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Microstructure evolution in friction-loaded layers of nickel

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ABSTRACT

The kinetics of microstructure formation and destruction in friction-loaded layers of nickel are discussed from the perspective of dislocation representations. The mechanisms of surface layer destruction are addressed in detail, along with the characteristics of the destruction material.

KEYWORDS nanocrystalline structures; friction loading; destruction of submicrocrystalline structure; mechanisms of intercrystalline destruction; electron-microscopic research; intensity of wear.

Introduction

The physical processes of deformation-induced dispersion and subsequent destruction in friction-loaded metal layers are affected mainly by dislocation structure kinetics. The microstructure elements of strengthening and destruction in friction-loaded polycrystalline surface layers of nickel have been partly described in [1-4]. It has been shown that, by comparison with other forms of loading (such as rolling or pressure), observed changes in the strength characteristics under friction loading follow a cyclic pattern. Cycles in structure evolution have been established by ferromagnetic resonance [1-4], electronic microscopy, electron diffraction analysis [1, 3], X-ray crystal analysis [3], and intensity of wear measurement [4].

This paper continues the examination of the strengthening and destruction mechanisms in the crystalline structure of friction-loaded surface layers of nickel consistent with the oscillating kinetics of the strength characteristics.

Experimental details

The experimental method was similar to one described in [5]. The dislocation structure in the nickel-molibdenum pair under friction loading was studied using the ferromagnetic resonance method (FMR) and electronic microscopy. FMR methods appear to be the most appropriate for the study of the dislocation structure in ferromagnetics. At external electromagnetic field frequencies of 9400 MHz (ν = 9400 MHz), the broadening of the FMR absorption lines in a deformed ferromagnetic is caused by elastic stress fields, while small-scale defects (such as impurity atoms or vacancies) and large-scale defects (e.g. cracks or pores) lie outside the absorption spectrum [5, 6]. In the above works, a linear relationship was established between the breadth of the FMR line (ΔH) and dislocation density (ρ). Another advantage of the method is that the depth of penetration in the skin metal layer (δ) is 10^{-7} – 10^{-6} m at external electromagnetic field density of 9400 MHz. This makes the method sufficiently selective and sensitive when used for the study of defect microstructures in thin surface layers, particularly under friction loading, when the depth of the surface layers affected by plastic deformation is comparable to the thickness of the skin layer [6]. Consistent with the above conclusions, the observed cyclic function $\Delta H = f(t)$ reflects the dislocation structure kinetics in friction-loaded surface layers of nickel.

Results and discussion

In the cited works, interpretation of the cycles in the function $\Delta H = f(t)$ is made within the framework of the loop theory of surface wear and destruction under friction [7]. Analysis of the electron microscopy images suggests that in each cycle, the surface layer structure undergoes extreme strengthening (with maximum dislocation densities (ρ) of $10^{16}m^{-2}$), before returning to the original specimen structure with minimum dislocation densities ($\rho = 10^{13}м^{-2}$) [1]. In this scenario, there is no high-dispersion phase, and grain size approaches that of the original specimen (only two cycles of the function $\Delta H = f(t)$ were considered in the study). A comprehensive examination of the cited works suggests that destruction and selective peeling of the entire layer of the dispersed material occur within a single cycle. Repetition of the cycles is due to the dispersion and wearing of the lower layers [1, 8].

Figure 1 (Curve 1) shows the function $\Delta H = f(t)$ during extended periods of friction loading. The changes in the strength characteristics in friction-loaded metal occur in two observable phases. In phase 1, the oscillating curve (uniquely related to dislocation density) rises to a peak, where a critical level of elastic strain energy is achieved. In each new cycle, the effects of residual deformations in the underlying material are seen at the minimum points, along with continual increases in the absolute values of the strength characteristics at the peaks. Bearing in mind the layered pattern of the strengthening and micro-destruction processes, this means that each new cycle starts at a progressively higher level of dislocation density. These peculiarities in the strength property kinetics are indirect evidence that microstructure transformation in friction loaded material is related to the duration of exposure. Peak strengthening at the end of the first friction loading stage, ultimately leading to sufficient levels of material dispersion, seems to be the prime cause of the deep break in the monotonously ascending cyclic curve. Subsequent phases in the course of the surface layer strengthening and destruction processes are characterized by these breaks in cyclicity.

