Mechanical Properties and Surface Relief of 35HGSA Steel under Active Straining after Magnetron Deposition of Nanocomposite Coating and Ion Implantation

P.V. Kuznetsov, V.E. Panin, V.P. Sergeev, I.V. Petrakova, and A.V. Voronov

Institute of Strength Physics and Material Sciences SB RAS, 2/1, Academichesky ave., Tomsk, 364021, Russia
Phone: +7(3822) 28-68-40, Fax: +7(3822) 49-25-76, E-mail: kpv@ispms.tsc.ru

Abstract – Surface relief formed under active straining of samples after ion implantation and magnetron deposition and subsequent bombardment by composite ion beams has been investigated. An identification of mesoscale levels of plastic deformation and their quantitative characteristic has been carried out on the basis of fractal analysis of the surface images of deformed samples. Dependencies of an empirical parameter which is meaningful quantities of the assembly unit forming surface fractal structure at two scale levels on degree of plastic deformation of samples has been obtained. It is shown, that the high levels of strength and plasticity properties of samples there correspond to great values of the parameter. This quantitative parameter is offered to be used for estimation of an efficiency of ion-plasma treatment of materials with the purpose of increase of their fatigue durability, by using results of active loading of samples.

1. Introduction

Recent studies [1], have shown, that nanostructure formation in surface layers of materials is one of effective ways of influence on processes which develop in solids under loading. As the deformed solid is especially nonlinear system, this allows to influence an activation of all hierarchy of structural levels of initial structure of a material and, thus, to change its macro mechanical characteristics [1]. The condition on the surface of deformed solid is especially important under cyclic loading.

Among the various technologies used for creation nanostructure in surface layers of metals and alloys, treatment by concentrated beams of energy – an ionic, electronic, plasma, laser irradiation have a particular value [2–5].

A positive influence of plasma treatment and ionic implantation of various ions in the titan, copper, an Ti6Al4V alloy, corrosion-resistant stainless steel, 30ХГСА steel on fatigue strength in the range of low- and high cyclic fatigue has shown in [4, 5]. In particular, it is shown in [6, 7], implantation ions of molybdenum, chrome, nitrogen in 30 HGSA steel samples leads to increase of a fatigue strength from a reference value $\sigma_{\omega} \sim 528$ to the values ~ 600, 620, and 720 MPa, accordingly.

Influence of ionic-beam and plasma treatment on fatigue strength is multifactor function and it depends by nature targets, a kind and energy of ions etc. The estimation of efficiency of performed treatment and optimization of used modes is necessary for the maximal increase of fatigue strength of materials. The most accurate evaluation of treatment efficiency can be obtained by fatigue test of samples. However, fatigue tests are long-continued and expensive.

Therefore, development of efficiency estimation methods of treatment and forecasting of materials fatigue strength on the basis of other characteristics which can be obtained by with a less outlay is a topical problem.

The present work is devoted to a substantiation of a method, which would allow carrying out an estimation of efficiency of plasma treatment and ionic implantation by using results of samples test by an active straining.

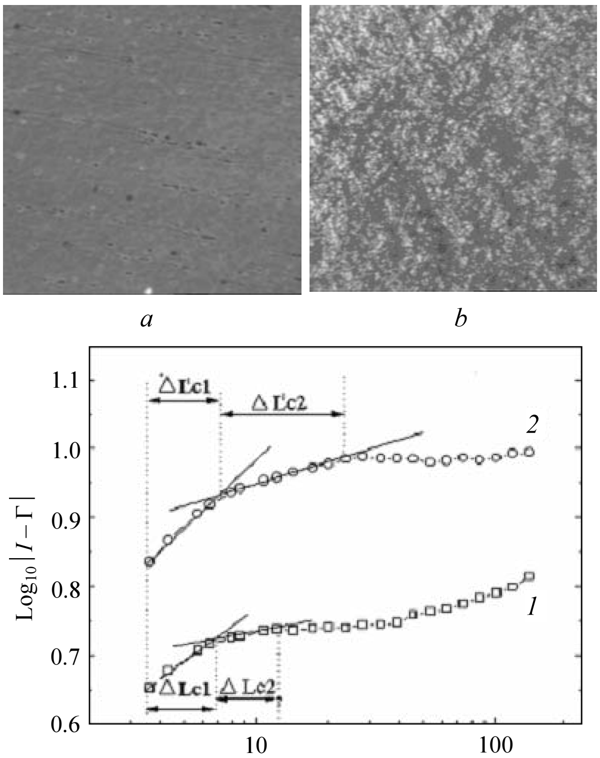
The proposed method is based on the physical mesomechanics approach [8]. According to [8], a rotation mode of deformation at formation mesostructure in surface layers of deformed solids plays a leading part in development of a fatigue pre-fracture stage of engineering materials. Therefore the analysis of mesostructure evolution in surface layers of samples subjected to plastic deformation is required for an efficiency estimation of carried out treatment. Mesoscale levels of self-organization of materials structure can be revealed on the basis of fractal analysis of the relief formed on the surface of samples under their active straining.

