ISSN 1063-7842, Technical Physics, 2018, Vol. 63, No. 3, pp. 385–390. © Pleiades Publishing, Ltd., 2018.
Original Russian Text © Yu.R. Kolobov, E.A. Korneeva, I.N. Kuz'menko, A.N. Skomorokhov, S.I. Kudryashov, A.A. Ionin, S.V. Makarov, A.Yu. Kolobova, S.S. Manokhin, V.I. Betekhtin, A.G. Kadomtsev, 2018, published in Zhurnal Tekhnicheskoi Fiziki, 2018, Vol. 88, No. 3, pp. 396–401.

PHYSICAL SCIENCE OF MATERIALS

Effect of Processing by Femtosecond Pulsed Laser on Mechanical Properties of Submicrocrystalline Titanium

Yu. R. Kolobov[a, b,*], **E. A. Korneeva**[c], **I. N. Kuz'menko**[a], **A. N. Skomorokhov**[d],
S. I. Kudryashov[e,f], **A. A. Ionin**[e], **S. V. Makarov**[e,f], **A. Yu. Kolobova**[b, g],
S. S. Manokhin[a, b], **V. I. Betekhtin**[h], **and A. G. Kadomtsev**[h]

[a] *Belgorod State National Research University,*
Belgorod, 308015 Russia
[b] *Institute of Problems of Chemical Physics, Russian Academy of Sciences,*
Chernogolovka, Moscow oblast, 142432 Russia
[c] *Joint Institute for Nuclear Research,*
Dubna, Moscow oblast, 141980 Russia
[d] *Institute of Physics and Power Engineering, State Research Center of Russian Federation,*
Obninsk, 249033 Russia
[e] *Lebedev Physical Institute, Russian Academy of Sciences,*
Moscow, 119991 Russia
[f] *National Research Nuclear University MEPhI, Moscow, 115409 Russia*
[g] *National Research Technological University MISiS, Moscow, 119991 Russia*
[h] *Ioffe Institute, Russian Academy of Sciences,*
St. Petersburg, 194021 Russia
**e-mail: kolobov@bsu.edu.ru*

Received May 31, 2017

Abstract—Effect of femtosecond laser processing on mechanical properties of plates made of submicrocrystalline VT1-0 titanium alloy is studied using active deformation and fatigue testing involving cantilever bending.

DOI: 10.1134/S106378421803009X

INTRODUCTION

Submicrocrystalline (SMC) and nanostructured (NS) alloy-free titanium is a promising material for modern metal biomaterials and surgical implants [1]. Formation of the SMC and NS states in titanium alloys leads to substantial improvement of mechanical and physicochemical properties, in particular, properties needed for applications as materials for medical implants in traumatology, orthopedics, stomatology, and several branches of medicine and technology.

Surface modification by femtosecond laser pulses is a promising method for an increase in biocompatibility of the SMC titanium and biological tissues. Femtosecond laser radiation (FLR) provides lower contamination in comparison with alternative methods for surface processing, is highly technological, and can be used for processing of items with complicated surfaces. A relatively small depth of the zone of thermal effect of less than one micrometer is an important advantage of ultrashort (femtosecond) laser pulses [2]. Such an advantage makes it possible to modify thin surface layers in the absence of heating of bulk material, which, normally, leads to a decrease in mechanical strength due to degradation of the SMC and NS states or complete elimination of such states owing to the reverse processes and recrystallization. Thus, the application of femtosecond lasers is important for modification of the surface of titanium alloys in the SMC and NS states, since the development of the above processes at relatively high temperatures leads to a decrease in the strength of the materials typical of course-grain structures [3, 4].

Controlled modification of surface profiles with the aid of FLR is a promising method for an increase in osteointegration, since the surface nanorelief specifically affects the behavior of various cells and, in particular, provides an increase in the bioactivity of osteoblasts [5, 6]. Controlled modifications of surface relief of metal implants using FLR and the corresponding effect on biocompatibility have been studied in several works (see, for example, [5–7]). Note insufficient data on the effect of FLR on the mechanical properties of titanium alloys. In this work, we study the effect of FLR on the mechanical properties of the SMC VT1-0 titanium.