Wear intensity kinetics (I) was also studied in connection with the microstructure transformations of the surface layer (Figure 1, Curve 2). By analysing the relationships in Figure 1, it may be concluded that the periodic surges in the intensity of wear coincide with the minimum values of dislocation density in the cyclic curve $\Delta H = f(t)$. This suggests that wear product generation may be concentrated in time, a process determined by the specifics of elastic stress deformation in the surface layers of solid-state bodies under friction. After maximum dispersion is reached (Curve 1, Point A), a series of deep declines in dislocation density occurs (points M, N, K), coinciding in time with extreme surges in wear intensity (by two orders of magnitude), exceeding the maximum values observed during the

run-in wear phase. For clarity, the intensity of wear (including at the peak point E) caused by layered microdestruction at the surface run-in phase (I = 10-7-10-6 kg/m) will be referred to as microdestruction, and the abnormal peaks in wear intensity coinciding with the deep declines in the strength parameters (points F, G for which I = 10-4 kg/m), will be referred to as microdestruction peaks. Further insights into the processes of surface layer destruction in nickel can be obtained by assessing the size and number of the wear particles using the optical method. Mean size of destruction fragments shows a tendency to decrease as loading time increases. The kinetics of this process was as follows. Mean particle size decreased from ≈ 4 μm at t = 25,2 ks to 3,2 and 2,5 μm, respectively, at a t = 93,6 ks and t = 126,8 ks. The percentage distribution of the particles at t = 25,2 ks was ≈ 80 % for small particles (d=2,5 μm), ≈ 20 % for medium sized (d=3,2 μm) and large particles (d ≥ 4 μm); at t = 93,6 ks, these percentages were 90% for small particles and ≈ 10% (medium sized and large particles); at t = 126,8 ks, there were 90 % of small sized, and 10% of medium-sized particle, with no large particles present. The progress of material embrittlement during friction loading is related to this dynamic, if indirectly.

Thus, along with the cycle of selective layered micro-peeling in the surface layer, another cycle is observed, related to the accumulation of elastic stress energy in the underlying layers and the accelerated micro-destruction of these layers under friction loading (represented by the dashed line in figure 1). Electronic microscopy analysis was performed to compare the FMR results with data on microstructure changes in friction loaded surface layers of nickel.

As suggested by the microstructure analysis, a highly dispersed crystal structure is formed at the initial stages of friction loading, with block size ≈ 10-2 μm. (Figure 2). The nanostructuring of the thin surface layer alters the deformation mechanism within the layer to a significant degree. As seen from Figure 2, a growing tendency for the dislocation clusters to texturise unidirectionally can be observed already at the beginning of the loading. Fine microcracks emerge within this microstructure. Typical dimensions are 0,01 μm in diameter and 0,1 μm in length (Figure 2, Insert at t =0,9 ks). As indicated in [9], these cracks may be characterized by dislocation defect nucleation even in isolated slip lines, independent of the activation effect from the same or adjacent slip line. As seen from the figures, a series of open microcracks spreads unidirectionally across isolated dispersion chains in the dislocation clusters. According to [8], the origin of these micropores related to the action of two mechanisms: 1) vacancies merging into micropores under the action of temperature gradients in the actual contact points; 2) formation of surface slides and breaks, caused by the dislocation transformations; these specifically involve dissociation of dislocation loops, and dissociation of dipoles to form edge dislocations of opposite orientation; the surface shifts, extrustions and intrusions caused by these edge dislocations act as powerful stress concentrators [10].