2. Samples end experimental procedure

Flat samples of 35HGSA high strength steel in the form of double blade with a test portion 17×2.5×0.6 mm in sizes were investigated. Samples were subjected to oil quenching from temperature the 880 °C and then low-temperature annealing at the 400 °C during ten minutes. The flat faces of the samples were polished and then subjected to deposition of coating and ion implantation. Direct implantation of ion of nitrogen (N^+) was carried out with use of a source of gas ions IGIND at accelerating voltage 20 kV and a current density 50 μA/cm^2. Preliminary magnetron deposition of thin nanocomposite films TiN with the subsequent implantation of ions $Al^+ + B^+$ was performed using an ionic source DIANA. Direct implantation of high dose of metal and nonmetallic ions TiAlBN compositions was carried out using a vacuum-arc pulse ionic source “Diana 2” with the accelerating voltage 80 kV. The composite cathodes were used for obtaining two element ion beams. They were made using powder metallurgical techniques. The specimens were subjected to active tensile loading in an IMASh-2078 test machine with simultaneous in situ recording of optical images of specimen surface relief in format 512×512 pixels with 256 grey gradations. The gauge portion of the specimen surface had dimensions 370×370 μm. Fractal analysis of the images of surface relief of samples was carried out using the technique described in [9, 10].

3. Results and discussion

The “stress-strain” diagrams of the tested samples in all cases have one stage of parabolic hardening in the range of $0 < \varepsilon < 0.01$. Magnetron deposition of thin nanocomposite films TiN and the subsequent ionic implantation by ions $Al^+ + B^+$ as well as ionic implantation by ions N^+ do not change plastic and strength property of a steel concerning initial samples.



a *b*

c

Fig. 1. Surface of 35HGSA steel after ionic implantation by N^+: *a* – reference state; *b* – $\varepsilon \sim 0.02$; *c* – the logarithmic dependencies of the correlation sums on the separation of optical image dots [5, 6] (*1* – $\varepsilon \sim 0.00$, *2* – $\varepsilon \sim 0.02$)

Ionic implantation by ions composition TiAlBN leads to small (~8%) decrease of strength characteristics of the steel and does not change its plasticity.

Figures 1, *a* and *b* illustrates the surface of the sample after implantation by nitrogen ions in an initial condition and after plastic deformation by active straining to $\varepsilon \sim 0.02$. As seen from the figure, the distribution of brightness dots in the image plane changes with increasing degree of strain. Figure 1*c* presents the corresponding logarithmic dependencies between the correlation sums and logarithm optical image dot separation [9, 10]. Clearly, the dependence obtained for the specimen surface shows two portions ΔL_1, ΔL_2 (Fig. 1, *c*) on which experimental points can be described by straight lines with an inclination in relation to axes. Inclinations of the portions ΔL_1, ΔL_2 dependences and their projections on an axis of scales changes with increase of deformation degree of samples (Fig. 1, *c*). This allows one to define dependences of two fractal dimensions D_{c1}, D_{c2} and corresponding upper limits of fractal behaviors L_{max1}, L_{max2} on degree of plastic deformation of samples.

