EXCHANGE OF EXPERIENCE

INSTRUMENTAL INDENTATION FOR EXAMINING
THE PROPERTIES OF SECONDARY STRUCTURES
OF METALS AND COATINGS

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The structure and properties of the surface layers of metals and coatings in the initial state and after friction contact are examined. Substantial increase in the hardness of surface layers after friction is mainly associated with the formation of nanostructure due to shear strains. Deformation does not noticeably change the properties of surface layers when the nanostructure has been formed in the initial material. The relationship is established between the wear resistance and the following properties of the initial coatings and secondary structures: plasticity, stress relaxation coefficient, and their structural component.

Keywords: coating, deformation, secondary structures, hardness, plasticity characteristic, stress relaxation coefficient, wear resistance.

Currently, coatings are widely used to increase the life of various products, including friction pairs. Modern technologies allow changing the structure, phase composition, and properties of coatings so as to endow them with the necessary combinations of physical and mechanical properties.

In friction, as well as in other processes of deformation, the structure of the surface layer changes, which is accompanied by structural adaptation. Some of the structures forming in friction exist virtually indefinitely in a metastable state even after cessation of friction, others gradually, with different periods, relax into the equilibrium state, and still others disappear almost immediately after the termination of friction. Among the other features of frictional interaction is the locality of deformation during friction, which entails nonuniform surface hardening.

The changes occurring in frictional contact of metal–metal pairs are studied in many publications [1–3], which discovered the formation of secondary structures, including oxygen-doped ones [4]. It should be noted that the secondary structures are a sophisticated object of research. Their properties are mainly determined by analyzing the chemical and phase compositions. The mechanical characteristics are difficult to determine because of small thickness (2–4 μm).

The objective of the present paper is to examine the influence of frictional contact in coating–iron and metal–iron pairs on the physical and chemical changes in the surface layer, to assess the level of these changes, and to ascertain their effect on service properties.
MATERIAL AND EXPERIMENTAL PROCEDURE

The objects of study are chromium monocrystal, 65G steel, VТ-1-0 titanium, Ti₃SiC₂ laminate, and chromium-based coatings produced using different techniques.

To examine the properties of secondary structures, we used the method of indentation under low loads with plotting indentation diagrams, holding under a constant load, and unloading. The Oliver–Pharr technique [5] allows determining the hardness and elastic modulus at the indentation point. Moreover, indentation diagrams can be used to determine material characteristics such as the plasticity and stress-relaxation coefficients [6].

Indentation was carried out at room temperature using a Berkovich diamond pyramid. The rate of dynamic loading was 0.01 to 0.3 N/sec. After the continuously increased load had reached its maximum level, it was held for 50 sec to examine the effect of holding under a constant load. The errors of determining the load $R$ and indentation depth $h$ were $10^{-3}$ N and ± 5 nm, respectively.

Figure 1 shows an indentation program, where $h_{\text{max}} = h_{\text{s}} + h_{\text{c}}$ is the indentation depth after loading to $P = P_{\text{max}}$ and subsequent holding at that level until the stop of the indenter (cessation of creep). It includes the elastic strain $\varepsilon_{\text{el}}$, the plastic strain $\varepsilon_{\text{res}}$, and the elastic deflection $\varepsilon_{\text{el,def}}$ near the indenter. Here $h_{\text{s}}$ is the design indentation depth (determined by the Oliver–Pharr technique) that represents the elastic deflection of the material around the indenter under the maximum load; $h_{\text{c}}$ is the design indentation depth (determined by the Oliver–Pharr technique) at which the indenter and the material are in contact until the maximum level of loading ($P_{\text{max}}$) is reached (at this depth, the indentation obeys the law of conservation of similarity under any load). The indentation depth $h_{\text{res}}$ is the one remaining after complete unloading ($R = 0$). It includes the plastic strain ($\varepsilon_{\text{pl}}$), the failure strain near the indenter ($\varepsilon_{\text{f}}$), and the remaining elastic strain ($\varepsilon_{\text{el}}'$). Thus, the residual strain $\varepsilon_{\text{res}} = \varepsilon_{\text{el}}' + \varepsilon_{\text{pl}} + \varepsilon_{\text{f}}$.

In addition to the determination of Oliver–Pharr hardness, we can calculate the work ($A_{\text{disp.mat}}$) done by the indenter to displace a volume of material (which is found from the indentation diagram bounded by the lines $OABh_{\text{c}}$) to determine hardness considering that it is equal to the work done to displace a volume of material. In our case:

$$H = A_{\text{disp.mat}}/h_{\text{c}}^{\text{3}} = (A_{\text{tot}} - A_{\text{s}})/h_{\text{c}}^{\text{3}}$$ (1)

Calculating the work, we can find the contribution of the plastic strain to the total work done to move the indenter:

$$K_{\text{pl}} = A_{\text{tot}} - A_{\text{el}}/A_{\text{tot}}$$ (2)

where $A_{\text{tot}}$ is the total area under the indentation curve; $A_{\text{el}}$ is the area under the indentation curve bounded by the lines $h_{\text{res}}Bh_{\text{tot}}$. 

**Fig. 1.** Indentation diagram

**Fig. 2.** Hardness of surface layers (regardless of elastic deformation) of titanium alloy versus indenter load: 1) initial state; 2) 90% rolling; 3) friction; 4) ECAP

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