AIMS Materials Science, 7(4): 453–467.
DOI: 10.3934/matersci.2020.4.453
Received: 05 May 2020
Accepted: 21 July 2020
Published: 03 August 2020

http://www.aimspress.com/journal/Materials

Research article

Specialties of deformation and damage of the topocomposite on a ductile substrate during instrumental indentation

Nikolay A. Voronin*

Department of Tribology, A.A. Blagonravov Institute of Machine Science Russian Academy of Sciences, Mal. Kharitonevsky per. 4, Moscow 101990, Russian Federation

* **Correspondence:** Email: voroninn@inbox.ru; Tel: +74991353558.

Abstract: We studied the nature of deformation and damage of topocomposites with instrumental indentation. The topocomposite is considered as a layered system consisting of a solid thin coating coherently bonded to a substrate of a ductile solid material. The object of the study is a topocomposite that consists a coating of titanium nitride deposited by magnetron sputtering in vacuum on a substrate of aluminum alloy. Comparing the balance of energy spent at all stages of indentation using model and experimental diagrams of indentation, the contribution of work dissipated on the deformation to the layered system, on the interfacial delamination at the coating-substrate interface and on the formation of through circular cracks in the coating is estimated. The research results can be used for a qualitative estimation of the efficiency of functional surfaces of machine parts hardened with coatings and for calculating the characteristics of deformation and damage.

Keywords: thin coatings; topocomposites; ductility; instrumental indentation; cohesive model; interfacial fracture; damage

1. Introduction

The instrumental indentation has now become one of the indispensable tools for evaluating the mechanical characteristics of compact heterogeneous materials and hardened surfaces, primarily the layered systems such as topocomposites [1]. This modern technique allows us to measure the hardness, modulus of elasticity, plasticity, adhesive strength and fracture toughness of coating in a wide range of thicknesses [2,3]. The results of tests for pressing the diamond pyramid into the hard surface are presented in a form of indentation diagrams—the loading curve and the unloading curve. The nature of deformation, damage and fracture of the test sample is shown on the loading and unloading curves in the form of specific changes in the curves (slopes, jumps, breaks, etc.) of the indentation diagram. In connection with the diversity of the material structures of the substrate and the coating, imperfections in the structure of materials in the area subjected to contact effects of the indenter, the difference geometry and manufacturing precision of the indenter, as well as loading conditions and environmental conditions, the interpretation of the nature of damage to the coatings of topocomposites with the indentation diagrams is extremely difficult. This primarily applies to the loading and unloading curves of indentation diagrams of surface-layered systems, which have features that differ sharply from traditional diagrams of deviations from the smooth change of curves from the depth of indentation. Such signs for a number of typical types of coating damage are straight sections ("plateaus") on the loading and unloading curves, the so-called "pop-in" and "pop-out" and a sharp change (curvature) of the trajectory of the unloading curves [3–9]. In studies on pressing spherical and pyramidal indenters into thin solid coatings it is often described the effect of interfacial delamination that is not accompanied by the destruction of the coating with the formation of cracks [4,6–9]. At the same time, the unloading curves of the indentation diagrams have a noticeable characteristic linear or non-linear unloading section with a sharply different slope, in relation to the slope of the unloading curve, which is observed at the initial section of the unloading curve. A specialty of the mechanical behavior of such layered systems is the delamination at the interface of the coating-substrate established by a number of researchers [4,6]. It is also noted in [8,9] that circular cracks around the indent appear in the coating together with interfacial delamination under certain circumstances. To assess the damage of such coatings, an analysis of the unloading curve associated with the elastic recovery of the components of the layered system and the elastic recovery of the detached coating is used.

In the indentation diagrams [4,6,7] obtained for coatings on elastic substrates, the delamination of the coating from the base appears as a linear section on the unloading curve. Linear recovery of the unloading curve is associated with elastic recovery of the detached coating due to the loss of the coating bond with the substrate under the indenter tip. It is assumed that the coating remains attached to the edge of the contact and as the load is removed from the indenter, the coating straightens out like a flat elastic membrane.

In the indentation diagrams [8,9] obtained for solid elastic coatings on ductile substrates, the delamination of the coating from the base appears as a non-linear section on the unloading curve. This type of unloading curve is associated with elastic interfacial delamination at the interface of the layered system due to the loss of bond of the coating with the substrate not only under the top of the indenter, but also at a distance from the center of load application, reaching up to 2–3 times the diameter of the imprint. The model of cohesive zone failure is used to describe the origin and destribution of a crack in continuum mechanics [10]. According to this model, a side crack is formed in a solid material when it is pressed in. When the load is removed a delamination of the material occurs. The origin of the crack and subsequent delamination are caused by tensile stresses that occur when the elastic recovery of the material is prevented by a zone of plastic deformation under the indenter. The model developed for compact solid materials has been widely used in recent years to describe failure in composite layered materials [8,9,11,12]. The strength of the interfacial connection depends largely on the internal properties of the coatings.

