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Morphological changes of deformed structural steel surface in corrosive environment

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ABSTRACT

Introduction. Internal factors, including phase heterogeneity, crystallographic texture, residual stress amplitude and the concentration of non-metallic inclusions, exert a nonlinear, multi-parametric effect on the corrosion resistance of metallic systems in aggressive environments. This complex interaction significantly complicates the prediction of corrosion degradation kinetics and the assessment of the operational life of metal structures. These parameters modulate the spatial distribution of corrosion defects, their morphology and penetration depth, necessitating a systematic approach to establish quantitative correlations. To gain a more accurate understanding and account for the influence of internal factors on the corrosion process, comprehensive research and analysis are required. The use of mathematical processing methods in the analysis of influence will reveal stronger regularities applicable to the process of corrosion damage. This will enable the development of methods and approaches for optimizing the design, production and operation of metal structures and products, as well as improving their reliability and durability. **Purpose of work** is to develop a multi-criteria model linking the depth of corrosion damage (an integral indicator of environmental aggressiveness) with microstructural, mechanical, and topographic characteristics of low-carbon steel *St3*. The objects of the study are samples from rolled sheet metal with varying degrees of residual plastic deformation (ε = 0–7%). **Methods of investigation.** Grain size, texture, and dislocation density were assessed through microstructural analysis using optical microscopy (*Olympus GX53*) and scanning electron microscopy (*JEOL 6008A*). Quantitative morphometry of corrosion damage was performed using digital image analysis (*AXALIT* software), with median depth determined as a key parameter. X-ray diffraction analysis of residual stresses was implemented to construct tensor stress fields. **Results and discussion.** Experimental data demonstrates a non-linear increase of the median depth of corrosion damage with the degree of deformation: at ε = 6.6%, a twofold increase in the median depth is observed compared to the undeformed state. Multivariate regression analysis revealed the dominant influence of internal residual stresses on the kinetics of the corrosion damage process (R^2 = 0.89). The scatter of the determined values for internal stresses is ±5 μm. The observed regularities are associated with the behavior of the material structure during plastic deformation, which occurs most significantly in the {111} <110> directions, leading to the generation of reverse residual stresses. The median depth of corrosion damage reflects the rate of corrosion. The group method of data handling (*GMDH*) allowed for the synthesis a complex parameter combining various parameters of steel structure. Polynomial approximation of the dependence of the median depth of corrosion damage in 5% *HCl* on the complex parameter shows high convergence (R^2 = 0.99) with a determination error of ±1 μm. The developed model confirms that residual stresses are one of the key factors modulating the corrosion activity of deformed *St3* steel. The results obtained allow for the optimization of cold treatment of steel to increase the corrosion resistance of metal structures. Further studies are planned to focus on the influence of dynamic loads and temperature gradients on the evolution of dislocation substructures.

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Introduction

The corrosion failure of steel is a complex process influenced by both external and internal factors. For example, the material structure determines the susceptibility of steel to the corrosion process [1–3]. Thus, it is shown in [4] that ferrite and austenite are more electronegative than cementite. The close location of phases with different electric potentials in the material structure leads to the appearance of microgalvanic couples and an intensive destruction process in an aggressive environment.

The presence of defects in the phases accelerates the corrosion damage of the material. In the steel structure, one of the phases with a high degree of defectivity is martensite [5–7].

The process of corrosion fracture is also affected by the homogeneity of the material surface [7–11]. It is especially important for steels with a low content of alloying elements [7, 12].

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Studies have shown that grain size also affects corrosion [1, 13–14]. This is usually due to the fact that grain boundaries attract crystal structure defects and inclusion atoms [6–7, 15–16].

Pitting corrosion of steel can be observed when impurity elements are present in the structure [7, 17–19]. The impurities can form chemical compounds that increase the activity of the material in the corrosive environment [7, 20–21] due to their electrochemical heterogeneity [6].

It has been demonstrated in studies [22, 25–26] that there is a direct correlation between the magnitude of internal residual stresses and the corrosion rate of structural steel. Internal stresses can reach values that exceed the yield strength of the material. This leads to plastic deformation and an increase in the number of linear defects in the form of dislocations [22, 25–29].

Residual deformation of the material under the influence of external loads increases the anisotropy of grains, which affects the rate of the corrosion process [3, 22].

The presented data from scientific studies confirm the existence of a multiparametric dependence of corrosion processes on exogenous and endogenous factors, including the crystal structure of the material, the degree of phase homogeneity, the morphology of the surface layer, the presence of foreign chemical elements in the alloy matrix, as well as dislocation and boundary-defect formations. Numerous experimental works [23–24] have contributed to the systematization of key aspects of the kinetics and thermodynamics of corrosion phenomena, which creates a theoretical basis for identifying factors determining material degradation under conditions of a specific operating environment.

