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PHYSICAL REGULARITIES IN THE CRACKING OF NANOCOATINGS AND A METHOD FOR AN AUTOMATED DETERMINATION OF THE CRACK-NETWORK PARAMETERS

FIZIKALNE ZAKONITOSTI POKANJA NANOPREVLEK IN METODA ZA AVTOMATSKO DOLOČEVANJE PARAMETROV MREŽE RAZPOK

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The regularities and spatial distribution of multiple cracking of a nanocoating are investigated. It is found that in the cracking zones the relaxation of the stresses accumulated in the coating takes place; moreover, the intensity of its failure is determined by the structural level of defect accumulation. A new algorithm for a digital identification of the elements of a crack network in a nanocoating is proposed, and its adequacy is checked.

Keywords: multiple cracks, nanocoating, damage, diagnostics, surface, strain

Raziskovali smo zakonitosti in prostorsko porazdelitev več razpok na nanoprevleki. Ugotovili smo, da pride na področjih z razpokami do relaksacije napetosti, nakopičene v prevleki. Intenzivnost njenega propadanja je določena s strukturnim nivojem nakopičenja defektov. Predstavljen je nov algoritem za digitalno identifikacijo elementov mreže razpok na nanoprevleki. Preverili smo ustreznost modela.

Ključne besede: več razpok, nanoprevleka, poškodba, diagnoza, površina, raztezek

1 INTRODUCTION

The tensile, compressive and shear deformations play key roles in many processes of failure in a nanocoating because they are the results of a mutual influence of multiple defects and blocks of a coating[1,2]. The activation of deformation processes is observed in the sections of maximum coating cracking. Therefore, an important aspect is the quantitative evaluation of multiple cracking needed for the development of optimum algorithms for the identification and calculation of the parameters of a network of detected cracks[2]. However, there are a number of problems, in particular, a significant dispersion of the sizes of the crack-like defects, the shape of the cracks, and the need for determining a degree of their coalescence.

In the previous papers[3,4], the authors offered a number of algorithms for evaluating the technical condition of the surface of a metallurgical equipment affected by multiple defects. However, the use of these methods for an investigation of a nanocoating requires a combination of material-science approaches, physical and mechanical approaches that will allow an analysis of the hierarchical structure of multiple cracking zones, bringing together the entire volume of information on the processes of initiation, growth and coalescence of defects into a single system, and identifying the main regularities in this process.

In this paper, the structure of multiple cracking of a zirconium nanocoating, with a network of fatigue cracks, is investigated.

2 RESEARCH TECHNIQUE

The main objective of the paper is to find the basic regularities in the cracking of a nanocoating and to analyse the possibility of using a digital processing method for identifying defects. The requirement to determine the main regularities in the formation of the geometrical structure of a multiple cracking of a zirconium nanocoating with a thickness of 100 µm predetermined the need to use the approaches of digital identification. Ion nanostructuring of the surface layer of the specimens made of steel 25Kh1M1F was performed using the high-current, vacuum-arc source of metallic ions on the UVN-0.2 "Quant" setup[5]. After reaching a vacuum of at least $3 \cdot 10^{-3}$ Pa in the chamber, the specimens were treated with a zirconium-ion flow with the energy of 0.9–2.8 keV and the ion-current density of 0.1–0.3 mA/m^2. The duration of the treatment was from 5 min to 20 min. The substrate holder with the specimens fixed on the specimen stage is connected directly to the ion-acceleration schema instead of the traditional extraction of the radial-ion beam from the implanter.

In this case, the acceleration of ions occurs in the dynamic self-organising boundary layer featuring a double electrical layer, which forms around the specimen surface under the negative potential[5].

Specimens from the ferrite-pearlite steel 25Kh1M1F in mass fractions[6]: w(C) = 0.23…0.29; w(Mn) = 0.40…0.70; w(Si) = 0.17…0.37; w(Mo) = 0.60…0.80; w(Ni) = 0.30; w(Cr) = 1.50…1.80; w(S) δ 0.025) with applied coatings were loaded, under cyclic tensioning, on the STM-100 test setup at the frequency of f = 1 Hz, σ_{max} = 500 MPa, σ_{min} = 0.1 σ_{max}. After the failure of the specimens the coating condition was investigated at different values of the real transverse necking $\widetilde{\psi}$ of the specimens destroyed by tensioning[7]:

$$\widetilde{\psi} = \ln(F_0 / F_k) \tag{1}$$

where F_0 and F_k are the initial and the current areas of the cross-section, respectively.