The authors of papers [9,11,13] believe that the effect of delamination at the interface is associated with the destruction of the layered system in the form of a plastic tangential flow of the base material at the coating-substrate interface during loading. It is difficult to evaluate the operation of interfacial delamination along the unloading curve due to the rather complex relationship between the elastic energy of the coating and the interfacial interaction energy at the boundary of the delamination. In this case, the elastic energy of the coating includes, in addition to the work of elastic deformation of the coating material as part of the layered system during unloading, also the work on delamination of the coating from the substrate, which is proposed to be approximated by the elastic energy of a centrally loaded disk with clamped edges at the boundary of the bundle. In paper [8], it was found by the finite element method using the model of cohesive zone failure that the interfacial lateral crack originates outside the contact zone in the tangential mode and extends a considerable distance from the indentation center. A section with a steep non-linearity was found on the unloading curve of the indentation diagram and it was established that the remaining depth of indentation depends on the size of the bundle. Two values of critical interfacial strength were calculated, the first, which suppresses the delamination process, and the second, which causes transverse destruction of the coating. The theoretical study of this work predicted the values of the strength of interfacial failure, at least by an order of magnitude different from the results of experimental studies [9,13]. In paper [9] it is shown that a significant difficulty in calculating the strength characteristic of interfacial failure is the determination of the critical load directly in the experiment due to the difficulty of visually determining the size of the delamination area. Determining the delamination radius without destroying the coating is possible only on transparent coatings, which dramatically reduces the range of materials and layered systems under study. Measurements made when critical indentation loads are reached, which lead to the fracture of the coating in the form of circular cracks around the indent, sharply reduce the accuracy of calculations, which include such parameters as the maximum and residual depth of indentation and the radius of the delamination area in power 4.

For layered systems with a ductile substrate, several types of damage occur in the process of indenting them, depending on the loading conditions, the presence of residual stresses, the nature of delamination, the conditions for implementing interfacial interaction during loading, which require the development of new methods for studying experimental indentation diagrams, and the interpretation of damage mechanisms and their relationship.

This work is aimed at investigating the specialties of experimental indentation diagrams obtained by instrumental indentation of a pyramidal tip into the surface of a layered system consisting of a thin solid elastic coating and a substrate made of a ductile material. The purpose of this study is to establish and substantiate the mechanisms of occurrence and development of all types of damage to the layered system during loading and unloading, depending on the value of the maximum (final) loading force during indentation. From the comparison of the energy balance of the indenting stages according to the model and experimental indentation diagrams, it is proposed to estimate the contribution of work utilized on all types of damage and fracture during indenting.

2. A solution of the problem

To solve this problem, we selected a research object in the form of a flat sample with a thin hard coating. The sample material (substrate) is an aluminum alloy D16T with a hardness of H_0 = 0.98 GPa and an elastic modulus of E_0 = 93 GPa. The coating material is titanium nitride (TiN) obtained by magnetron method. The coating thickness was 5 microns, the hardness H_1 = 15.2 GPa, the modulus of elasticity E_1 = 320 GPa. Measurements of the mechanical characteristics of the materials and studied surface of the layered system were carried out on the nanohardness tester NanoScan4D and 3D optical microscope Sneox (SENSOFAR, Spain). Experimental studies were conducted with the sample using a NanoScan4D in the form of indentation of a Berkovich diamond indenter under various loads and recording diagrams "load *P*-displacement *s*". The imprints after testing at each loading level were examined using an optical microscope. To analyze experimental indentation diagrams, we used theoretical (model) indentation diagram, which were constructed using the dependencies obtained in papers [14] and [15]. In paper [14], analytical dependencies are established that link the effective elastic modulus *Ec* and composite hardness *Hc* of the layered system with the corresponding parameters, characterize the "solo" values of the mechanical characteristics of the components of the layered system and the geometric parameters of the contact:

$$E_c = E_0 \Phi^{-3/2} \quad (1)$$

$$H_c = H_0 \left(\bar{\Phi}\right)^{1/2} (\Phi)^{-3/2} \quad (2)$$

where $\Phi = f\,(E_1/E_0,\ h/s)$—elasto-geometric parameter of the layered system, $\overline{\Phi}$ = f (E_1/E_0, H_1/H_0, h/s) is the limit elasto-geometric parameter of the layered system; E_1 and E_0 is the normal module of elasticity of the material of the coating and the substrate, accordingly; H_1 and H_0 is the hardness of the coating material and the substrate, accordingly. The elastic-geometric parameter Φ is an analytical weight function that describes the change in the elastic characteristics of a layered body from the depth of indentation *s* of the indenter, which is normalized by the thickness *h* of the coating. The range of existence of an elastic-geometric parameter $1 \le \Phi \le \left(\frac{E_0}{E_1}\right)^{2/3}$ for $0 \le h/s \le \infty$. The limit elastic-geometric parameter $\overline{\Phi}$ is an analytical weight function that describes the change in the plastic characteristics of a layered body from the depth of indentation of the indenter, which is normalized by the thickness of the coating. The derivation of weight functions and analytical Eqs 1 and 2 is described in more detail in paper [15].

The above Eqs 1 and 2 were used to construct the loading curve and the unloading curve of the model indentation diagram for the case of indenting an ideal pyramidal indenter into an ideal single-layer topocomposite. An ideal indenter was an indenter with an ideal top of the tip. An ideal single-layer topocomposite is a layered solid with known mechanical characteristics of the homogeneous components of the layered system, known coating thickness, and the presence of a coherent bond between the coating and the base that is maintained throughout the indentation test process. The methodology for constructing a model indentation diagram and analytical dependencies for constructing loading and unloading curves is described in detail in paper [15].