The purpose of this work is to develop a multi-criteria model linking the depth of corrosion damage (integral index of aggressiveness of the environment) with microstructural, mechanical and topographic characteristics of low-carbon steel *St3*. The subject of the study are samples cut from rolled steel sheets with varying degrees of residual plastic deformation (**ε** = 0–7 %).

It is possible to use such an approach by relying on mathematical methods that allow taking into account the influence of various factors, for example, by using a group method of data handling (*GMDH*).

The objectives of the study are:

– to study quantitative relationships between the depth of corrosion damage to steel, the magnitude of residual internal stresses, grain size anisotropy, and their number in an aggressive environment;

– to investigate the influence of plastic deformation (not exceeding 7 %) on the kinetics of corrosion processes, with emphasis on the change in the depth of damage and the role of residual stresses;

– to analyze possible crystallographic mechanisms determining the correlation between corrosion depth and residual stresses;

– to develop a physical and mathematical model describing the dependence of corrosion kinetics on structural and morphological parameters, taking into account strain anisotropy and dislocation dynamics using a group method of data handling (*GMDH*).

Methods

In this study, samples were taken from *St3* steel sheet stock, which was used in its initial state. The samples (4.0 × 70.0 × 25.0 mm) were prepared with their longitudinal axis perpendicular to the rolling direction of the steel.

The paper [22] presents data on the magnitude of internal stresses, degree of grain anisotropy, corrosion rate of the studied samples in a 5 % hydrochloric acid solution, and also reflects the methodology for determining these parameters.

The investigated samples had different values of residual strain (Table).

The microstructure of the investigated samples obtained on an optical microscope is shown in Fig. 1.

Sample number and corresponding residual strain value

Sample No.	1	2	3	4	5
ε, %	0	1.5	3.0	4.5	6.6

The study of the microstructure performed in [22] revealed that it consists of a ferrite-perlite mixture. It contains 81.7 % ferrite and 18.3 % pearlite. According to *GOST 8233*, the structure rating is 8. The minimum grain size rating is 8, the maximum grain size rating is 13, and the most frequently observed grain size rating on the micrographs is 11.

The study of corrosion damage of the investigated samples was carried out on a *JEOL 6008A* scanning electron microscope (Fig. 2). The corrosion products of the material were removed from the surface before the study.

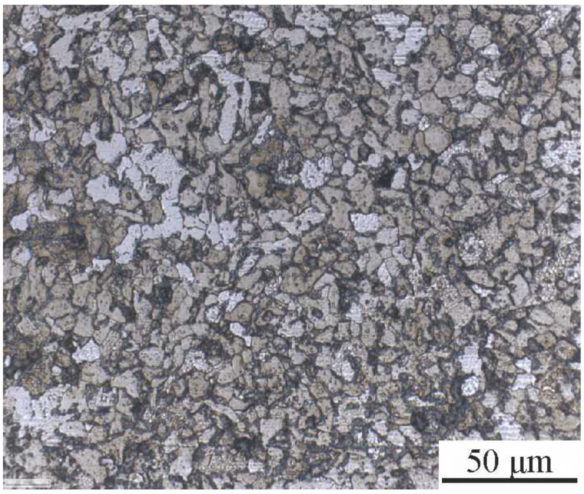


Fig. 1. Microstructure of undeformed *St3* steel sample

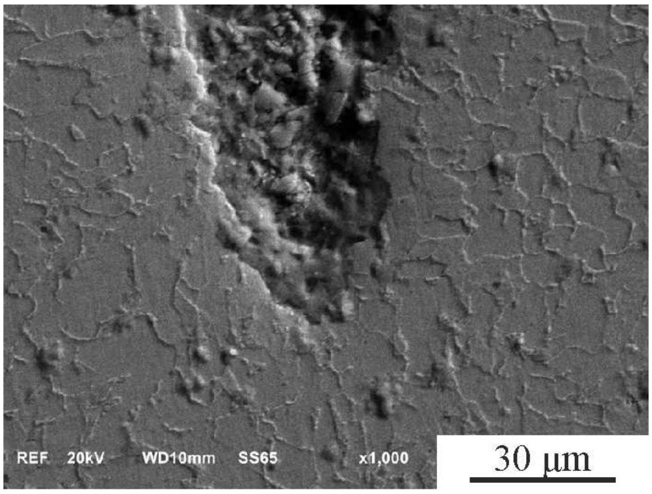


Fig. 2. Structure of sample No. 1 of *St3* steel

Determination of the geometric parameters of the polished microsection surface was performed on images acquired using a scanning electron microscope (within the scanning area) with *AXALIT* software [30]. The values of the damage depth distribution were set in the software. Using the color gamut of the image, the depth of the observed damage was determined. Image scaling was performed in *AXALIT* to allow more accurate measurement results.