Intrusions may obviously be identified with surface microcuts; in the presence of these microcuts, tensions are concentrated at the tips, and vertical and horizontal elastic stresses are generated [11]. During the formation of the microcut, normal stresses greatly exceed the shear stresses in magnitude, and the tendency towards proliferation of cleavage cracks is readily observable, the result of constrained material flow in the small localized space at the tip of the cut. This type of destruction is related to atomic plane breaking by elastic stresses perpendicular to the fracture surface. In sufficient numbers, microcracks coalesce into major cracks through macroscopic material destruction (Figure 2). However, given the variety of the factors active during friction loading, and the wide variations in stress and temperature gradients, brittle fracture mode may be transformed into elastic or complex fracture modes.

Further microstructure transformation is marked by increased material fragmentation through slide line formation (Figure 3, A). At the initial stages of the process, the microstructure resembles the common structure of a stable fatigue line with uniform distribution of dipole dislocation clusters. Continued loading transforms these lines into centres of localized deformation as extrusion material (meso-stripes). These meso-stripes are uniquely characteristic of the elastic strengthening proccesses in nanostructure surface layers of nickel under friction loading. The boundaries of these structure elements and the cleavage pattern have a clearly localized structure (Figure 3, B, arrows). This conclusion, however, cannot be made with certainty. As suggested by the data from a number of microscopy images, the formation and migration of localized meso-stripes of elastic deformation, a dissipative subgrain structure can be observed within the surface layer areas adjacent to the micro-stripes, with block sizes of 0,08 – 0,1 μm (Figure 4). This microstructure is charactarised by quasi-uniform distribution of the dislocation clusters. According to [12], the formation of dissipative subgrain and nanocrystal structures in nickel and nickel alloys leads to significant increases in cyclic hardening stress, while also raising cyclic loading strength and decreasing cyclic crack growth parameters. This is apparently due to main microcrack generation along and across the textural formations (Figure 4, arrows). The mechanism through which these microcracks are generated is related in complex ways to the above-mentioned properties of dissipated subgrain structures.

Consistent with Figures 2 the localization area of the slide lines is rich in crack nuclei (as marked by the arrows). Crack formation can be attributed to crystalline grid dispersion. Alternating deformation intensifies dispersion, and causes slide packets and cracks to form at lower stresses than under static deformation. The most probable explanation is that during the transitional friction loading phase (t = 54 ks), the fatigue cracks are formed exclusively in areas where the slide traces are located.

According to [13, 14], the deformation and destruction processes in friction loaded surface layers may be interpreted from the perspective of mesomechanics. Plastic strain localization in the contact areas of conjugate planes creates a mesoscopic layer. The structural evolution of this layer defines the strengthening and destruction mechanism in the material under friction. As suggested in these works, this structure evolves into a highly dispersed polycrystalline structure with maximum strength performance. Consistent with the physical mesomechanics scale level scheme presented in [15], surface layer strengthening and destruction at maximum dispersion (Figure 1, Position A) may be identified as global loss of shear stability in the surface layer after nucleation and migration of localized plastic deformation meso-lines (Figure 5, A and B). Fragmented layer formation, affecting with time the underlying layers to depths of tens of micrometers was the key determinant of the destruction process [16]. Surface layer destruction led to catastrophic wearing, consistent with [4].

The effect of growing porosity in the material during the destruction process should also be noted. Prior to the destruction phase, the formation of multiple micro-pores along and within the tabular structure boundaries under friction loading (Figure 5, A and B) is related to thermal cycling in friction contact areas, vacancy diffusion mobility, and conglomeration. Under applied stress action, the micropores become stress concentrators, potentially causing transcrystalline and intercrystalline destruction (Figure 5, B, positions A and B). High porosity is observed within the tabular microstructure (Figure 5, A). The degree of porosity reaches 15% of the entire observation area.

The presence of stable slide lines determines the hardening and destruction kinetics in the surface layer. As established in [8], the slide lines are transformed into tabular structure elements along the boundaries formed by elements of crystalline plane non-alignment and acting as elastic stress concentrators. As seen from Figure 6, microcracks are formed along the boundaries of the slide lines within a highly fragmented band

structure (arrows). Localised peeling and shifts along the slide line boundaries leads to main microcrack formation (Figure 7, arrows). These processes are facilitated by temperature surges at the friction contact points.