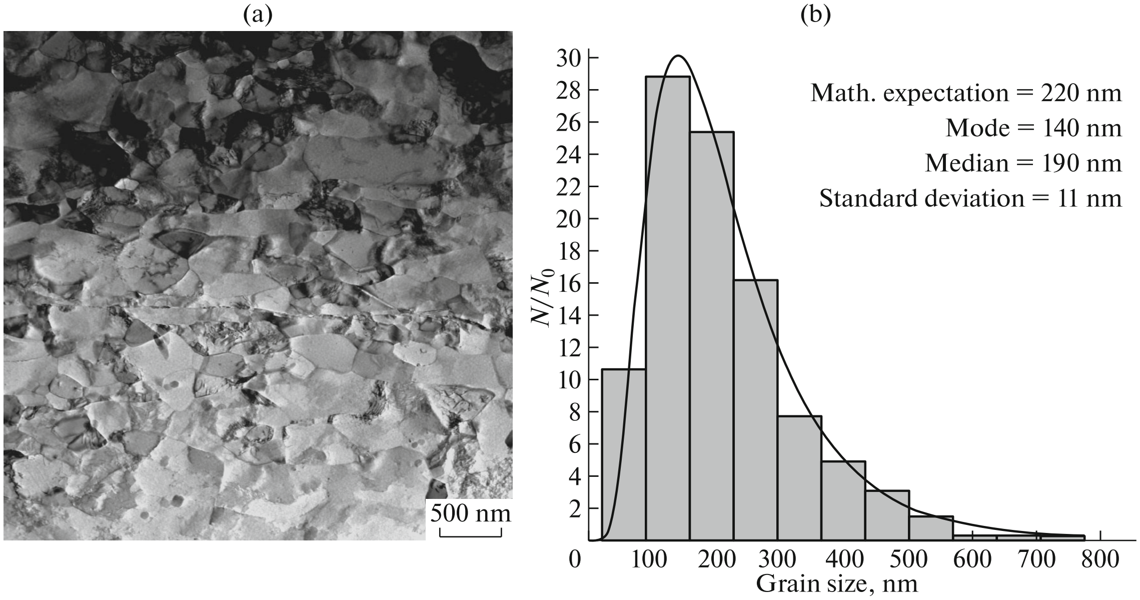

Fig. 1. Original SMC VT1-0 titanium sample: (a) TEM image of the microstructure and (b) histogram of the grain distribution with respect to size.

MATERIALS AND METHODS

We study mechanical properties of the original SMC VT1-0 titanium alloy and the FLR-modified sample. The SMC state with homogeneous globular structure with a mean grain size of about 220 nm (Fig. 1) is obtained for rods with a diameter of 8 mm using mechanical and thermal processing that involves radial-displacement, helical, and section rolling [8]. Residual microstress (first-order stress) is eliminated due to 3-h-long annealing in a Nabertherm LT 5/13/P330 muffle furnace in air at a temperature of 350°C. Then, test samples (plates) are cut from the annealed rods with the aid of spark cutting using a SODICK AQ 300L device. The plates with working sizes of 2 × 0.5 × 27 and 2 × 0.2 × 27 mm are used for tension tests, and the plates with working sizes of 3 × 0.6 × 16 mm are used for fatigue tests using symmetric cantilever bending. The samples are mechanically grinded and polished using a Struers LaboPol-5 device prior to the laser processing and testing.

The mean sizes of the grains are determined using the random linear intercept method [9, 10]. The ImageScope software is employed for construction and mathematical processing of histograms of grain-size distributions. Figure 1b presents the histogram of the size distribution of grains for the original VT1-0 titanium alloy and the corresponding statistical parameters. The distribution density function is approximated using the logarithmic law.

Laser Processing

In the experiments on the surface modification of the SMC titanium using FLR, we employ linearly polarized radiation of an Avesta Project Ti:Sa femtosecond laser setup with a central wavelength of $\lambda \approx$ 744 nm, a FWHM of the radiation band of 12 nm, a pulse duration of about 100 fs in the interaction region, a pulse energy of up to 8 mJ, and a pulse repetition rate of 10 Hz. The laser energy is chosen to avoid noticeable degradation of the energy density distribution on the surface of the target due to self-focusing in air and the corresponding chromatic emission, filamentation, and scattering by air plasma.

The processing is performed in air at a fluence of $F = 0.3$ J/cm^2 and a scanning speed of $v = 18$ μm/s. The scanning direction and polarization vector are oriented in such a way that the periodic parallel grooves that result from the laser modification are directed along the plate (i.e., in parallel to the axis of quasi-static tension and tension–compression axis in cyclic tests).