The measurement was performed by delineating the defect contour, after which the software automatically calculated the area in μm². Depth variation was performed along a straight line, with the software counting darker areas of the image as deeper. The graph obtained in this way represents a profilogram of the surface along the selected direction. The constructed graphs determine the dependence of the depth in μm (determined by brightness) on the coordinate of the line position on the sample (μm).

The methodology for calculating the depth of corrosion damage is as follows. The values of the corrosion damage depth obtained by measurements along a straight line (profilogram) on the base image surface were used to find the median value.

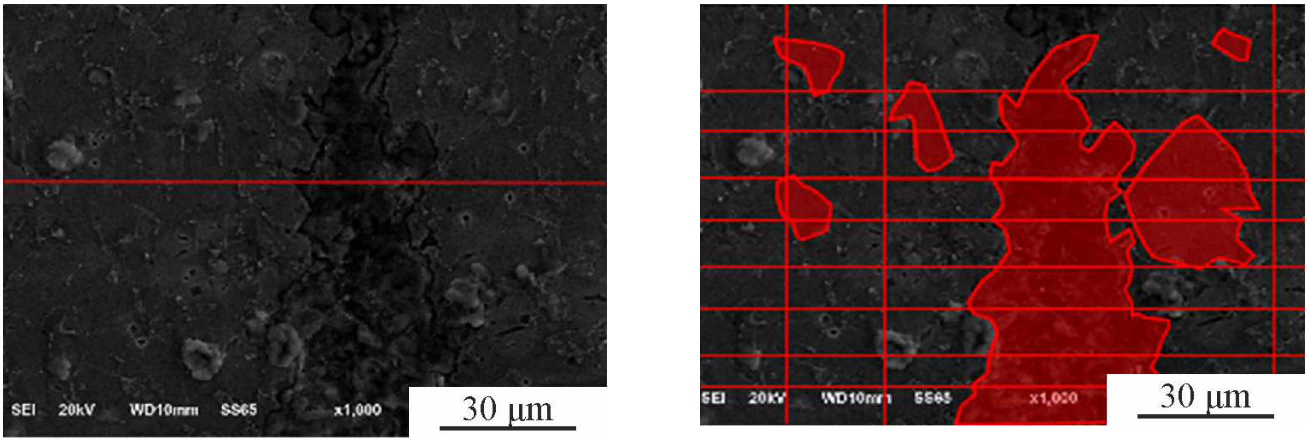
Results and discussion

Fig. 3 shows an example of the surface image of the investigated sample No. 2, obtained on a scanning electron microscope and analyzed in *AXALIT* software.

An example of a profilogram is shown in Fig. 4.

According to the obtained depth values from several measurements, the median corrosion damage depth values were determined for each sample. Determination of the median corrosion damage depth of the material under study was based on the following considerations:

– the sample surface exhibits significant variations in height, even in areas without apparent corrosion damage (variations can reach 45 μm). this is attributed to the fact that the material under investigation is a multiphase system, in which different components react differently to the aggressive environment. furthermore, the grain boundaries are highly heterogeneous due to the high concentration of crystalline defects;



a *b*

Fig. 3. Microstructure of Sample No. 2:

a – profile line location; *b* – highlighted area of corrosion damage)