In this paper, we use the following analytical dependence to describe the loading curve of a model topocomposite that simulates an experimental sample:

$$P = E_c \left(\frac{1}{tg\alpha\sqrt{\pi}} \sqrt{\frac{E_c}{H_c}} + \frac{2(\pi - 2)}{\pi} \sqrt{\frac{\pi}{4}} \sqrt{\frac{H_c}{E_c}} \right)^{-2} s^2 \quad (3)$$

The unloading curve of the model topocomposite is described according to the following dependence

$$P = \left((\frac{2E_c}{\pi} \tan\alpha) \right) s^2 \quad (4)$$

where α is the equivalent angle of the indenter cone.

3. Results and discussion

The results of experimental studies of a coated sample in the form of indentation diagrams for various final loads of indentation are shown in Figure 1. The analysis of the obtained loading and unloading curves shows that with the increase in the final value of the indentation load, the type of unloading curve differs. As the final load value increases, the lower part of the unloading curve bends more and more towards the origin of the indentation diagram.

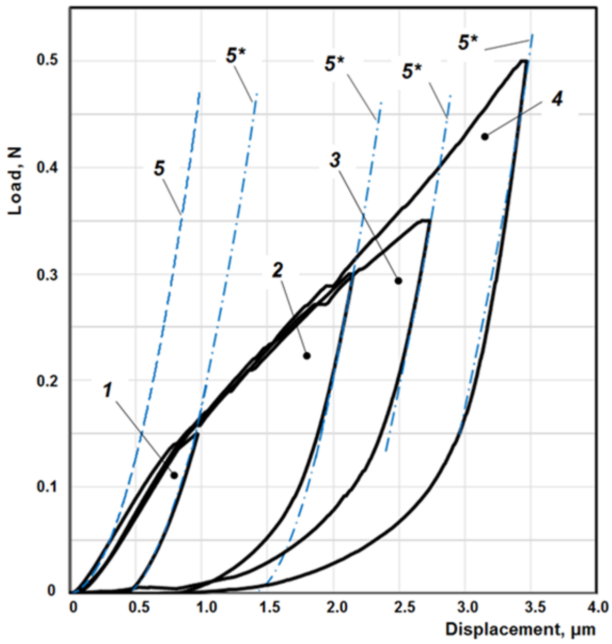

Figure 1. The view of experimental indentation diagrams at the final load value in N: 1–0.15; 2–0.30; 3–0.35; 4–0.50; 5—the unloading curve of the model layered system; 5*—offset on the axis of the abscissas unloading curves 5.

The results of visual studies of indenter imprints on the surface of the coating showed that under loads corresponding to the indentation diagrams 1–3 (see Figure 1), there are no discontinuities, bloating and cracks in the coating (Figure 2a). For the indentation diagram 4 (see Figure 1), circular cracks were found around the imprints at a distance from the center of the imprint about 1.5 times larger than the size of the circle described around the vertices of the imprint (Figure 2b).

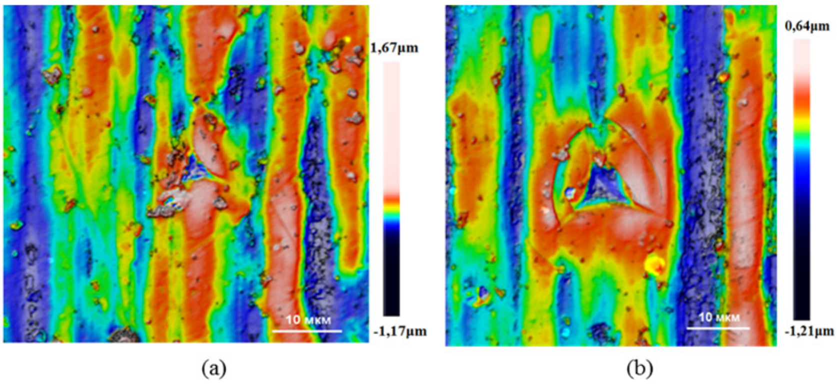

Figure 2. The view of indents obtained when the indenter loads with a maximum load of 350 mN (a) and 500 mN (b). Color scales—the height of the roughness of surfaces investigated.

From the literature [8,13], it is known that a strong curvature of the end of the curve of the unloading of the indentation diagram indicates the delamination of the coating from the base in the form of a membrane, the edges of which are fixed away from the imprint location. For the topocomposite studied in this work, the place of the maximum possible location of the edges of the attachment of the detached coating is indicated by circular cracks (see Figure 2b) found around the indentation imprint for diagram 4 (see Figure 1).