Highly dispersed meso-substructure areas can be observed during the maxium hardening phase (Figure 1, Position A), explained by dislocation shifts and micropore formation along grain-oriented structure boundaries (Figure 8, A and B). Elements of intercrystalline destruction at this phase are considered in [8]. In this paper, the mechanisms involved in this type of destruction are studied by electron microscopy analysis. In the unique conditions of friction loading, determined by temperature fluctuations and alternating interactions of extrusion and intrusion, the processes of recuperation, dynamic recrystallisation, tenacity and creep may be initiated in the surface layer. In this respect, grain boundaries become vulnerable to microcrack and pore formation under the influence of combined loading pores and temperature gradients under friction loading. They act as primary areas of vacancy drain and as vacancy sources. This causes pore formation during vacancy conglomeration and pore growth during migration of the pore defects to the surface. Through the action of combined stresses and temperature surges, the surface metal layer may approximate plane flow conditions, enabling slippage along the grain boundaries. The number of pores and microcracks is proportionate to the degree of the slippage [17].

At the rapid destruction stage, multiple micropores were found inside the blocks and along block boundaries. Through observed micropore coalescence, near-spherical cavities are formed. Localised deformation within the micropores creates fault-like shear cracks. The observed direction of the destruction is also related to material strength reduction along the grain boundaries.

The microstructure at the minimum points of the cyclic curve $\Delta H = f(t)$ is shown in Figures 9. The images show no elements of dispersed microstructures, apart from several isolated dislocations at the residual traces of stress-induced destruction that exhibit oscillating contrast. Thin extinction contours indicate the presence of crystalline grid torsion elements facilitated by the action of torsional modes in the overlying layer. In this process, the structure in Figure 9 is similar to that of the undeformed specimens. This conclusion is further supported by the similarity of the electron microscopy images (Figure 9, insert) is further evidence to this conclusion.

Conclusion

In friction-loaded metals, intense grain refining occurs to nano-range sizes, with quasi-uniform distribution of the nanocrystalline blocks over the volume of the surface layer. In the course of the friction loading, the microstructure is transformed, first into texturized elements shaped as thin dislocation lines, and subsequently into slide lines and a lamellar mesoscopic structure. Microcracks are generated along the boundaries of these lines that coalesce into main cracks, accompanied by the formation of material wear blades.

Numerous micropores within the deformed grid and along the grain boundaries - accompanied by micropore coalescence - create centres of transcrystalline and intercrystalline destruction. Extended exposure to friction loading leads to progressive roughening of the surface layer, facilitated by proliferation of the centres of destruction. At the maximum dispersion phase, the roughening affects the underlying layers; in combination with advanced surface layer roughening, this process leads to rapid increases in the amount of loose material peeled as a whole layer. Escalation of this process leads to isolated release of the destruction products from the friction contact zone.

The curves shown in Figure 1 confirm that each minimum point in the function $\Delta H = f(t)$ has a corresponding maximum in the intensity of wear cycle, suggesting time localization of the wear product generation process. Visible troughs in the ΔH curve and the major surges in wear intensity corresponding to those troughs suggest that the embrittlement process penetrates the underlying material layers latent stress energy. In the process, thin layer destruction (and peeling at the end of each cycle) precedes the peeling of a thicker underlying layer (tens of microns in length) during the buildup of strain energy, which corresponds to intensive destruction.

In sum, the results of the study confirm that destruction of thin surface layers of metal under friction loading follow a bladed and layered pattern and is localized in time.

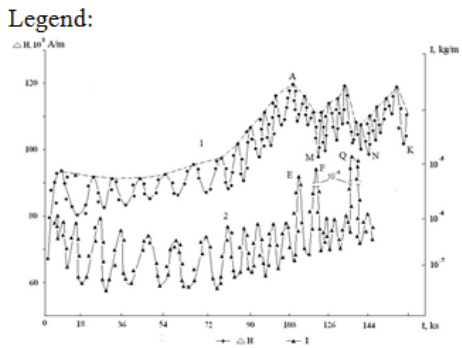


Figure 1 – Relationship of the ferromagnetic resonance line width of the ΔH (1) and the intensity of wear of I (2) from friction loading time t.