Based on the analysis of the photo images, the structural and morphological data about the cracks and the mechanisms of their formation in the zones of localised tensioning and shear were analysed[8]. The measurement of the residual crack opening was determined by using the standard measurement of the Kappa software for TEM. The spatial orientation of the crack-network elements and their relation to the formation of the meso- and macroscale failure zones were determined.

3 STAGE-LIKE NATURE OF A NANOCOATING FAILURE

It is known that multiple defects have a multilevel structural organisation. So, any section of a specimen is an aggregate of structural elements separated by cracks of various sizes, which are stress concentrators[9,10]. Failure of the coating material takes place in the vicinity of a number of defects simultaneously, which prevents the peeling of the coating. Fatigue failure of a nanocoating is a step-by-step process, taking place in the following successive steps:

- formation of multiple defects – elastic-plastic deformation of a coating, nucleation of a network of individual cracks, their growth and partial coalescence followed by a gradual accumulation of local plastic strains in the most inhomogeneous sections of the material, **Figure 1a**.
- formation of a longitudinal deformation relief – this phenomenon is connected with the macrolocalisation of the plastic strain in individual sections of a specimen (**Figure 1b**), and shears of the grain conglomerates in the substrate material (steel 25Kh1M1F).
- activation of the transverse shears of the boundaries, formation of local microfragments of a coating – this process is preconditioned by a relative shear of the blocks of a coating due to the attainment of limiting values at the boundaries of the mesostructural elements (blocks), **Figures 1c, d**.

Further plastic deformation of the substrate material causes an intensified accumulation of plastic strains at the boundaries between the newly created blocks and the generation of damage[6]. At this stage, the opening of the crack-like defects is determined by the mutual effect of the elements of the block structure and the adhesive strength of the coating. Upon attainment of the ultimate state failure takes place, as well as the shear with the formation of a rupture, and the shear of the coating fragment.

In **Figure 2** the dependence of the residual crack opening (δ) on the real necking of a specimen is presented. With respect to this dependence, three sections are noticeable and they are reflected in the change of the slope angle of the curve. Within the first section, the

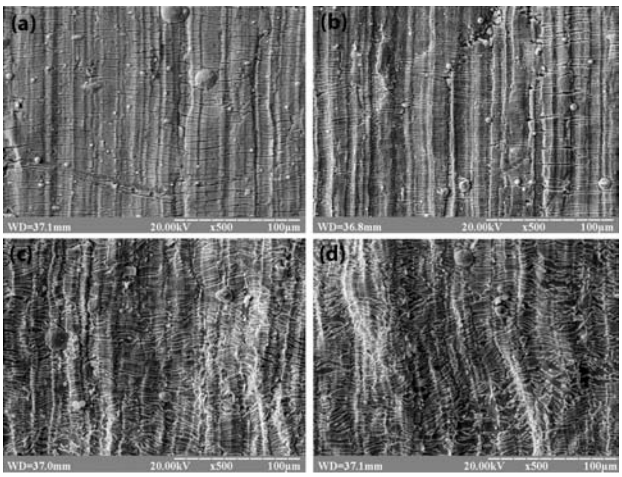

Figure 1: Stage-like nature of failure of a zirconium nanocoating at various plastic strains of the coating steel ($\widetilde{\psi}$ = 0; 0.23; 0.32; 0.35, respectively)

Slika 1: Stopničasta narava poškodb cirkonijeve nanoprevleke pri različnih plastičnih deformacijah jekla ($\widetilde{\psi}$ = 0; 0,23; 0,32; 0,35)