To choose the modification regimes, we use the results of the previous work, in which we have structurally modified SMC titanium using FLR. The results of [11] show that different types of relieves can be obtained at different laser fluences and scanning rates. The modification regime with the parameters $F = 0.3$ J/cm^2 and $v = 18$ μm/s is optimal with respect to biocompatibility. Note the absence of deep pores and cracks that may serve as concentrators of stress and cause a decrease in the strength parameters of the material (especially, under cyclic loads).

Methods for Mechanical Tests

The fatigue tests are performed using an Instron Electropuls 3000 device and supplemented with the

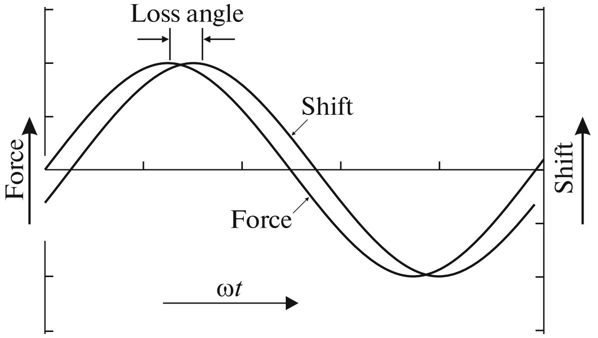

Fig. 2. Loss angle φ between load φ_σ and shift φ_ε.

study of viscoelastic properties using the dynamic mechanical analysis (DMA) [12]. In the fatigue tests, we employ the cantilever bending at an oscillation frequency of 10 Hz at room temperature. The tests are performed in the symmetric mode with a cycle asymmetry coefficient of $R = -1$ in the presence of loading at constant amplitude of the deflection. The size of the working region of the sample in the tests is 0.60 × 3.0 × 16.0 mm. The amplitudes of force are used to calculate stress with the aid of the formula

$$\sigma = 6\frac{Fl}{bh^2},$$

where F is the amplitude of the force exerted on the sample, l is the length of the working part of the sample, b is the width of the sample, and h is the thickness of the sample.

In the fatigue tests, we measure the instantaneous force (load) and shift (deformation) (Fig. 2) and perform the DMA using the correlation method. In the corresponding procedure, we determine the phase difference (loss angle φ) of the periodic variation in the load and deformation.

The measured tangent of the loss angle $\tan\varphi$ characterizes scattering of the oscillation energy ΔW due to inelastic internal microscopic processes in solid:

$$\tan\varphi = \frac{1}{2\pi}\frac{\Delta W}{W}.$$

Here, $\Delta W/W$ is the part of energy that must be transferred to the oscillation system over a single period to provide constancy of total oscillation energy W. An increase in quantity $\tan\varphi$ indicates an increase in the intensity or activation of additional inelastic processes that accompany the cyclic deformation of solid (e.g., reversal and microplastic deformation, growth of cracks, etc.) [13].

The tensile tests are performed with the aid of an Instron 5882 electromechanical device at a deformation rate of 1.5 mm/min using an extensometer with a working length of 10 mm.

A Quanta 600 FEG scanning electron microscope with field emission is used to study surface morphology and elemental composition of the processed samples.

EXPERIMENTAL RESULTS AND DISCUSSION

Figure 3 shows the morphology and cross section of the FLR-processed titanium surface.

The regimes of laser processing and the structure that is formed due to such processing have been analyzed in [11]. The structure exhibits ordered micro- and nanorelief, in particular, grooves that are parallel to the scanning direction.

Figure 4 presents the deformation curves obtained in the tensile tests for the original SMC titanium plates with thicknesses of 0.2 and 0.5 mm and the plates with laser-modified surfaces. It is seen that the laser-induced modifications of the surface and near-surface layers weakly affect characteristics of the material