For relatively small final loads of indentation, the indentation diagram has a classic appearance, that is, there is no strong curvature and flatness at the end of the unloading curve in such an indentation diagram (see Figure 1, diagram 1). This indicates that there is no delamination of the coating during loading–unloading of such layered systems. For qualitative ranking of indentation diagrams with and without damage, we construct a model elastic deformation curve describing the theoretical unloading curve of a layered system with the mechanical characteristics of the component materials corresponding to the characteristics of the studied topocomposite. To do this, use the Eq 4. Such a curve, for the layered system under study, constructed from the origin of coordinates, is represented by a curve 5 (see Figure 1). Shifting the abscissas axis and combining the curve 5 with the points of maximum (final) load for the obtained diagrams, we obtain a series of curves 5*. According to the location of the model and experimental unloading curves relative to each other, we can determine the presence and type of damage, without conducting a visual inspection. So it is clearly seen that for diagram 1 (see Figure 1), the curve 5* completely coincides with the unloading curve over the entire range of load changes during the indenting process. For indentation diagrams 2 and 3, the overlap occurs in the initial part of the unloading curves and the lower sections of the corresponding curves 5* are located outside the contour of the indentation diagrams. For the indentation diagram 4, there is almost no overlap between the 5* curve and the unloading curve. The curve 5* is located inside the indentation diagram. The coincidence of the elastic deformation curve of the model layered system with the experimental unloading curves of the indentation diagrams indicates that there are no conditions in the indentation process that lead to any damage of the layered system and, in particular, delamination of the coating from the substrate. The coincidence of the curve of elastic deformation of a model layered system, only at the initial section of the experimental unloading curve of the indentation diagram, and the presence of a significant curvature of the lower section of the experimental unloading curve can be clearly interpreted as the presence of damage to the layered system, in which there is an interfacial bundle at the interface in the form of delamination of the coating from the substrate during unloading. In this case, the delamination of the coating is manifested in a larger scale of the destruction area due to additional damage to the coating-substrate interface during loading. A reason to talk about the gain (intensity) of the process of flaking of the coating and the presence of additional damage to the layered system is based on the comparison of the curves of unloading from this work with the curves of unloading of experimental indentation diagrams, given in a number of papers [4,7] on the interfacial destruction of layered systems only in the form of a stratification of the layered system with the size of the region bounded by the size of the indent. In these works, the lower section of the unloading curve of experimental indentation diagrams has a characteristic and different from the typical section—a section with a linear character of change with a decrease in the depth of indentation (applied to the indenter load), and the model unloading curve coincides in most cases with the experimental unloading curve. It is believed that the linearity of the unloading section and the coincidence of the unloading curves clearly indicates the elastic recovery of the detached coating as a flat membrane under the top of the indenter with a decrease in the load value.

The unloading curves of experimental indentation diagrams presented in this paper in Figure 1 (see diagrams 2 and 3) are characterized by a clear and significant variation in size and type at the lower part of the unloading curve. According to the research results presented in papers [8,9], the reason for this behavior is explained by the high plastic properties of the substrate material and the high elastic characteristics of the coating material. In these works, it is noted that the presence of such behavior of the layered system is caused by damage of the coating - substrate interface during loading during indentation. In paper [8], interfacial delamination in a layered system is theoretically studied using a model of cohesive zone destruction. The model of cohesive failure assumes the presence of a plastic flow of the base material at the coating-substrate interface on the periphery of the contact area during loading without loss of contact between the coating and the substrate. As a result of this damage, the adhesive bond is weakened and when unloaded, the coating peels off not only under the indenter, but also outside it, at a certain distance from the imprint. The curvature, rather than the linear appearance of the lower section of the unloading curve, can be explained by the presence of an influence on the elastic recovery of delaminated coating in the interface area beyond the actual contact spot of the indenter with the topocomposite. If we assume that the plastic flow of the base material at the coating-substrate interface weakens the adhesive bond of the coating to the base, we can assume that the delamination of the coating during unloading begins not under the indenter, where the adhesive bond is stronger, but at the edges of the area of damage to the interface created during loading. The model of elastic recovery of the coating as a membrane with a central loading and edges rigidly fixed on the periphery in this unloading scheme cannot be applied, since it is assumed that there are at least two circular areas of the membrane with different stiffness. The nonlinear form of the unloading curve in the area of coating rectification can be approximated as an elastic deformation of a membrane with a rigid center located at the contact point of the indenter with the coating [16].

The research results obtained in works [9,13] show that when certain values of the final load are reached during indentation, the elastic recovery energy of the coating is partially spent on the formation of through circular cracks away from the imprint. In this paper, the formation of circular cracks was observed on an experimental sample at a final indentation load of 400 mN (see Figure 2). An indirect and objective indicator of the occurrence of circular through cracks when indenting into a coating on a pliable substrate can be the picture of the location of the model unloading curve relative to the experimental one when they are superimposed on each other. Figure 1 clearly shows that the curve 4 of the model unloading drawn from the top of the experimental indentation diagram is located inside the diagram, although the unloading curves for the experimental indentation diagram and the model one coincide at a small initial section. To estimate the amount of work spent on damage to a layered system with a pliable substrate during indentation, we will analyze the work spent on the deformation process of experimental and model topocomposites by comparing the areas of the model and experimental indentation diagrams. To build a model indentation diagram we use Eqs 3 and 4.