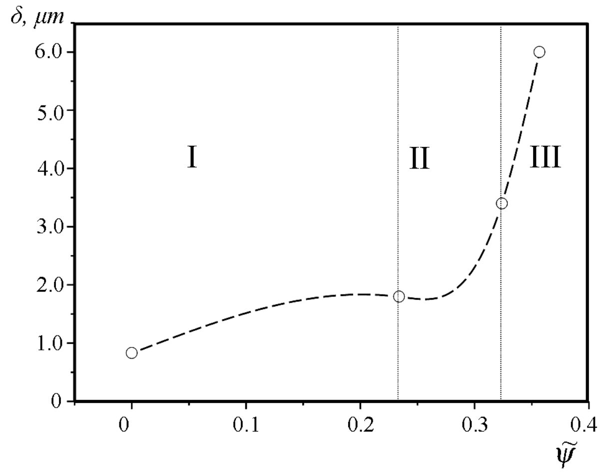

Figure 2: Stage-like nature of an increase in the mean value of a crack opening (δ) with an increase in the real necking of the specimen $\widetilde{\psi}$

Slika 2: Stopničasta narava srednje vrednosti odpiranja razpoke (δ) z naraščanjem resnične kontrakcije vzorca $\widetilde{\psi}$

newly formed cracks of the fatigue origin are located perpendicularly to the direction of loading; they have an insignificant residual opening (δ = 1.0…2.0 μm) and are separated from each other. So, the processes of the mutual effect of multiple defects are insignificant.

The second section corresponds to the opening and growth of the already formed network of defects. The coating acquires the properties of a breakup-block medium by dividing itself into separate "islands" of the material surrounded by multiple cracks. The residual crack opening in this section varies within the range of δ = 2.0…4.0 μm.

The third section corresponds to the critical opening of the defects of δ > 4.0 μm and a fragmentation of the coating. In this case, individual fragments of the coating are slipping relative to one another. In our opinion, local strains can be determined by using these fragments as the markers of displacements.

4 DISCUSSION OF IDENTIFICATION OF MULTIPLE CRACKS IN NANOCOATING

The algorithm for identifying the crack positions consists of the following main steps: binarisation of the original grayscale image, its filtering, repeated binarisation of the obtained image and its skeletonisation[3,4]. After the completion of the above operations we obtain an array of points that describe the geometrical parameters of the network of cracks in the image. While setting the algorithm, several parameters are used, which are crucial to the correct and precise identification of a crack.

The original image is a colour image obtained with an electron microscope (**Figure 3**). In order to simplify the analysis it is transformed into a black-and-white one by means of an adaptive binarisation, which allows us not only to identify potential objects (cracks), but also to eliminate the effect of inhomogeneous illumination during the image acquisition.

The most important parameter of this stage of the algorithm, which has a significant effect on the final result of the identification of the geometrical crack parameters, is the background L edge. A wrong choice of the background edge leads to the situation when a part of an object to be identified is mistaken for the background, or fictitious objects are found that are actually a part of the background. The "correctly chosen parameter" allows a value of the background border, at which the number of the identified crack objects approximates, as much as possible, the number of the cracks available in the image that can be identified by the operator. The range of the optimal values for the said parameter was determined by an experimental method[11]. The crack identification algorithm and the background border effect on the final result are considered in greater detail in our previous papers[8].

A change in the background edge causes a displacement of the edges of the objects found in the image, due to which the geometrical characteristics of the cracks calculated as a result of the algorithm operation may vary a little.

The result of the transformation is a monochrome image of the surface studied. It contains basic information about the location of potential objects sought; however, it also contains a large amount of noise elements, which complicate the process of identification and, to a certain extent, distort the general view of the cracked surface. In order to reduce the effect of the noise elements (which are present after the binarisation of the original grayscale image) and join the close-set individual fragments of a crack into a solid object, the filtering was performed followed by a repeated binary transformation of the filtered image.

Filtering is carried out in two stages: first, a discrete gauss filter is used, next the obtained image is enhanced by applying the filter to increase the image contrast[12]. The gauss filter is effective in suppressing the noises and smearing the edges of the image objects, as a result of

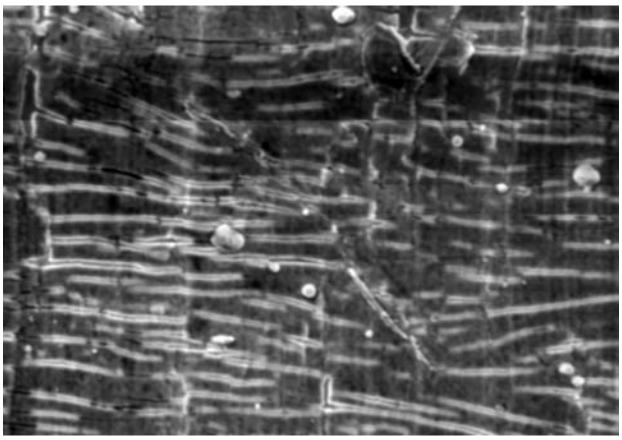

Figure 3: Original grayscale image
Slika 3: Originalen sivinski posnetek

Figure 4: Final image with detected cracks and a network of reference points
Slika 4: Končna slika z odkritimi razpokami in mrežo referenčnih točk

which the objects are formed that correspond to the crack positions. The contrast filter allows an increase in the contrast of the image elements.