Distribution of brightness of light in an image plane is caused by scattering ability of the surface. Accordingly to [11], in the geometrical optics limit a surface is assumed to be a collection of planar micro-facets. The micro-facets are specular (mirror-like) in reflectance and much larger in dimension than the wavelength of incident light. This condition is satisfied in our research as a wavelength of visible light $\lambda \sim 0.5$ μm less than the minimum size of a measuring step at calculation of the correlation sums $d \sim 3$ μm (Fig. 1, *c*). The orientation of each facet deviates from the mean orientation of the surface by an angle α; α is referred to as the facet slope and is random variable. It is convenient to assume, that α to be normally distributed. The surface is also assumed to be isotropic. Hence, surface radiance L_r is determined by the roughness parameters σ_a and the angle of refraction θ_γ [11]:

$$L_r = \frac{A}{\cos(\theta_r)} \exp\left(-\frac{\alpha^2}{2\sigma_\alpha^2}\right),$$

where A is the constant, σ_α is the standard deviation of facet slope.

Examination of the dependences $D_{c1}(\varepsilon)$, $D_{c2}(\varepsilon)$ reveals that they change in ranges $\Delta L_1 \sim (4 \div 7.5)$ and $\Delta L_2 \sim (7.5 \div 33)$ micron, accordingly. Thus, the relief of the investigated samples is characterized by two functions of inclination distribution of micro-facets with various dispersions which, obviously, are caused by different mechanisms of their formation.

Dependencies $D_{c1}(\varepsilon)$, $L_{max1}(\varepsilon)$ is supposed to be characterize surface relief on scales within the range of average grain size, and $D_{c2}(\varepsilon)$, $L_{max2}(\varepsilon)$ correspond to scale of order of a few grains.

An empirical parameter N_j which can be calculated on the basis of quantities L_{max1}, L_{max2}, D_{c1}, and D_{c2} has been offered in work [10]. The meaning of the parameter N_j is a quantity of blocks forming fractal structure on *j* scale level. We have calculated dependences $N_1(\varepsilon)$, $N_2(\varepsilon)$ for the investigated samples using dependences $D_{c1}(\varepsilon)$, $L_{max1}(\varepsilon)$, $D_{c2}(\varepsilon)$, $L_{max2}(\varepsilon)$ (Fig. 2). A character of the dependencies behavior $N_1(\varepsilon)$, $N_2(\varepsilon)$ and the change range of their values depends on a mode of ion-beam and plasma treatment of samples (Fig. 2).

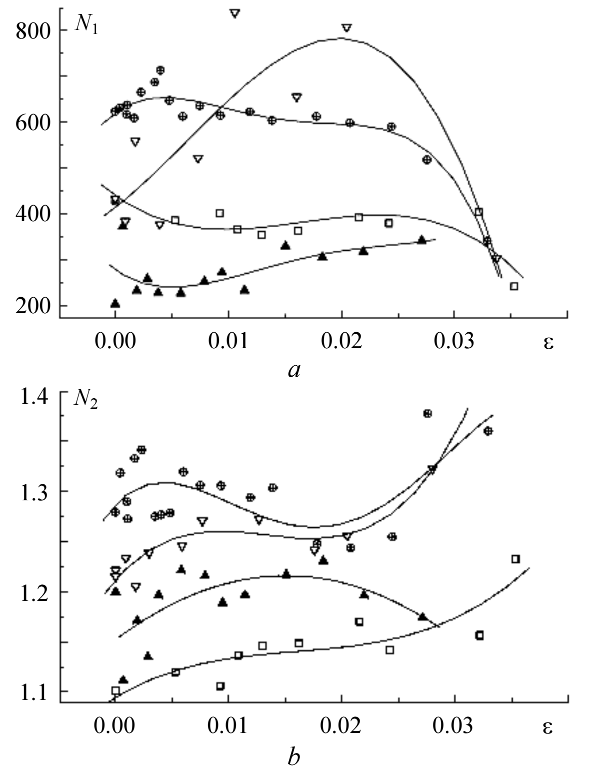


Fig. 2. Dependences N_1, N_2 on degree of plastic deformation of samples an active straining (ε): □ – initial samples; ⊕ – implantation by ions N^+; ▲ deposition of TiN covering and implantation by ions $Al^+ + B^+$; ∇ – implantation by ions TiAlBN

Clearly expressed nonlinear character of behavior of the dependences $N_1(\varepsilon)$, $N_2(\varepsilon)$ is observed for the samples implanted by ions TiAlBN and ions N^+ (Fig. 2).