Figure 3 shows the experimental 1 and model 2 indentation diagrams with a maximum load value of 400 mN. The comparison of the model loading curves *OA* and the experimental *OA** clearly shows that the maximum penetration depth of the experimental diagram is almost 0.325 microns greater than that of the model diagram. The presence of a deviation in the arrangement of loading curves in the model and experimental indentation diagrams for the ideal and studied topocomposites indicates a different amount of work spent on deforming layered systems with the same mechanical characteristics and under the same loading conditions. The area *OA*C** of the experimental indentation diagram corresponds to the total energy spent during indentation on elastic-plastic deformation of the layered system and damage to the layered system at the coating-substrate interface.

The area *OAC* of the model indentation diagram corresponds to the energy spent only on elastic-plastic deformation of the layered system during indentation. The difference of the squares *OA*C** and *OAC* is the amount of energy spent on plastic shift deformation of the substrate material in the interface area beyond the boundaries of the actual imprint under the indenter, which can be considered as a shear damage. The specified work is shown as square $A_{sh.dam}$.

A more thorough analysis of the loading curves of experimental indentation diagrams and their comparison with the results of experimental studies of indentation diagrams, for example [3,6,7,9] suggest that in the layered system under study in this paper, a lot of small damages are realized at the interface, which constantly occur as the indenter loads the layered body. They are implemented in the form of acts of cohesive destruction, which are practically not observed on the loading curve of the indentation diagram. Such multiple small shear deformations (pop-ins) are observed in the presented experimental indentation diagram. The number of visible shear moments is indicated by arrows in Figure 3.

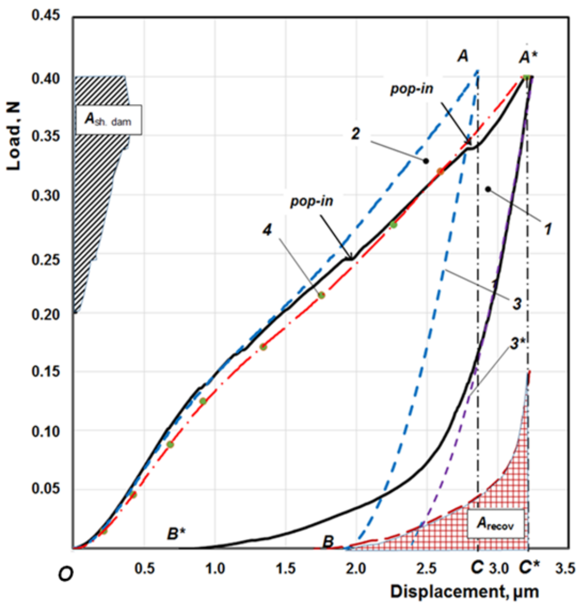

Figure 3. The view of experimental 1 and model 2 indentation diagrams. Description of symbols is in the text.

The term ductility in the technical literature mainly refers to the ability of solids to exhibit increased plasticity when deformed. A decrease in the yield strength of the deformable material indicates a greater ductility of the material. In our case, when considering the elastic-plastic deformation of the layered system during indentation, the mechanism of cohesive destruction of the substrate material at the interface can be considered as a condition for reducing the yield strength (hardness) of the substrate material. Elastic-plastic deformation during indentation takes place in a very small local area and a decrease in the yield strength of the substrate material can only be said in relation to a very small volume of the material, which is actually located in the area of significant contact stresses. In other words, we are not talking about changing the yield strength (hardness) of the entire volume of the substrate material, but about a certain small volume (layer) of the substrate material, which decreases the value of the yield strength (hardness) as it loads. Representing the changed volume of the substrate material as a layer with a reduced yield point, an experimental layered system can be considered as a two-layer topocomposite consisting of a coating (the first layer) lying on a layer with a reduced yield point (the second layer), which relies on the substrate material. Today, the model indentation diagram can also be theoretically calculate for a two-layer topocomposite, if we know the value of the new yield point and the thickness of the changed layer. If the new model diagram coincides with the experimental one, it will be possible to assert that the mechanism of cohesive destruction is applied objectively in the case of instrumental indentation based on the type of plastic shear. We made an approximate calculation using the Eq 3 for a single-layer topocomposite with a substrate material hardness value equal to 0.75 GPa (less than that of the experimental sample under study). It gave a loading curve (see Figure 3, curve 4), which is relevant in terms of the nature and values of the experimental loading curve, especially in areas close to the maximum indentation load, based on the assumption of a reduction in the yield strength of the entire volume of the substrate material. The noticeable difference between curve 4 and the experimental loading curve *OA** in the initial part of the loading curve is explained by the fact that when calculating the parameters of curve 4, the calculated dependence takes into account the final value of the reduced hardness of the substrate material even at low load values. While for experimental topocomposite at low loads, the effect of cohesive destruction is minimal and the substrate hardness is not lowered, that is why the experimental loading curve coincides with the model one in the initial part of the loading curves.

