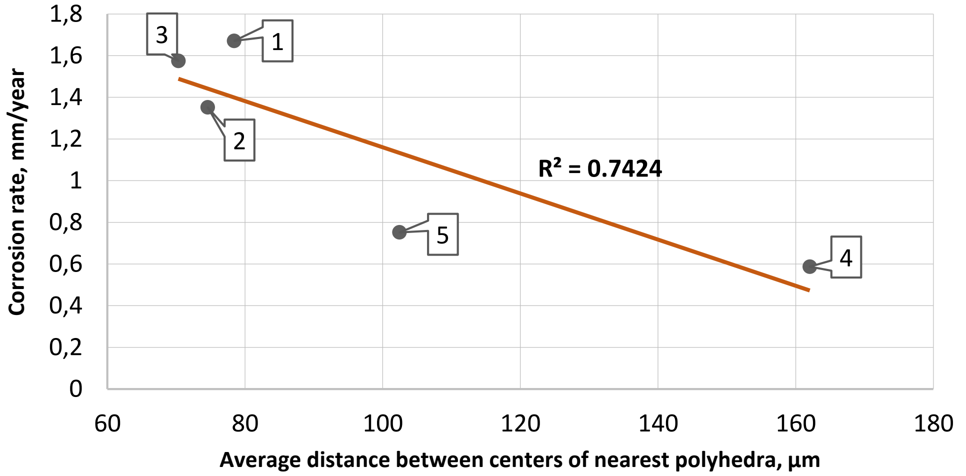
The results of the work showed the importance of an integrated approach to the study of the morphology of nonmetallic inclusions. This involves the combination of a physically supported image-processing algorithm to get ideas about the geometry of inclusions, and the patterns of their arrangement in the volume of metal with an analysis of their composition using transmission and scanning microscopy, including submicroscopic and nanoscale observations. It appears to be essential for understanding the differences in corrosion resistance of oilfield steels.

CONCLUSIONS

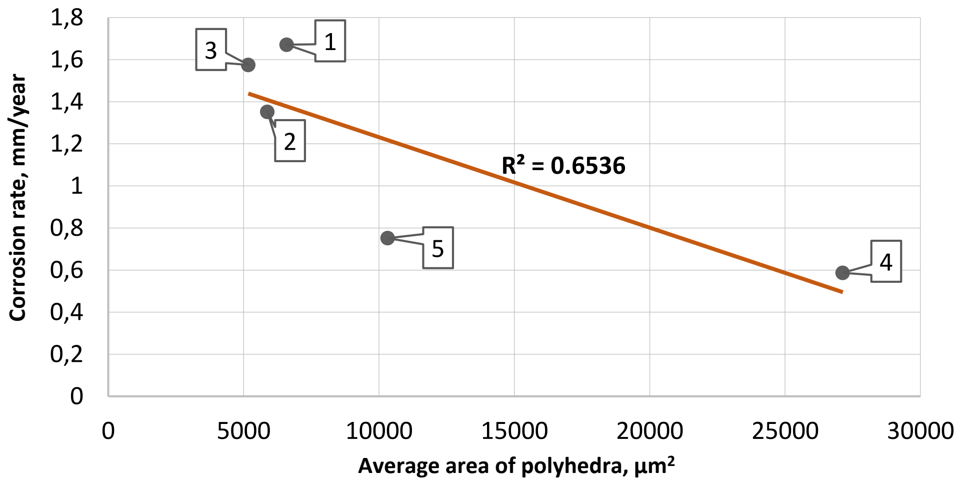
1. The authors proposed a method for processing panoramic images of NMIs (on a metallographic specimen) based on an analysis of the patterns of their brightness fields (in 256 shades of gray) to compile reasonable algorithms for illumination, equalization and binarization of images, which is necessary to obtain metrologically assured results. The study shows the importance of justifying the choice of filtering threshold for objects of noise nature, to obtain significant results.

2. The study shows the effectiveness of using the statistics of Voronoi polyhedra to assess the heterogeneity of NMIs location, which includes obtaining statistical estimates of the type of distribution of parameter values.

3. Comparing the results of corrosion tests, and quantitative assessment of the NMIs of samples of the steels under study, the influence of density, values of the polyhedron area distribution kurtosis coefficient, and the average distance between the centers of inclusions (in the size range from 2 nm^2 to 10 μm^2) on corrosion resistance was discovered. The coefficients of determination of linear dependencies