The highest values N_1, N_2 correspond to the samples implanted by ions TiAlBN and by ions N^+, accordingly. The lowest values of parameters N_2, N_1 are observed for samples of steel in the reference state, and after deposition of nanocomposite films TiN with the subsequent ionic implantation by ions $Al^+ + B^+$, accordingly (Fig. 2). Taking into account that $N \sim 1/\delta^D$ where δ is the measuring step and D is the fractal dimension accordingly, we can conclude that great values of parameter N corresponds with a formation of thin surface fractal structures.

Ionic implantation of samples with various thicknesses was carried out by ions N^+ in the same conditions. Therefore, thickness of the implanted layer of the investigated samples was identical.

The "stress-strain" diagrams of samples with various thickness, implanted by N^+ ions, and corresponding dependences $N_2(\varepsilon)$ are shown in Fig. 3. The samples with the thickness 0.65 mm have higher characteristics of strength and plasticity relative to the samples with thickness 0.49, 0.58, and 0.70 mm. Dependences $N_2(\varepsilon)$ for samples with the minimum thickness of ~0.49 mm and with the thickness of ~0.65 mm reveal strong nonlinearity. Dependence $N_2(\varepsilon)$ for samples with the thickness of ~0.49 mm has minimum values at an initial stage of plastic deformation, and then increases before their fracture. This testifies that the basic resistance of plastic deformation in the system "the sample – the modified surface layer" of the samples with thickness 0.49 mm at an initial stage is caused by the strengthened surface layer, and stress in the basic material of the sample is low.

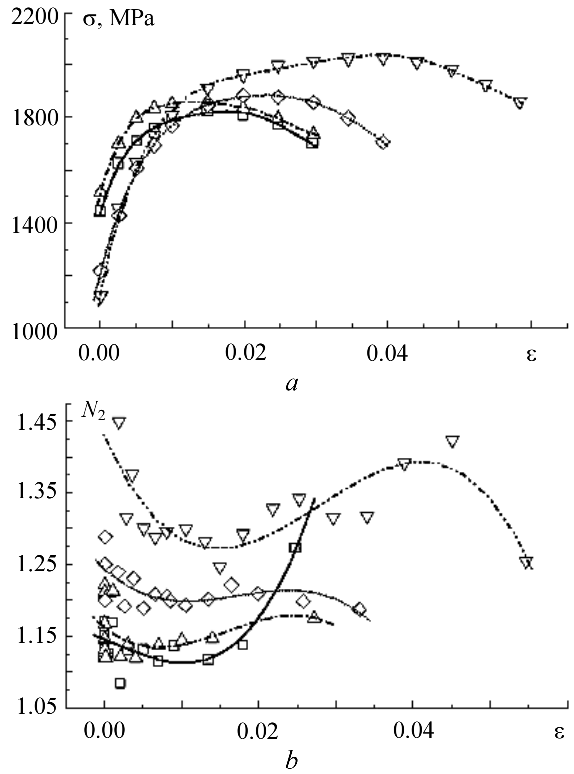


Fig. 3. The "stress-strain" diagram (*a*) and the dependences of parameter N_2 on degree of plastic deformation of samples with various thickness, implanted by N^+ ions (*b*): Δ – 0.70; ∇ – 0.65; ◊ – 0.58; □ – 0.49 mm

It is known, that ionic implantation and plasma treatment leads to change of mechanical properties of surface layers of samples [1–7]. Thus, the samples are supposed as being two-layer system "the sample – the modified surface layer». According to [1, 12, 13] periodic stress and strain distributions arise at the interface of two-layer systems with various mechanical characteristics of parts making it under external actions. The period of formed structures is proportional to a thickness of the surface layer and depends on the stress in a substrate [13]. Accordingly to [13] than the higher stress in a substrate, the thinner structure is formed on the surface of coating.

It is shown in [6], that unlike ions B^+ and Ni^+ distribution of ions N^+ has no a distinctly expressed maximum at the same modes of irradiation. Besides, the depth of penetration of ions N^+ significant exceeds the depth of penetration ions B^+ and Ni^+. Possibly, it means, that thickness of the modified layer at ionic implantation by ions N^+ more than at ionic implantation by ions B^+ and Al^+ . Thus, formation of thin fractal structures under active straining of the samples implanted by ions N^+, possibly, testifies to high values of stress in the base material.

It is known; thickness of the implanted layer is a small portion of a samples thickness and varies from several tens to hundred nanometers. It depends on a kind of materials, energy and type of ions, temperature of treatment and other factors [1–7].