In the mechanics of deformation and fracture of solid bodies, the term compliant also occurs. In particular, when considering an infinite plate loaded with a concentrated force and lying on an elastic base, it is considered as a plate lying on a compliant elastic base. This approach implies that compliant indicates a significant difference in the elastic properties of the plate and the base. The authors of papers [17,18] apply this approach to solving the problem of axisymmetric frictionless indentation of a thin rigid film associated with a compliant substrate, which is considered a homogeneous isotropic elastic half-space. The problem is solved in a purely elastic formulation. Although the paper [17] takes into account the effect of elastic-plastic indenter insertion in the coating on the change in the elastic modulus of the layered system from the depth of indentation, it is not possible to use the calculation method proposed in papers [17,18] for our task, since we take into account the results of elastic-plastic deformation of both the coating and the base material. Even if you imagine that a layered topocomposite body made of a titanium nitride coating and an aluminum alloy substrate is an ideally elastic body, you can use the data from the curve 5* in Figure 1 to calculate the depth of indentation in the indentation center. This curve describes the elastic deformation of a layered body. At a load of 400 mN, the depth of indentation is approximately 0.8 microns. This depth is 6 times less than the coating thickness. This corresponds to the requirements of paper [17]. The calculation of the maximum deflection of the layered system according to the formulas of paper [18] using the elastic characteristics of the experimental sample of this work was ~0.03 microns. Figure 3 shows that the offset of the model indentation diagram relative to the experimental one is 0.325 microns. Even with the accepted assumption, consideration of the bending of the base of the topocomposite could have little effect on the analysis of the experimental indentation diagram.

In Figure 3, curve *AB* is the unloading curve of an ideal layered body, it describes the elastic recovering of a model layered system, that is, a system without the occurrence of layered delamination during unloading. Curve *A*B** is the unloading curve of experimental diagram of the introduction of a layered system, describes the elastic deformation of the layered system already subjected to damage in the form of cohesive destruction at the interface and includes elastic recovery of the actual material of the layered system and elastic recovery of the detached coating at the interface of the coating-substrate. Curve 3* is the unloading curve of the 3 model layered system combined with the unloading curve of the experimental layered system. Due to the absence of circular surface cracks in the experimental layered system, the initial part of the model unloading curve coincides with the experimental unloading curve. Area *B*A*C**—the work spent on elastic recovery of the actual material of the layered system and elastic recovery of the coating that has peeled off at the interface. The area of the figure bounded by the curve 3* and the straight *A*C** is the work spent on elastic recovery of the actual material of the layered system.

We estimate the contribution of energy spent on damage (interfacial delamination) of the interface due to elastic recovery of the coating that was detached along the interface. To do this, we subtract the area of the figure that characterizes the work spent on elastic recovery of the actual material of the layered system from the area *B*A*C**. As a result, we have a figure designated in Figure 3 as an A_{recov}, which characterizes the work spent on elastic recovery (interfacial delamination) of the coating that was detached at the interface.

Figure 4 shows the experimental 1 and model 2 indentation diagrams with a maximum load value of 500 mN.

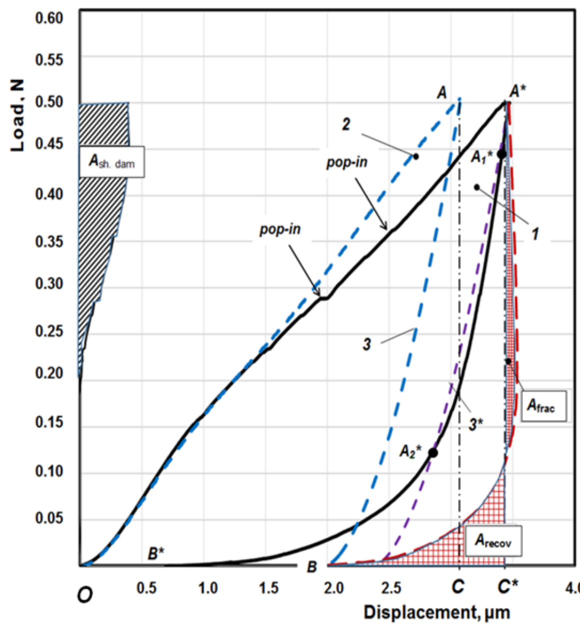

Figure 4. View of the experimental diagram 1 and the model indentation diagram 2. Description of symbols is provided in the text.

As follows from the analysis of the indentation diagrams shown in Figure 1, loading the sample before reaching the final load when indenting 500 mN, leads to the appearance of circular cracks around the indent (see Figure 2b). The experimental indentation diagram itself (see Figure 4) is almost identical in appearance to the indentation diagram obtained at a final load of 400 mN (see Figure 3). Comparison and analysis of deformation under loading by experimental and model loading diagrams leads to the possibility of calculating the energy of shear damage at the interface of the layered system under study. In Figure 4, it is indicated by the $A_{sh.\ dam}$. Similar to the indentation diagram obtained when loading up to 400 mN (see Figure 3), the indentation diagram shown in Figure 4 shows the presence of characteristic pop-in locations. Comparison of the unloading curves of the experimental and model indentation diagrams showed a difference in their location relative to each other in comparison with a similar comparison of the curves obtained at a maximum indentation load of 400 mN (see Figure 3). Curve 3*—the curve 3 model unloading, shifted along the axis of the abscissas and drawn from the top of the experimental indentation diagram is located inside the diagram, although at a small initial section the unloading curves for the experimental indentation diagram and the model one coincide. Using the method of calculating the amount of interphase energy spent on elastic recovery of the detached coating during unloading, proposed above in the analysis of the indentation diagram at the maximum final load of indentation of 400 mN, we will calculate the work spent on the damage that occurs when unloading the layered system, considered in Figure 4. The result of calculations and geometric representation was the formation of a figure consisting of two, the area of which we will designate as A_{recov} and A_{frac}. The total area describes the work of interfacial destruction of the coating, in which the work of A_{recov} is responsible for the energy of elastic recovery of the detached coating, and the work of A_{frac} is responsible for the energy of formation of circular cracks, fixed in tests by instrumental indentation (see Figure 4b). Comparison of areas responsible for elastic recovery during unloading for cases when the final loads during tests correspond to 400 mN (see Figure 3) and 500 mN (see Figure 4), it can be noted that with a lower final load of indentation, the elastic recovery area of the detached coating is greater than with a final load of 500 mN. This indicates that part of the elastic delamination energy of the coating in tests with a higher final load is used for cracking. It is clear that circular cracks occur at a distance limited by the size of the existence of damage to the interface and at the beginning of the removal of the load of indentation (unloading stage). Taking into account that the coating is in a curved state at the border of the sample exit to the surface and under conditions of a weakened adhesive bond with the substrate, when the load is removed, elastic deformations of the imprint size recovery increase the tensile stresses that lead to the generation of through cracks in the coating (Figure 5).