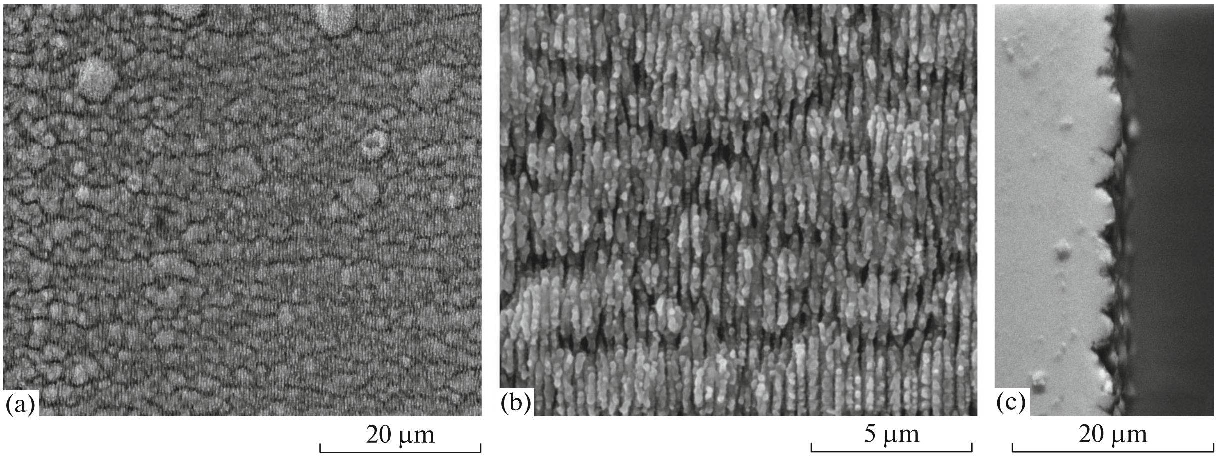

Fig. 3. Surface morphology of the FLR-processed VT1-0 titanium samples: (a) ×5000, (b) ×20 000, and (c) cross section ×5000.

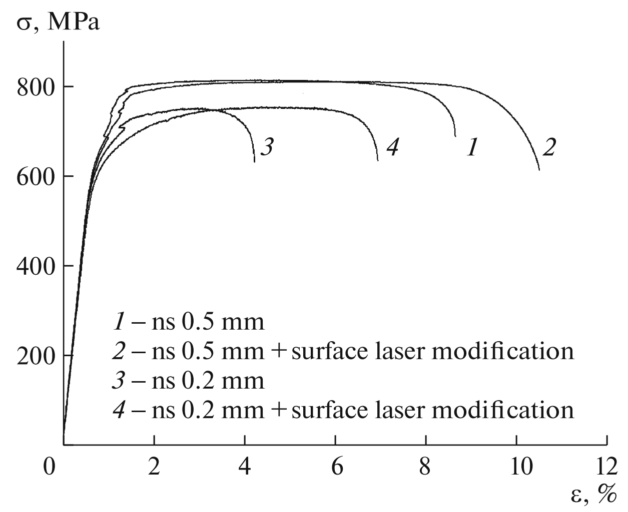

Fig. 4. Deformation curves for the SMC VT1-0 titanium samples with thicknesses of 0.2 and 0.5 mm with laser-modified surfaces in comparison with the deformation curves of the original SMC titanium samples.

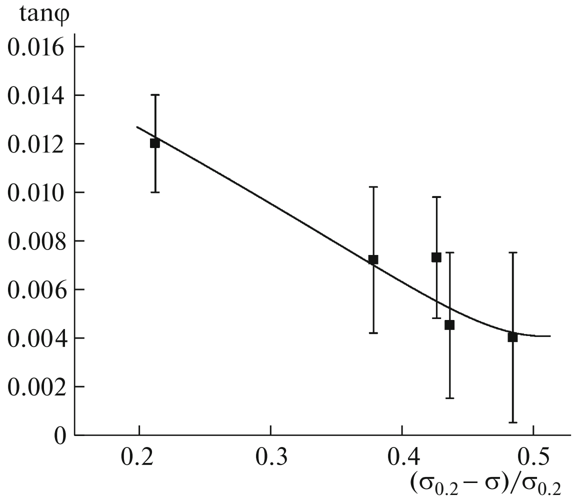

Fig. 6. Plot of tangent of loss angle tanφ vs. relative deviation of the maximum stress per cycle from the yield point.

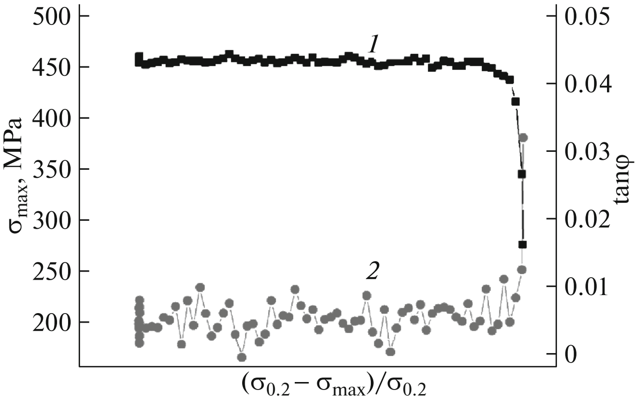

Fig. 5. Synchronously recorded experimental results on (*1*) maximum stress per cycle σ and (*2*) tangent of loss angle φ versus number of deformation cycles.