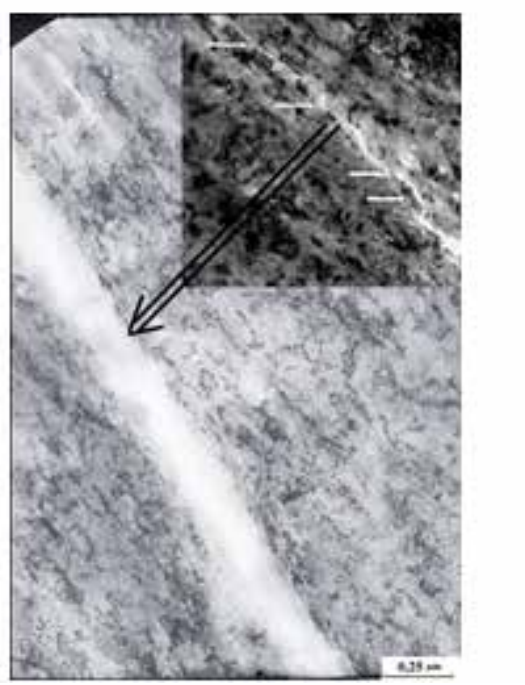


Figure 2 – Destruction of the submicrocrystalline structure (t=5,4 ks).

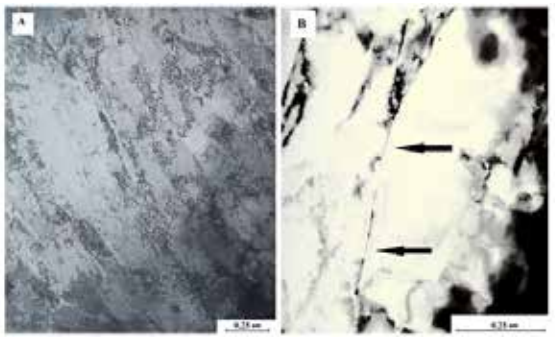


Figure 3 – Initial stage in the formation sliding strips (A, t=18,5 ks). Destruction by a chip on lamel boundaries (B, t=25,2 ks).



Figure 4 – Subgrain microstructure (t=50,7 ks).

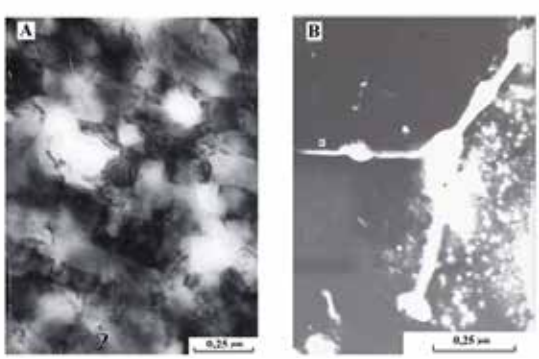


Figure 5 – Porosity in lamellar structure (A, t = 9,2 ks, B =113 ks).