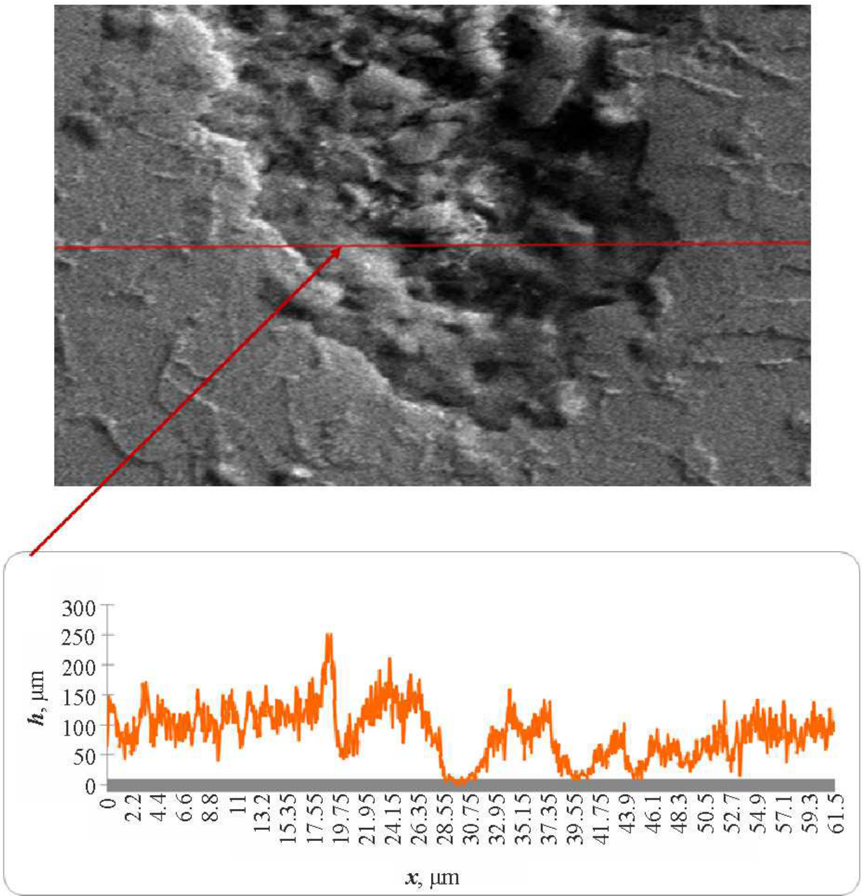


Fig. 4. Profilogram obtained from the micrograph of Sample No. 1 using *AXALIT* software

– material destruction occurs non-uniformly, even within a localized corrosion site. this is attributed to the varying rates of the corrosion process and the slower diffusion processes in the material-electrolyte system;

– the median value represents the average value within the considered sample set, rather than averaging the obtained measurement results.

The dependence of the median value of the corrosion damage depth on the value of residual strain is presented in Fig. 5.

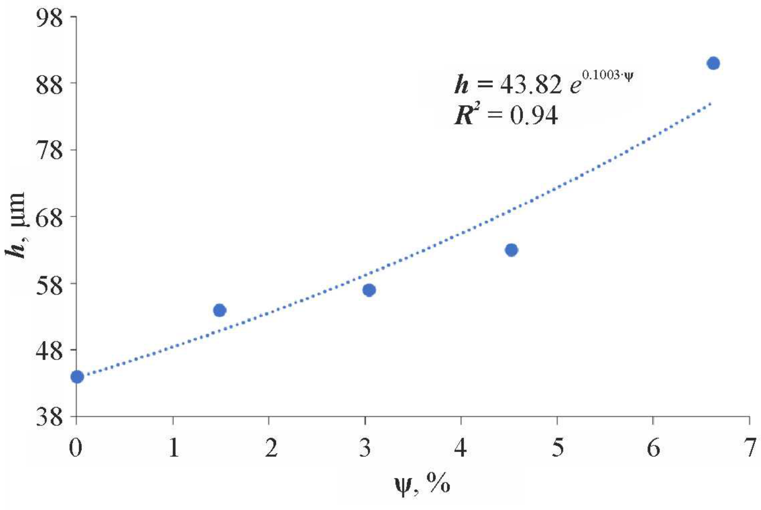


Fig. 5. Median depth of corrosion damage as a function of residual strain in the material

From the samples after corrosion tests, cross sections were additionally made. Microphotographs were taken, and the distance from the surface of the sample to the upper forming surface at the points of corrosion damage registration was measured. These measurements are presented in Fig. 6, where there is a direct measurement of the corrosion damage depth. The distance from the surface of the sample to the upper

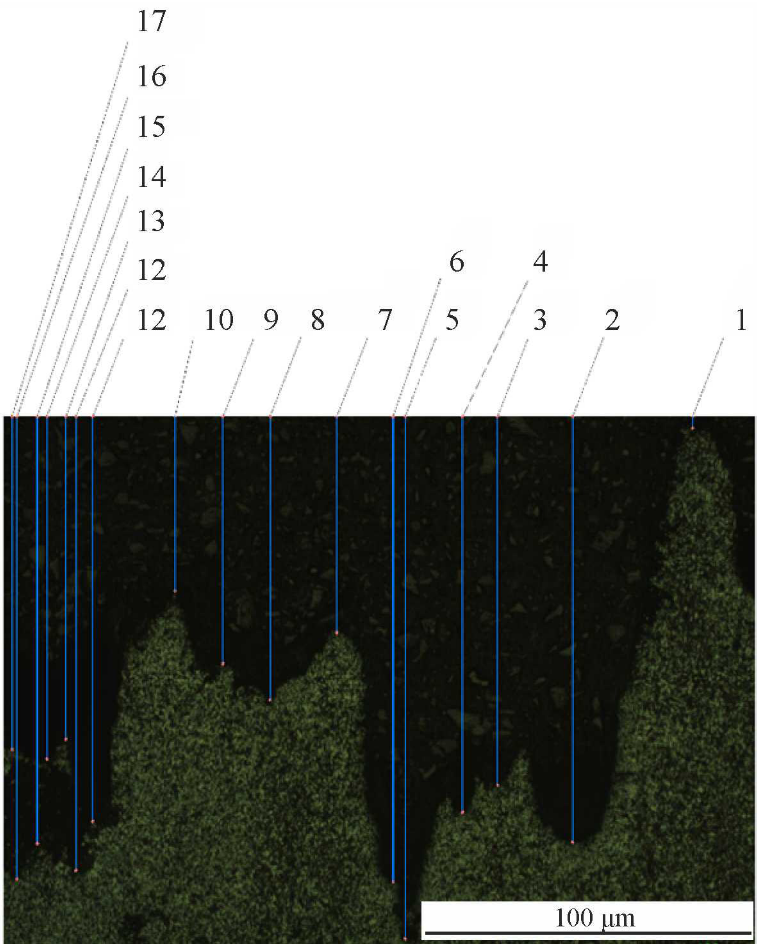


Fig. 6. Micrograph of a cross-section prepared from Sample No. 3 after corrosion testing, showing the locations where corrosion damage depth was measured

forming surface at the points of corrosion damage registration is shown in Fig. 6 in the form of blue lines. According to the measurements obtained, the median depth of corrosion damage was determined.