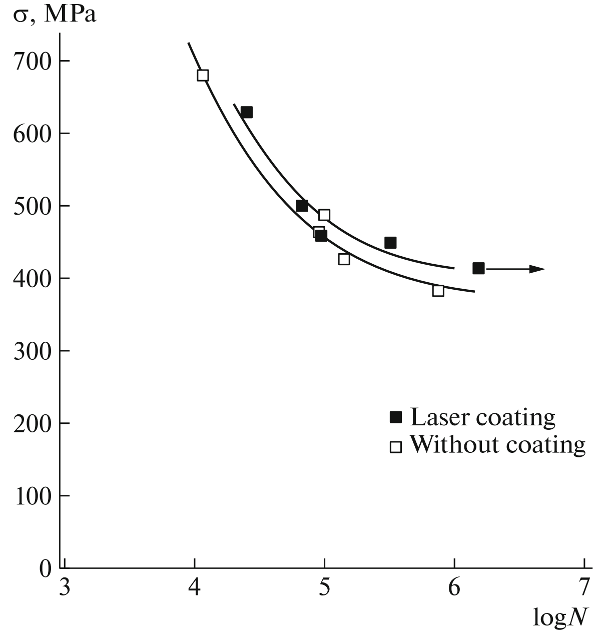

Fig. 7. Plots of maximum stress per cycle vs. number of cycles preceding destruction (fatigue curve) for the nanostructured VT1-0 titanium plates with a thickness of 0.6 mm (open squares) in the absence of coating and (closed squares) after femtosecond laser processing.

under study (mechanical strength, yield point, and Young modulus). The Young modulus can be estimated in the first approximation using the slope of the linear fragment of deformation curve σ–ε. However, the laser-modified samples exhibit higher plasticity in comparison with original samples.

Figure 5 shows the synchronously recorded results on the maximum stress in the cycle σ (curve *1*) and tangent of the loss angle (curve *2*) versus number of cycles. The curve of cyclic strengthening (curve *1* in Fig. 5) shows that the maximum stress in the cycle (at the fixed deflection) remains almost unchanged and decreases only before destruction. Quantity tanφ (curve *2*) also remains unchanged and sharply decreases only prior to destruction.

Thus, the beginning of the destruction of the sample under cyclic load can be determined using both curves of cyclic strengthening and a decrease in the amplitude of stress and an increase in the tangent of the loss angle.

Figure 6 presents the dependence of the tangent of loss angle on the relative deviation of the maximum stress per cycle from the yield point of the material under tensile stress $(\sigma - \sigma_{max})/\sigma_{0.2}$ for the domain in

Table 1. Results of mechanical tension and fatigue tests for the original plates with a thickness of 0.5–0.6 mm made of the SMC VT1-0 titanium alloy and the laser-processed plates

Sample	Tension			Fatigue limit σ_{RN} for 10^6 cycles, MPa	σ_{RN}/σ_B
	$\sigma_{0.2}$, MPa	σ_B, MPa	$\sigma_B/\sigma_{0.2}$		
Original	642 ± 7	805 ± 7	1.25	385	0.47
Laser-processed	652 ± 7	802 ± 5	1.23	422	0.53

which the tangent of loss angle is independent of the number of cycles (Fig. 4). It is seen that the tangent of loss angle decreases with a decrease in the maximum stress per cycle relative to the yield point. Such a result corresponds to a decrease in the intensity of microplastic deformation with a decrease in the maximum stress per cycle, which is in agreement with an increase in the number of cycles preceding destruction with a decrease in the maximum stress per cycle.

To determine the fatigue limit, we use the experimental results to plot the maximum stress per cycle versus number of cycles preceding destruction (fatigue curve) for the original VT1-0 titanium plates (open squares) and laser-processed samples (closed squares). The results of Fig. 7 are obtained for the stress of the curves of cyclic strengthening that are similar to the curves of Fig. 5. For the experimental stresses, the FLR-processed samples exhibit greater number of cycles prior to destruction in comparison with the original samples.