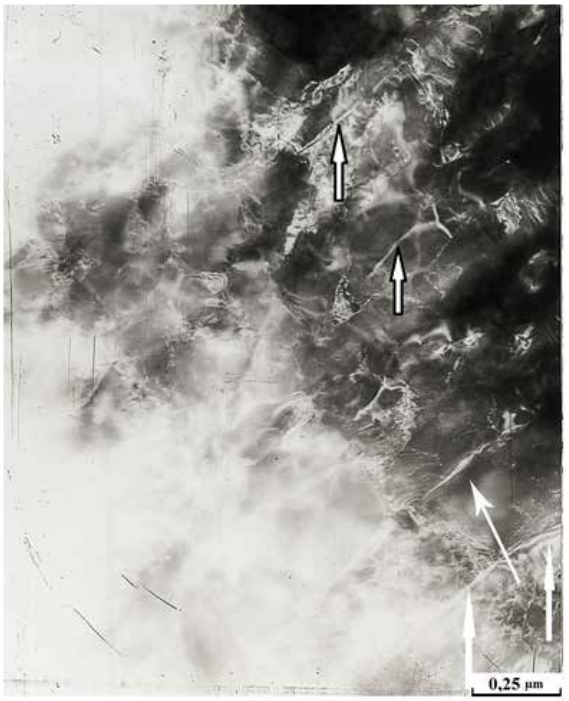
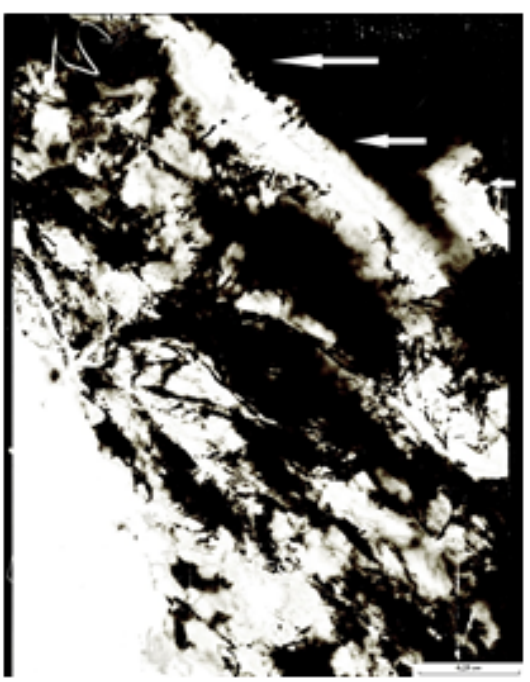


Figure 6 – Formation of microcracks in the slide lines.



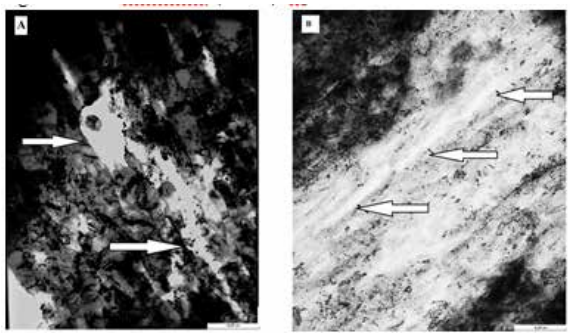


Figure 8, A and B – Microstructure of nickel at maximum hardening (t = 113,4 ks).

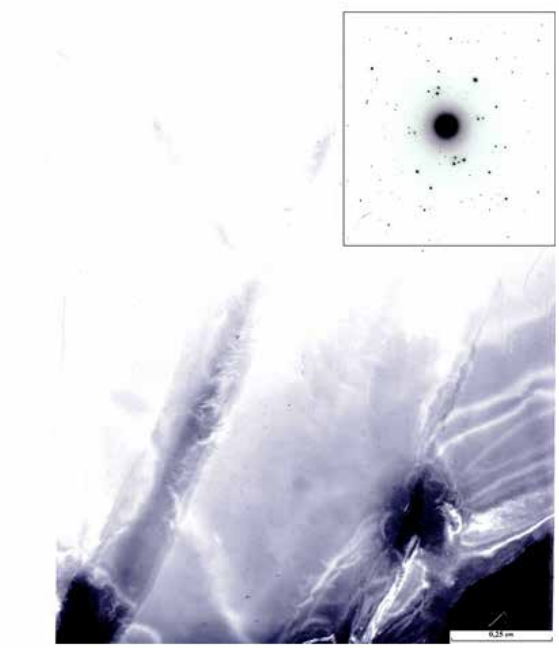


Figure 9 – Microstructure at the minimum points of the cyclic curve Δ H = f(t) at t = 123 ks