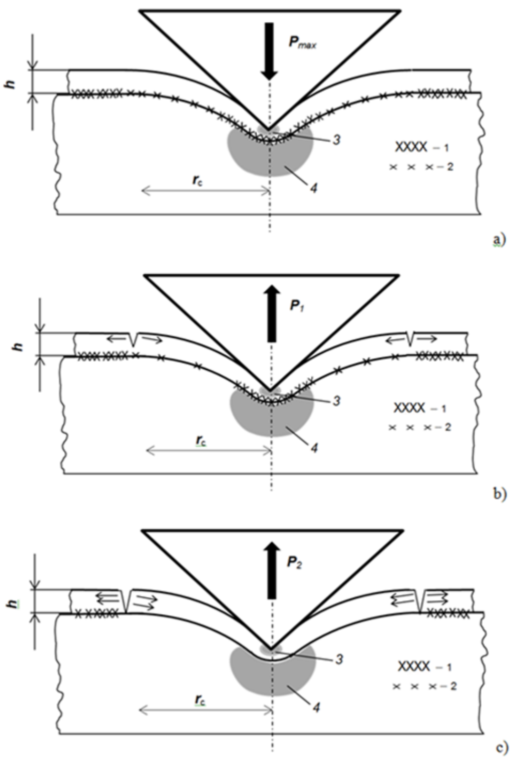

Figure 5. Schematic representation of the mechanism of formation of circular cracks in the topocomposite during unloading. 1—areas with a strong adhesive bond; 2—areas with a weak adhesive bond; 3—the area of plastic deformation in the coating; 4—the area of plastic deformation in the substrate.

The mechanism of circular cracks formation can be represented as follows. At the initial moment of unloading (see Figure 5a), which corresponds to point A^* in the experimental indentation diagram (see Figure 4) at the interface we can note the presence of regions with high (initial) adhesive strength of the coating to the substrate and areas with weakened adhesive bond resulting from cohesive destruction of the boundary of the layered system as a result of the process of loading a layered system with the indenter. The maximum load in contact corresponds to 500 mN and the indentation diagram at point A^* shows no signs of bending the loading curve, steps or breaks. As the load decreases, elastic recovery of the deformed imprint begins and the coating recovering reaction occurs. When the load P_1 is reached (see Figure 4, point A_1^*), the adhesive bond between the coating and the substrate weakens (see Figure 5b) and in the area of the coating located at a distance rc from the center of contact, the tensile stresses that increase due to elastic deformation of the layered system lead to the origin of transverse cracks from the coating surface. When the load is further reduced to the P_2 value (see Figure 4, point A_2^*) the elastic recovery forces of the layered system and the coating as a membrane lead to complete neutralization of the adhesive bond both under the indenter and in the area beyond it. Tensile stresses in the area where surface cracks occur ensure their growth to the coating-substrate interface (see Figure 5c). Further reduction of the unloading forces leads to delamination of the coating, which occurs unevenly due to the difference of delamination of the coating surface under the indenter and in the area beyond it. Under the indenter, elastic deformation and bending of the coating are limited by the speed of movement of the indenter, and in the indenter-free region of separation, the bending of the coating is limited only by the coating's own stiffness. This difference in coating recovery results in a non-linear form of the experimental unloading curve (see Figure 4, curve $A_2^*B^*$).

The analysis of the mechanism of circular cracks occurrence when certain indentation loads of topocomposites are reached allows us to explain the phenomenon of non-coincidence of the model unloading curve from the experimental unloading curve at the final part of curve (see Figure 1 diagram 4 and Figure 4). Since the origin of transverse cracks in the coating (see Figure 5b) and before they germinate to the coating-substrate interface (see Figure 5c), the adhesive bond between the coating and the substrate is reduced, which allows the coating to move with elastic recovery along the interface during unloading. It is known that the effective elastic modulus of a layered system (topocomposite) depends on the stiffness of the bond between the coating and the substrate [18,19]. When the adhesive strength at the interface decreases, the effective elastic modulus of the layered system decreases, the experimental load-indentation curve should be located to the right of the model load-indentation curve, which is observed in the indentation diagrams (see Figures 1 and 4). Thus, when comparing the experimental and model unloading curves, the location of the model unloading curve relative to the experimental unloading curve can clearly indicate the absence or appearance of circular cracks in layered systems with a substrate.