The above results on the effect of surface processing with the aid of femtosecond laser pulses on the mechanical properties of the material under study (in particular, improvement of the fatigue limit and an increase in the plasticity in the quasi-static breaking tests) are in agreement with each other, since the propagation of fatigue microcracks in materials with enhanced plasticity is slower than the propagation of such cracks in fragile materials. Thus, the surface FLR-modification of implants that leads to the formation of relief with complicated structural hierarchy leads to improvement of both biocompatibility and mechanical properties. Note that an increase in the fatigue resistance and plasticity of the SMC VT1-0 titanium resulting from the femtosecond laser processing shows the advantages of the method in comparison with alternative methods for surface processing of titanium alloys. In particular, the formation of bioactive and bioinert coatings on the surface of titanium alloys using microarc oxidation leads to lower fatigue limit in the fatigue tests of the Ti–Al–Zr titanium alloy with controlled load amplitude in the tension–compression procedure [14] and undoped titanium (Grade 4 alloy) in the test using four-point bending (yield point and mechanical strength) [15]. The microarc oxidation also leads to a decrease in the mechanical strength of the SMC VT1-0 titanium under quasi-static load [16].

Table 1 presents the results of mechanical tension and fatigue tests of the original SMC plates of the VT1-0 titanium alloy with a thickness of 0.6 mm and plates processed with the aid of the femtosecond laser pulses.

It is seen that the yield points and mechanical strengths of the laser-processed and original samples are almost identical and the conditional fatigue limit for 10^6 cycles is significantly higher for the laser-processed samples.

CONCLUSIONS

Surface processing of the SMC VT1-0 titanium plates with a thickness of 0.6 mm by femtosecond laser pulses leads to an increase in the conditional fatigue limit (for the tests consisting of 10^6 cycles). The tensile tests of the SMC VT1-0 titanium plates with thicknesses of 0.5 and 0.2 mm show that the laser processing weakly affects the measured yield points and mechanical strength but leads to a significant increase in the prefracture deformation.

The study using the DMA method shows that the microplastic deformation of the samples under study decreases with a decrease in the maximum stress per cycle in the cyclic tests. The destruction of the samples under cyclic loads is accompanied by a sharp increase in the microplastic deformation (tangent of loss angle). Such a result can be used as a diagnostic feature of the fatigue limit of the material.

ACKNOWLEDGMENTS

The study of the effect of processing with the aid of femtosecond laser pulses on the mechanical properties of the SMC VT1-0 titanium alloy in the fatigue tests using the cantilever bending was supported by the Russian Science Foundation (project no. 15-12-30010). The study of the regimes of laser irradiation was supported by the Program for Improvement of Competitiveness of the National Research Nuclear University MEPhI.

REFERENCES

1. Yu. R. Kolobov, Nanotechnol. Russ. **4**, 758 (2009).
2. R. Le Harzic et al., Appl. Phys. Lett. **80**, 3886 (2002).
3. Yu. R. Kolobov, A. G. Lipnitskii, M. B. Ivanov, I. V. Nelasov, and S. S. Manokhin, Russ. Phys. J. **54**, 918 (2012).

4. Yu. R. Kolobov, M. B. Ivanov, S. S. Manokhin, and E. Erubaev, Inorg. Mater. **52**, 128 (2016).
5. E. Fadeeva et al., Langmuir **27**, 3012 (2011).
6. R. J. Crawford et al., Adv. Colloid Interface Sci. **179**, 142 (2012).
7. E. Luong-Van, I. Rodrigue, et al., J. Mater. Res. **28**, 165 (2013).
8. Yu. R. Kolobov, M. B. Ivanov, E. V. Golosov, and A. V. Penkin, RF Patent No. 2389568 (2010).
9. S. A. Saltykov, *Stereometric Metallography* (Metallurgiya, Moscow, 1986).
10. M. N. Stepnov, *Statistical Methods for Processing the Results of Mechanical Tests* (Mashinostroenie, Moscow, 1985).
11. A. Ionin et al., Laser Phys. Lett. **10**, 045605 (2013).
12. N. E. Fomin, V. A. Yudin, and A. A. Kireev, Innovatsionnoe Obraz., No. 4, 129 (2013).
13. *Internal Friction Method in Metallography*, Ed. by M. S. Blanter and Yu. V. Piguzov (Metallurgiya, Moscow, 1991).
14. Y. M. Wang, P. F. Zhang, L. X. Guo, J. H. Ouyang, Y. Zhou, and D. C. Jia, Appl. Surf. Sci. **255**, 8616 (2009).
15. J. M. Wheeler, C. A. Collier, J. M. Paillard, and J. A. Curran, Surf. Coat. Technol. **204**, 3399 (2010).
16. E. A. Korneeva, A. N. Skomorokhov, Yu. R. Kolobov, I. N. Kuz'menko, G. V. Khramov, and V. V. Rakityanskii, Kompoz. Nanostrukt. **4**, 32 (2011).

Translated by A. Chikishev