a



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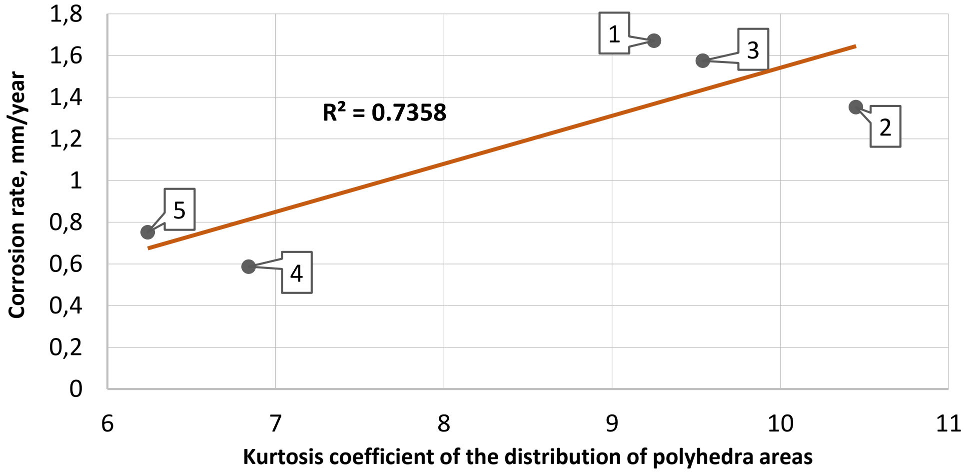
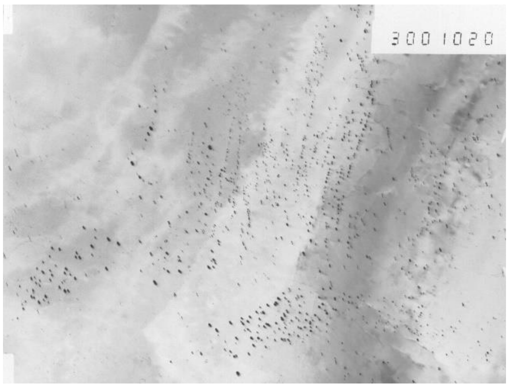


c

Fig. 3. *Correlation of the results of determining the NMI geometry (according to the Voronoi polyhedra statistics) and the corrosion rate:*
a *– average distance between centers;* ***b*** *– average area of polyhedra;*
c *– kurtosis coefficient of the distribution of polyhedra areas*
Рис. 3. *Соотношение результатов определения геометрии НВ (по статистике полиэдров Вороного) и скорости коррозии:*
a *– среднее расстояние между центрами;* ***b*** *– средняя площадь полиэдров;*
c *– коэффициент эксцесса распределения площадей полиэдров*

Table 3. *The results of NMI analysis using scanning microscopy*
Таблица 3. *Результаты анализа НВ методом сканирующей микроскопии*

Steel	Area of analyzed surface of a metallographic specimen, mm^2	NMI density, pcs/mm^2	Chemical element average content in inclusions, wt. %				Al/(Mg+Ca)
			Mg	Al	S	Ca	
1	3.79	51	3.75	10.60	2.95	6.04	1.18
2	4.54	37	6.73	6.95	2.73	7.14	0.53
3	3.57	48	3.40	3.11	1.32	8.38	0.32
4	3.90	35	4.03	7.92	5.48	4.60	0.97
5	3.82	43	4.17	3.00	3.11	9.19	0.35



a ***b***

Fig. 4. *Nanosize particles of carbonitrides in the steel 1:*
a *– formed according to the interphase mechanism;* ***b*** *– formed in ferrite*
Рис. 4. *Наноразмерные частицы карбонитридов в стали 1:*
a *– сформировавшиеся по межфазному механизму;* ***b*** *– образовавшиеся в феррите*

were 0.91, 0.74, and 0.74, respectively. At the same time, the influence of inclusions on the fracture resistance was not revealed, which can be explained by the absence of differences in the contamination of steels with larger NMIs playing a decisive role in fracture.

4. Using SEM methods, the composition of the NMIs was revealed – predominantly aluminum-magnesium spinel; using TEM methods, the authors identified the presence of a large number of nanosized particles having a significant impact on the development of the corrosion rate of the steels under study.

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Цифровые измерения неметаллических включений в стали

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Аннотация: Опыт многолетних исследований показал, что существенно повысить коррозионную стойкость сталей нефтепромысловых трубопроводов и увеличить сроки их безаварийной эксплуатации можно, оптимизируя химический состав и микроструктурные особенности стали, а также понижая ее загрязненность неметаллическими включениями (НВ). Влияние комплексных НВ на коррозионную стойкость стали обусловлено как химическим составом НВ, так и их количественных соотношением. Поэтому получение металлопродукции требуемого качества возможно только с применением принципа «управления по структуре». В работе на основе анализа полей яркости изображений (в масштабе образцов) в 256 оттенках серого предложены цифровые метрологически обеспеченные процедуры измерения неоднородности НВ низкоуглеродистых сталей нефтепромыслового назначения, такие как устранение неоднородности освещения поля зрения, обоснование критериев бинаризации и фильтрации шумов. Для низкоуглеродистых сталей различной выплавки выявлена ключевая роль дисперсных неметаллических включений размером от 5–10 мкм^2 до 2 нм^2 в формировании коррозионной стойкости сталей. Это может объяснить, почему в ряде случаев отсутствует взаимосвязь между скоростью коррозии и сопротивлением сталей разрушению, в формировании которого определяющее влияние оказывают частицы большего размера. В представлении НВ как множества случайных точек на плоскости распределение расстояний между ближайшими из них оценено на основе статистики полиэдров Вороного. Показано, что повышению коэффициента эксцесса распределений площадей полиэдров сопутствует увеличение скорости коррозии исследуемых сталей. Это указывает на отрицательное влияние неоднородности в размещении дисперсных НВ на коррозионную стойкость сталей.

Ключевые слова: цифровые измерения структур; управление качеством по структуре; неметаллические включения в стали; коррозионная стойкость нефтепромысловых труб.

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