REFERENCES

[1] V. G. Pihcnuk, B. A. Savitskiy and A. S. Bulatov. Osobennosti izmenenija dislokacionnoj struktury nikelja pri trenii [Changes in the Nickel Dislocation Structure under Friction]." Poverhnost'. Fizika, himija, mehanika. No. 9, 1983, pp. 72-75. | [2] V. G. Pinchuk, S. V. Korotkevich and S. O. Bobovich. Strukturnye aspekty mikroplasticheskoj deformacii i razrushenija metallov pri trenii [Structure of Micro Plastic Deformation and Metal Destruction under Friction]. Deformacija i razrushenie materialov. No. 9, 2007, pp. 23-28. | [3] V. G. Pinchuk , B. D. Harhasov, V. V. Torop, Ju. Gerberger. Dislokacionnaja struktura nikelja pri trenii [Dislocation structure of nickel under friction]. Trenie i iznos. Vol. 2, No. 3, 1981, pp. 389-392. | [4] V. G. Pinchuk, Ye. G. Shidlovskaya. Vzaimosvjaz' mikrostrukturnyh izmenenij s kinetikoj iznosa poverhnostnogo sloja metalla pri trenii [Interrelationship between microstructural changes and the kinetics of wear in metal surface layers under friction]. Trenie i iznos. Vol. 10, No. 6, 1989, pp. 965-972. | | [5] A. S. Bulatov, V. G. Pinchuk and M. B. Lazareva. Zavisimost' shiriny linii FMR ot plotnosti dislokacij v nikele [Relationship of FMR line width with dislocation density in nickel]. Fizika metallov i metallovedenie. Vol. 34, Issue 5, 1972, pp. 1066-1069. | [6] V. G. Pinchuk, S. V. Korotkevich, S. O. Bobovich, Yu. M. Pleskachevsky. Issledovanie dislokacionnoj struktury ferromagnetikov metodom ferromagnitnogo rezonansa [Investigation of the dislocation structure of ferromagnetics using the FMR method]. Pribory, instrumenty, materialy. Vol. 15, No. 4, 2010, pp. 108-112. | [7] N. P. Suh. The Delamination Theory of Wear. Wear. Vol. 25, No. 1, 1973, pp. 111-124. | [8] V. G. Pinchuk, S. V. Korotkevich. Microstructure and Strength Properties of Submicro and Nanocrystalline Nickel under Friction. Modeling and Numerical Simulation of Material Science. No. 3, 2013, pp. 8-13. | [9] Vladimirov V.N., Hannanov Sh.H. Actual zadachi teorii zarogdeniya dislocation treschin // Fizika metallov i metallovedenie. Vol. 30, No. 3, 1970, pp. 490-510. | [10] P. Neumann, "Coarse Slip of Fatigue" Acta Metallurgy. Vol. 17, №9, 1969, pp. 1219-1225. | [11] J. D. Embury, N. J. Petch, A. E. Wraith, "The Fracture of Mild Steel Laminates. Trans. AIME. Vol. 239, 1967, pp. 114–118. | [12] B. Moser, T. Hanlon, K. S. Kumar et al. Cyclic Strain Hardening of Nanocrystalline Nickel. Scri. Mater. Vol. 54, No. 9, 2006, pp. 1151-1155. | [13] S. Yu. Tarasov, A. V. Kolubaev, "Effect of Friction on Subsurface Layer Microstructure in Friction and Martensitic Steels. Wear. Vol. 231, 1999, pp. 228-234. | [14] V. Panin, A. Kolubaev, S. Tarasov, V. Popov. Subsurface Layer Formation During Sliding Friction. Wear. Vol. 249, 2002, pp. 860-867. | [15] V. E. Panin, A.V. Panin, "Scale Levels of Plastic Deformation and Destruction of Nanostructural Materials. Nanotehnika. Vol. 3, 2005, pp. 28-42. | [16] S. Yu. Taracov and A. V. Kolubaev, Development of deformation at various scale levels to surface layers under friction. Deformation and fracture of materials. No. 1, 2008, pp. 21-27. | [17] J. Intrater and E. Machlin, "Grain Boundary and Intercristalline Cracking," Acta metal. Vol. 7, No. 2,1959, pp. 140-142. |