It has been conclusively shown [1–7], that relative changes of characteristics of fatigue strength of the samples caused by ionic implantation, does not submit to the estimations which are starting with the law of additivity of the contribution of thickness of the implanted layer and thickness of a substrate. Special research of influence of thickness of samples of high-strength steel 35HGSA with the modified barrier surface layer formed by implantation of ions N^+, on change of mechanical characteristics and character of formation of a deformation relief has been carried out. Thickness of samples changed in limits from ~0.49 to ~0.7 mm.

With increase of straining stress, stress in the basic material of samples has increased too that leads to formation of more thin fractal structures on the surface and to increasing in parameter of $N(\varepsilon)$. The dependence $N_2(\varepsilon)$ for samples with a thickness of ~0.65 mm has the greatest values in all range of plastic deformation. It testifies to high values of stress in the basic material and, possibly, their more homogeneous distribution in the system «the sample – the modified surface layer», that provides high strength and plastic properties of samples (Fig. 3, *a*).

Thus our results indicate that formation thin surface fractal structures under an active straining of the samples subjected to plasma and ion beam treatment and ionic implantation, testifies of high accommodation abilities of mesostructure which provides high characteristics of strength and plasticity of samples. Possibly such structure it is capable to provide high level of fatigue strength of samples. To answer this question direct comparison with results of fatigue tests of samples is necessary.

4. Conclusion

Traditional approaches to an estimation of results of ion beams and plasma materials treatment, for example by measurement of hardness, give the information about local mechanical characteristics of surface layer. However, load-carrying capacity of materials structure with the modified surface layer can be revealed only in dynamics during its development under external actions. This approach is offered in our work.

Our preliminary researches show high sensitivity of the parameter based on the fractal analysis of optical images of surface mesostructure formed under an active straining of samples, to modes of ionic and plasma treatment of high-strength steel 35HGSA.

References

[1] V.E. Panin, *Surface Layers and Internal Interfaces in Heterogeneous Materials,* Novosibirsk, Publishing house SB RAS, 2006, pp. 5–69.

[2] V.P. Sergeev, A.R. Sungatulin, O.V. Sergeev, G.V. Pushkareva, and V.E. Panin, Phys. Mesomech. **8**, 121–124 (2005).

[3] V.E. Panin, V.P. Sergeev, Yu.I. Pochivalov, S.V. Panin, and A.V. Voronov, Phys. Mesomech. **8**, 113–116 (2005).

[4] D.K. Hirvonen, *The ion implantation*, Moscow, Metallurgiya, 1985, pp. 1–391.

[5] F.F. Komarov, *The ion implantation in metals,* Moscow, Metallurgiya, 1990, pp. 1–216.

[6] A.V. Fedorov, E.V. Vasiljeva, V.G. Vladimirov, M.I. Guseva, S.M. Savicheva, and V.F. Terentjev, Surface. Phys. Chemistry. Mechanics. **8**, 123–131 (1983).

[7] V.G. Vladimirov, M.I. Guseva, S.M. Ivanov, V.F. Terentjev, A.V. Fedorov, and V.A. Stepanchikov, Surface. Phys. Chemistry. Mechanics. **7**, 139–147 (1982).

[8] V.E. Panin, ed., *Physical Mesomechanics of Heterogeneous Media and Computer-Aided Design of Materials*, Cambridge, Cambridge Interscience Publishing, 1998.

[9] P.V. Kuznetsov, V.E. Panin, K.V. Levin, A.G. Lipnitsky, and J. Schreiber, Phys. Mesomech. **3**/4, 89–94 (2000).

[10] P.V. Kuznetsov, A.A. Oksogoev, and I.V. Petrakova, Phys. Mesomech. **7**/2, 49–57 (2004).

[11] S.K. Nayar, K. Ikeuchi, and T. Kanade, IEEE Transactions of pattern analysis and machine intelligence. **13**, 7, 611–633 (1991).

[12] S.L. Bazhenov, A.V. Volynskii, O.V. Lebedeva, E.E. Voronina, and N.F. Bakeev, High-molecular compound A. **5**/43, 844–851 (2001).

[13] S.L. Bazhenov, A.V. Volynskii, O.V. Lebedeva, E.E. Voronina, and N.F. Bakeev, High-molecular compound. A. **9**/41, 1442 (1999).