A comparison of the results of the median depth measurement from the cross-section micrograph and from the surface photograph in the *AXALIT* software is shown in Fig. 7.

As can be seen from Fig. 7, the results of determining the median depth of corrosion damage using *AXALIT* software are satisfactory. The obtained dependences of the change in median corrosion damage depth on the residual strain of the material exhibit a similar trend in both cases, indicating a consistency between the results.

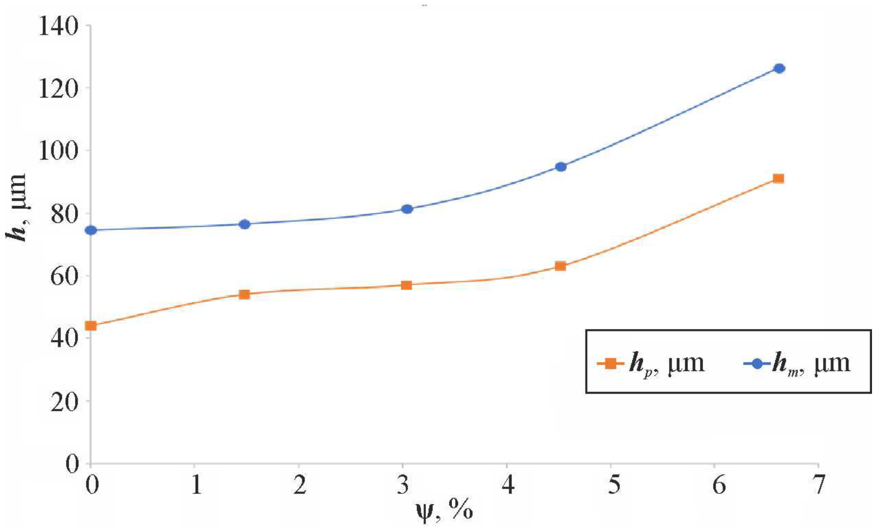


Fig. 7. Median depth of corrosion damage at varying levels of residual strain:

h_p – results obtained using *AXALIT* software; h_m – results obtained from cross-sectional analysis

The results from direct measurement and those obtained using the software differ by a factor of 1.45, as shown in Fig. 8. However, the median corrosion damage depths obtained by the two methods are directly correlated, as evidenced by the linear dependence presented in Fig. 8. The coefficient of determination (R^2) for the obtained relationship is 0.91, further indicating a strong agreement between the results.

The data observed in Figs. 5, 7, and 8 indicate that during plastic deformation, there is an increase in the penetration depth of corrosion damage into the material. This is because the material fracture process initiates at surface micro-inhomogeneities, which can be considered as dislocations and atoms of chemical elements. Plastic deformation of the material leads to an increase in dislocation density [22, 25-27].

During material deformation, dislocations move due to the slip process. During movement, dislocations collide with grain boundaries, which act as obstacles. Accumulation of dislocations occurs in the grain boundary regions.

Plastic deformation leads to the generation of new dislocations and an increase in dislocation collisions. This results in the formation of dislocation clusters that are unable to move through the crystal lattice. Together with impurity atoms diffusing into the grain boundaries, this intensifies the corrosion fracture process. In this case, the grain boundaries are the initiation sites for this process (Fig. 9).

Restriction of dislocation movement and formation of a more extensive cluster leads to hardening of the material [20], which affects the grain shape and its average size.

The average size is in direct relation to the number of grains in the microstructure, and it affects the extent of grain boundaries. The greater the number of grains, the greater the probability of accumulation of crystal structure defects in these areas and the higher the corrosion susceptibility of the material. However, other parameters of the system under consideration also change during deformation.