For the final distinguishing of a crack a repeated binarisation of the filtered image is used. Following all of the above transformations, the final image is obtained, describing the picture of a cracking on the surface analysed (**Figure 4**). In this picture, white pixels correspond to the background and black ones to the cracks.

The most important parameter of the algorithm at this stage is the filter kernel size h_F. It has an effect on the processes of "screening" the background pixels and coalescence of the separated fragments of the crack, therefore its change may significantly affect the final result of the identification. The image obtained as a result of the above transformations contains information about the shape and the area of the cracks; however, it cannot be used directly for determining the quantitative parameters, such as the number, the length, the slope of the cracks, etc. In order to approximate the aggregate of pixels, which form the detected objects/cracks, a skeletonisation is performed using the simplest array of points[3,4]. The skeletonisation allows us to distinguish the wireframe lines of the cracks by detecting medial lines with the thickness of one pixel in the image. Reference points of the medial lines are the final set of data, on the basis of which a conclusion is made about the crack location, direction of the crack propagation and its length[12].

Both of the algorithm parameters considered have an immediate effect on the position of the obtained wireframe (medial) lines; therefore, an important issue is an assessment of their influence on the accuracy of determining the identified cracks[13]. The L parameter is the background border of the binary transformation. The h_F parameter is the filter kernel size. The corresponding corrections are made to the paper results. The background border of the binary transformation is the generally used parameter, which does not require any special clarification.

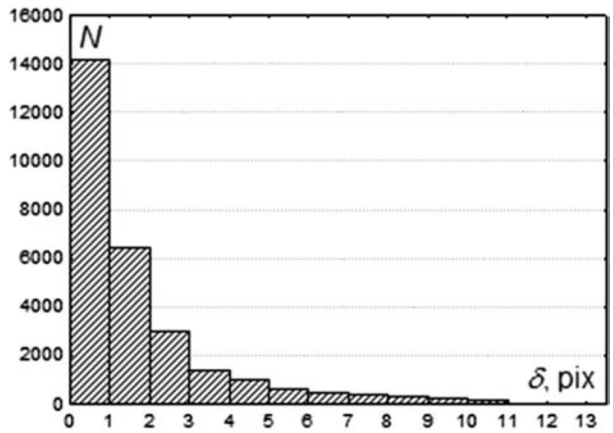

Figure 5: Displacement-distribution diagram for the points of the wireframe line
Slika 5: Diagram premika točk v liniji mreže

The effect of these parameters was assessed by observing the algorithm operation with the variation of their value within a certain range. To this end, the arrays of the ***P*** point coordinates, which form the wireframe line, were fixed for each group of the parameters (h_F and L). The obtained arrays of ***P*** points were used to determine the displacements of the wireframe line pixels depending on the values of the algorithm parameters studied. It is clear that the other algorithm parameters have insignificant effects on the identification results. Therefore, in general, the total error of determining the position of the crack wireframe line ξ, caused when setting the algorithm parameters, does not exceed the following value:

$$\xi \leq \xi_L + \xi_h \tag{2}$$

where ξ_L is the error in setting the background edge; ξ_h is the error caused by a displacement of the coordinate centre in setting the filter kernel size.