The work values calculated using the method developed in this study are not characteristics of strength or damage. However, the elastic recovery energy and the fracture energy can be used to calculate, respectively, the cohesive and adhesive strength, the fracture strength of the coating material, as well as the damage area in the case of cohesive failure without the formation of circular cracks. The author's next work will be devoted to these issues.

4. Conclusion

The features of experimental indentation diagrams obtained by instrumental indentation of a pyramidal tip into the surface of a layered system consisting of a thin hard coating and a substrate made of a ductility material are studied. The mechanisms of occurrence and development of various types of damage to the layered system during loading and unloading, depending on the value of the maximum (final) loading force during indentation, are established and justified. From the comparison of the energy balance of the indentation stages according to the model and experimental indentation diagrams, the contribution of work spent on all types of damage and destruction during indentation is estimated. A method has been developed for calculating the energy of shear damage at the interface during loading, the energy of elastic delamination of the coating from the substrate during unloading, and the energy of formation of through circular cracks in the coating applied to a ductility base. The research results can be used for a qualitative assessment of the performance of functional surfaces of machine parts hardened with coatings and for calculating the characteristics of deformation and damage.

Acknowledgments

The research was supported by the Ministry of education and Science, Agreement No. 075-15-2019-1865 of 02.12.2019, RFMEFI60719X0300 project. I thank the researcher Kravchuk K. S. for obtaining experimental indentation diagrams and the image of the indents.

Conflict of interests

The author declares that there is no conflict of interest regarding the publication of this paper.

References

1. Voronin NA (2011) Actual problems of creating tribotechnical topocomposites. *Izvestiya of the Samara Scientific Center RAS* 13: 695–698 (in Russian).
2. Golovin YI (2008) Nanoindentation and mechanical properties of solids in submicrovolumes, thin near-surface layers, and films: A review. *Phys Solid State* 50: 2205–2236.
3. Bhushan B (2017) Depth-sensing nanoindentation measurement techniques and applications. *Microsyst Technol* 23: 1595–1649.
4. Lu M, Huang H (2015) Interfacial energy release rates of SiN/GaAs film/substrate systems determined using a cyclic loading dual-indentation method. *Thin Solid Films* 589: 822–830.
5. Yeo A, Kai Y, Che F, et al. (2017) Study on damage and fracture of thin-film stacked structures through indentation test with acoustic emission sensing. *Int J Mech Sci* 128–129: 159–167.
6. Voronin NA (2019) The effective and true adhesive strength of thin protective coatings. *J Mach Manu Reliab* 48: 320–327.
7. Chen J, Lin Z, Bull SJ, et al. (2009) Experimental and modelling techniques for assessing the adhesion of very thin coatings on glass. *J Phys D Appl Phys* 42: 214003.

8. Abdul-Baqi A, Van der Giessen E (2001) Delamination of a strong film from a ductile substrate during indentation unloading. *J Mater Res* 16: 1396–1407.
9. Lu M, Xie H, Huang H (2013) Characterization of interfacial adhesion of thin film/substrate systems using indentation-induced delamination: A focused review. *Key Eng Mater* 533: 201–222.
10. Ruiz G, Pandolfi A, Ortiz M (2001) Three-dimensional cohesive modeling of dynamic mixed-mode fracture. *Int J Numer Meth Eng* 52: 97–120.
11. Yan Y, Shang F (2009) Cohesive zone modeling of interfacial delamination in PZT thin films. *Int J Solids Struct* 46: 2739–2749.
12. Hu J, Chou YK, Thompson RG (2008) Cohesive zone effects on coating failure evaluations of diamond-coated tools. *Surf Coat Tech* 203: 730–735.
13. Voronin NA (2019) Calculation of the adhesive strength of hard coatings, taking into account the ductility of the substrate material. *Proceedings of 6th International Conference* "*Fundamental Research and Innovative Technologies in Mechanical Engineering*", Moscow: IMASH RAS 96–98 (in Russian).
14. Voronin NA (2012) Composite and real hardnesses of thin coatings. *Adv Mater Res* 560–561: 803–808.
15. Voronin NA (2018) Modeling of an indentation diagram for topocomposites. *J Mach Manu Reliab* 47: 434–441.
16. Ponomarev SD, Andreeva LE (1980) *Calculation of the Elastic Elements of Machines and Device*s, Moscow: Machinostroenie, 326 (in Russian).
17. Argatov II, Sabina FJ (2016) Small-scale indentation of an elastic coated half-space: The effect of compliant substrate. *Int J Eng Sci* 104: 87–96.
18. Lee D, Barbera JR, Thoulessa MD (2009) Indentation of an elastic half space with material properties varying with depth. *Int J Eng Sci* 47: 1274–1283.
19. Voronin NA (2008) Effect of coating thickness and base material on mechanical properties and bearing capacity of hardened surfaces, In: Moskvitin GV, *Methods of Surface Hardening of Machine Parts*, Moscow: KRASAND, 91–122 (in Russian).