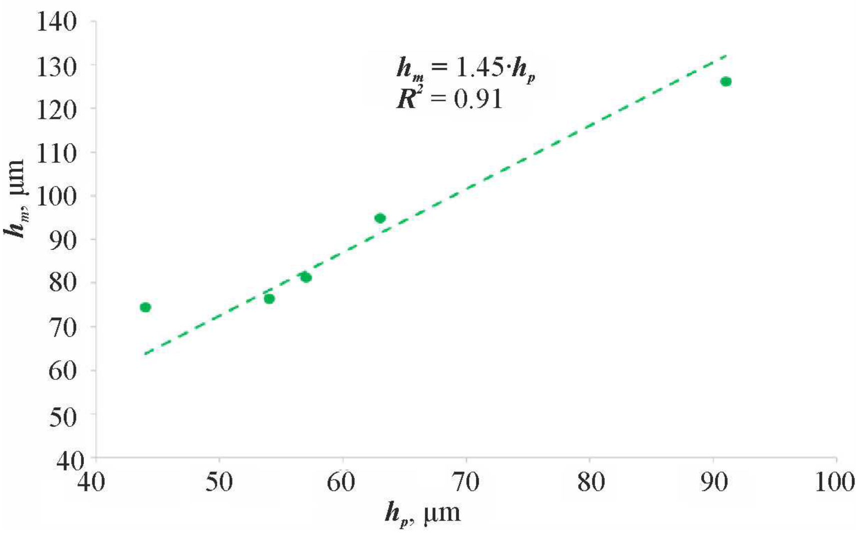


Fig. 8. Relationship between median corrosion damage depth $\boldsymbol{h_m}$ and $\boldsymbol{h_p}$

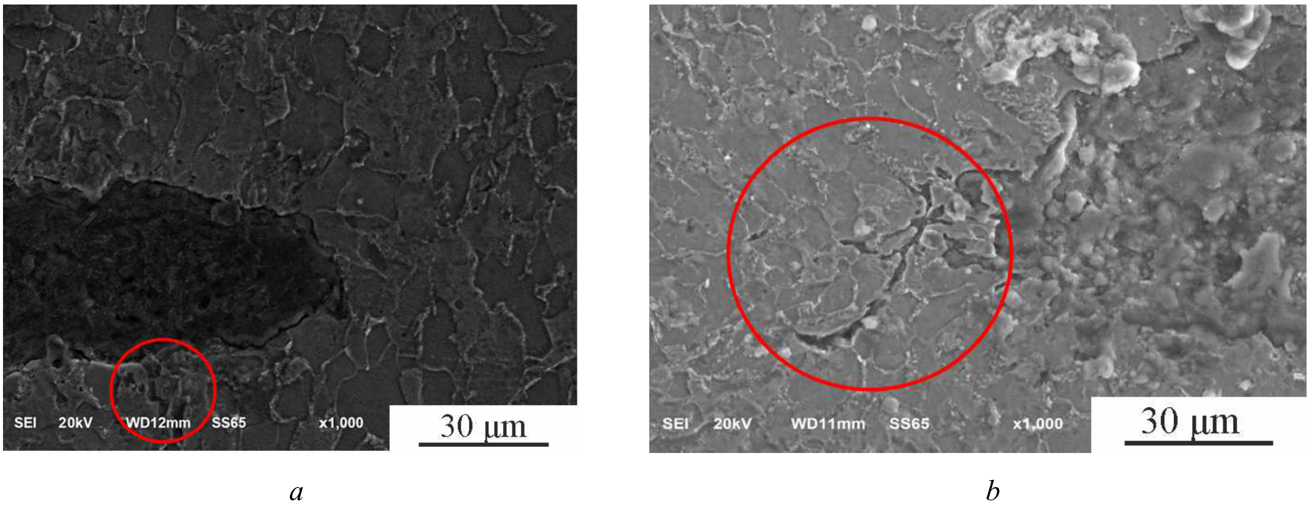


Fig. 9. Microstructure of Sample No. 4 (*a*) and Sample No. 5 (*b*), showing the highlighted area of corrosion initiation

The grain shape can be evaluated by its degree of anisotropy, which increases with plastic deformation of the material [22]. Plastic deformation in a phase with a body-centered cubic (*BCC*) lattice most commonly leads to slip along crystallographic planes and directions {110} <111> [31]. This accounts for the described process.

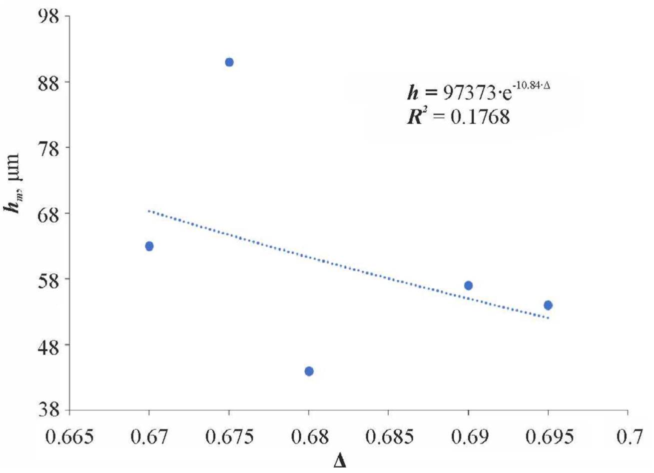
Due to the change in grain shape, there is a decrease in internal residual stresses as a consequence of the appearance of stresses of opposite sign [28]. The resulting anisotropy of texture and material properties affects the corrosion fracture process (Fig. 10).