With a view to assessing the effect of the variation of the above algorithm parameters on the displacement of the wireframe line, the relative background edge L was varied within 10…30 %, and the filter kernel size h_F varied from 5 to 10 pixels. In this case, the array of $\boldsymbol{P}^{\mu}$ (L, h_F) points was fixed for every set of parameter values individually, where $\mu \in (1...M)$, and M is the number of observations. In order to calculate the displacement of the wireframe line, the closest point of the $\boldsymbol{P}^{\mu 2}$ (L, h_F) array was found for every $\boldsymbol{P}^{\mu 1}$ (L, h_F) point, and the distance between them was calculated. As a result of the above calculations, the aggregate of sets was obtained, which characterises the displacement of the points of wireframe line δ^{μ} (L, h_F). Each δ^{μ}_i element equals the distance between point $\boldsymbol{P}^{\mu 1}_i$ and point $\boldsymbol{P}^{\mu 2}_j$. To make the sample homogeneous by removing errors from it, the sample was censored using Wright's criterion. In this case, the values, for which the $\left|\xi_i - \bar{\xi}\right| \geq 3S_{\xi}$ condition was fulfilled (where $\bar{\xi}$ is the mean value and S_{ξ} is the standard deviation of the sample), were removed from the δ^{μ} (L, h_F) aggregate of values.

A typical view of the δ^{μ} (L, h_F) displacement-distribution histogram for the image (**Figure 3**) at the variation of the filter kernel size from 5 to 7 pixels and the constant background edge of 20 % is shown in **Figure 5**. It is clear that the majority of the points shift very insignificantly (only by a few pixels).

The investigation of a series of images showed that, at the constant background edge of L = 20 % and the variation of the filter kernel h_F from 5 to 10 pixels, the standard deviation of the sample varies within the range of 1.7–2.2 pixels.

When recalculated in the units of the real object length, the displacement varies within the range of 0.20…0.26 µm. The maximum displacement fixed for various measurements varied from 0.18 µm to 0.29 µm. Moreover, an increase in the kernel size proportionally influences the displacement of the crack wireframe lines.

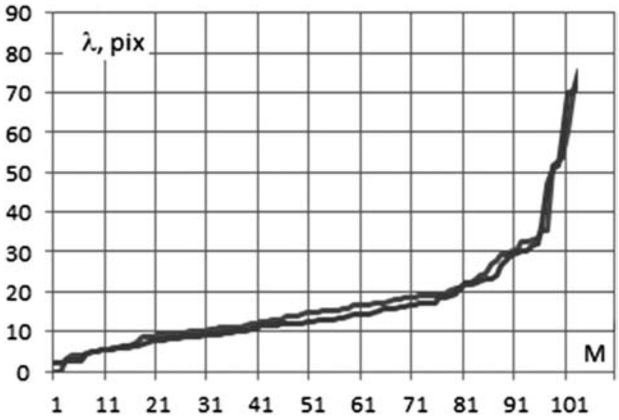

Figure 6: Graphs of detected crack lengths λ for the limiting values of the algorithm parameters
Slika 6: Graf dolžine odkritih razpok λ za omejene vrednosti parametrov algoritma

At the constant kernel size of h_F = 5 pixels and the variation of L from 5 % to 20 %, the standard deviation varies within the range of 1.14…1.95 pixels (which corresponds to 0.13…0.23 μm on the test specimen). In order to reveal the effect of the changes of any of the algorithm parameters studied on the final result – the geometrical parameters of the crack network – a number of investigations were carried out, during which the background edge varied within the range of 5…20 %, and the filter kernel size varied from 5 to 10 pixels.

Figure 6 shows the crack-length-distribution graphs for the image (**Figure 3**) at the limiting values of the background edge and the filter kernel size. The results obtained show that, for different images within the range of the algorithm parameters investigated, the deviation of the length from the mean value is within 0–0.30 μm. With the confidence probability of 95 % this deviation does not exceed 0.26 μm.

5 CONCLUSIONS

On the basis of the results of the investigations into the cracking processes in the surface of the heat-resistant steel with a nanocoating, it is established that multiple defects are caused by a plastic deformation of the substrate material. Multiple defects are nucleated in the strain-localisation areas and in the zones of microstructural inhomogeneity.

The new algorithm of digital defectometry has been developed, which is intended for the identification of the crack-network elements in a nanocoating. The effect of the main algorithm parameters on the measurement results relating to the geometrical characteristics – the coordinates, the lengths and the slope angles of the cracks – is investigated. The results obtained allow a compilation of such a combination of the algorithm parameters, with which the general measurement error of the cracking parameters will be minimal.

It was found that the optimum parameters of the algorithm for identifying the cracks have the following limits: h_F = 5…7 pixels and L = 8–14 %. With the confidence probability of 95 % the deviation of length is less than 0.30 μm.

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