The correlation between corrosion processes and internal stresses, arising during plastic deformation, is due to the modification of the defect substructure of crystalline material, realized through the activation of dislocation dynamics. This mechanism involves the coordinated slip of linear defects in the crystal lattice along preferred slip systems, determined by the crystallographic configuration with maximum atomic packing density, which minimizes the activation energy for shear processes [21, 32-33].

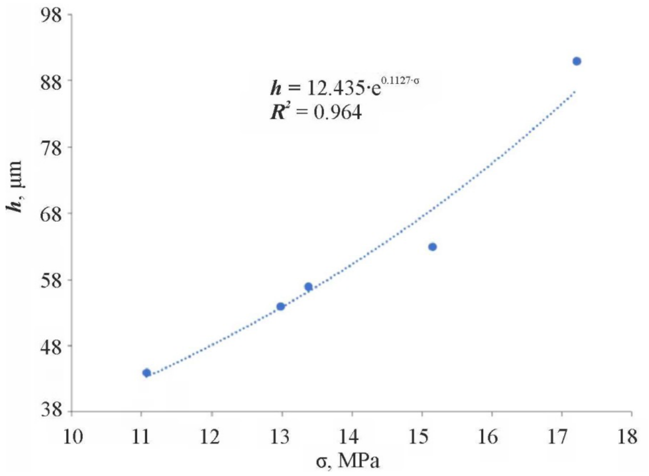
Plastic deformation induces directed migration of dislocations, accompanied by their interaction within the bulk of the material, including annihilation upon encountering dislocations of opposite sign, as well as the formation of stabilized configurations (dislocation walls, networks) [21, 32-33].

These structural transformations modulate local electrochemical potentials, creating regions of enhanced reactivity, which catalyzes corrosion processes, as seen in Fig. 11. This occurs due to the following factors:

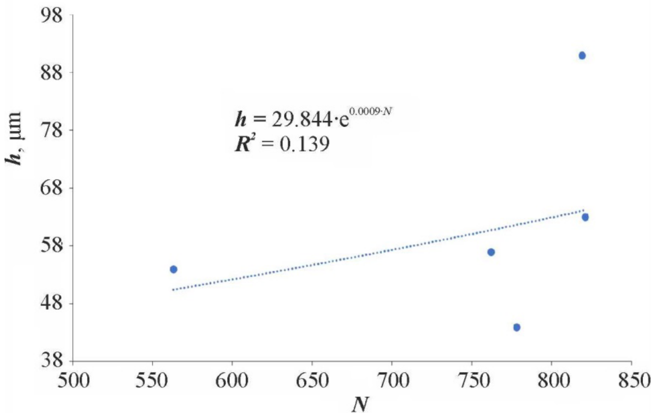
– formation of microgalvanic couples between deformed and undeformed regions;



a



b



c

Fig. 10. Dependence of median corrosion damage depth on:

a – degree of grain anisotropy; *b* – value of residual stresses; *c* – grain count in the material structure

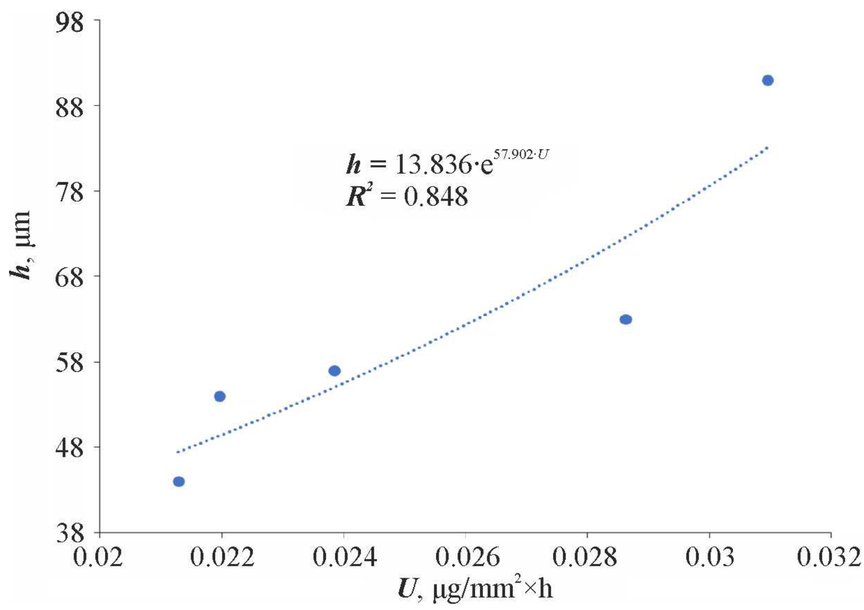


Fig. 11. Dependence of median corrosion damage depth on corrosion rate of *St3* steel samples at varying residual strain values

– intensification of reagent diffusion through dislocation channels;

– accumulation of residual stresses, which lowers the energy barrier for oxidation reactions.

The above data show that the depth of corrosion damage depends on many internal factors. Estimating it using a single characteristic can yield both positive and negative results.

However, when estimating multi-dependent parameters, it is possible to use methods that allow for a comprehensive determination of the influence of the considered values on the desired outcome. One such method is the group method of data handling (*GMDH*) [34-36]. The *GMDH* method does not perform an exhaustive search of all possible models. However, with a sufficient amount of initial data, it allows us to find the optimal solution, represented as a complex parameter that exhibits the best correlation with the considered value.

Using the *GMDH* method, a complex parameter closely related to the corrosion damage depth of the studied material after plastic deformation was determined. This parameter is expressed by the following equation:

$$P_2 = k_1 \cdot N + k_2 \cdot \sigma + k_3 \cdot \Delta \quad (1)$$

where ***N*** is the number of grains; **σ** is the internal residual stresses; **Δ** is the degree of grain anisotropy; ***k*** is the coefficient of mutual influence of the considered characteristics and the depth of corrosion damage.

The obtained complex criterion reflects the influence of internal material parameters on the corrosion damage depth during plastic deformation (Fig. 12). When considering individual parameters, a significant scatter is observed, which, even with a coefficient of determination greater than 0.9, makes it difficult to estimate the value of interest accurately. When the complex parameter is applied, the scatter of median depth values determined from the image is reduced to a range of ±1 μm, and the coefficient of determination approaches **1**.

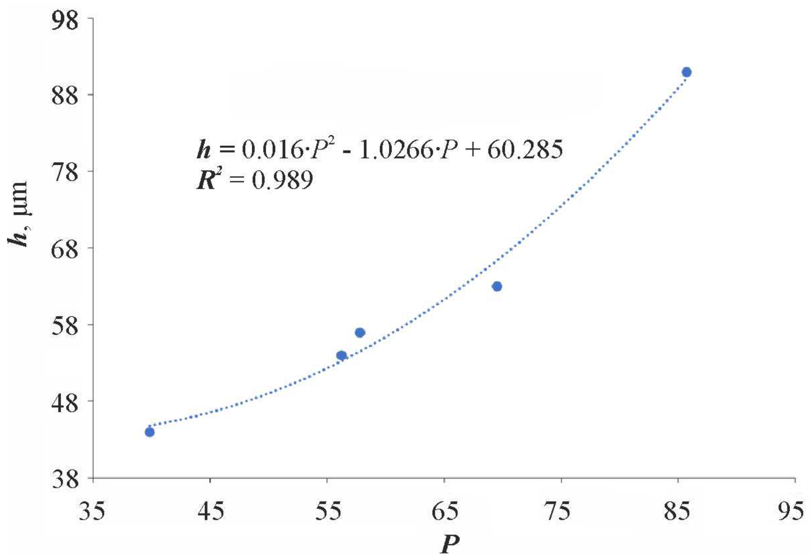


Fig. 12. Dependence of median corrosion damage depth on the complex parameter

Conclusion

– The study of the results obtained for *St3* steel after aging in a 5 % hydrochloric acid solution demonstrates a direct correlation between the depth of corrosion damage and internal residual stresses, as well as between the depth of corrosion damage and grain size anisotropy and the total number of grains.

– It was found that at a deformation of 6.6 % in the investigated material, the median depth of corrosion damage increases twofold. This is consistent with the data on the increased rate of corrosion of the material with increasing plastic deformation due to the increase in internal residual stresses.

– The highest statistically significant correlation (***R²*** > 0.85) was found in the system “median depth of corrosion damage - magnitude of residual stresses”, where the standard deviation for the latter parameter does not exceed ±5 μm at a 95 % confidence interval. This relationship is determined by the crystallographic anisotropy of plastic deformation, manifested by the preferential activation of {111}⟨110⟩ slip systems in materials with a *HCC* lattice. Selective dislocation mobility along these crystallographic planes is associated with the formation of gradient fields of residual compressive stresses and localized relaxation processes occurring through the formation of dislocation substructures such as walls and cells.

– The group method of data handling (*GMDH*) allows us to determine a complex parameter that reflects the median depth of corrosion damage of *St3* structural steel in a 5 % hydrochloric acid solution. This parameter takes into account the magnitude of residual internal stresses, grain anisotropy, and their number. It exhibits a high degree of correlation with the median depth of corrosion damage. The obtained dependence is described by a second-order polynomial equation, with a coefficient of determination ***R²*** ≈ 0.99 and a scatter in the determination of the median depth of ± 1 μm.

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Conflicts of Interest

The authors declare no conflict of interest.