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Methodology of Mechanical Testing for Experimental Detection of Microdestruction Viscosity in Local Regions of Thin Ribbons of Amorpho-Nanocrystalline Material

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Abstract

Development of new thin ribbons of amorphous and nanocrystalline materials and their practical application is accompanied by the development of new and existing methods of mechanical testing. This is due to the fact that conventional mechanical tests cannot always accurately determine the particular mechanical properties of new materials.

In the present work, the analysis of the existing methods of mechanical testing of thin ribbons of amorphous-nanocrystalline metal alloys based on the local loading samples with the Vickers and Berkovich pyramids, and the ball. The main factors influencing the process of testing and the accuracy and reliability of their results are determined.

It was found experimentally that the deviations in the symmetry of the location of the system of cracks, their deviation from "standard" patterns of microfractures are caused by the presence of pores in the region of impact, delamination of the sample from the substrate, the influence of the edge regions of the samples. The existence of an optimal range of loads was revealed. In the case of insufficient load, a symmetrical system of cracks necessary to determine the viscosity of microfractures is not formed. When excessive loads are used, the symmetry is also disturbed due to the excessive number of chaotically located cracks and the broken sections of the sample. The requirements to mechanical tests for the experimental detection of the microdestruction viscosity in local sections of thin ribbons of an amorphous-nanocrystalline metal alloy are formulated.

Keywords

Mechanical testing; brittle thin films; pictures of microdestruction; amorphous-nanocrystalline alloys; viscosity of microfracture; mechanical properties.

Introduction

The development and creation of modern technologies, the application and processing of new materials necessitate the development of methods of mechanical testing. The development of modern industry and new requirements for performance characteristics of materials and equipment necessitates the development, manufacture and practical application of different kinds of materials. Materials which can acquire qualitatively new features are of particular value [1–3].

From the perspective of using materials in a particular field, one of the most important groups of indicators is their mechanical properties. The major mechanical properties include hardness, elasticity, strength, fragility, toughness, ductility, etc. Currently, there are various methods for determining plastic properties of solid materials. The plastic properties of the samples, which differ in the shape, the material from which they are made (chemical composition of the material), temperature testing, etc. are examined.

The hardness of metals and alloys is examined using the methods of Brinell, Rockwell, Vickers, where, as a rule, you can calculate plastic characteristics of the sample. The methods of bending and stretching the sample, showing the plastic characteristics when stretched on special machines, are used.

To obtain accurate reliable information about these or other mechanical indicators of studied materials there are various methods for the mechanical testing. It is obvious that various impacts will give different information. Of particular importance is the search for the latest methods of mechanical testing, their development and improvement.

The existing research methods of characteristics of solid plastic materials is not fully applicable to nanomaterials in the form of thin films with a local milli- and micro-sized areas of heat treatment, in which the mechanical properties are significantly different from the rest of the sample [4, 5].

Accordingly, there is a fundamental challenge in the research of structure and properties of nanomaterials, with the aim of identifying micro mechanics of deformation and destruction under local loading of the samples of amorphous thin-nanocrystalline metal alloys.

Thus, the purpose of this research is to test experimentally methods used to determine the viscosity of microfractures samples of thin amorphous-nanocrystalline metallic materials, analyze typical distortions of micro-patterns of deformation and fracture, and determine experimentally the criteria for valid studies using new methods of mechanical testing.

Experimental procedure

The ribbon with a thickness of 30 microns from amorphous metal alloy brand 82K3XCP (83.7 % Co + + 3,7 % Fe + 3,2 % Cr + 9,4 % Si) was studied. Tests were carried out on samples with dimensions 10×20 mm, which was preliminarily subjected to annealing in a furnace at T_{healing} = 503–1008 K.

Stack of samples of 20 pieces were superimposed on each other and placed between two stainless steel plates to avoid the possibility of warping of the samples during high-temperature annealing. The samples were not exposed to the stainless steel plates. Oven samples were heated and cooled at a speed of ≈ 10 K/min and kept at a predetermined temperature T_{healing} within 10 min.

After annealing of the samples, the X-ray diffraction analysis was conducted to clarify the structural state of the material and compare the results obtained with literature data by the method described in [6].

The study of the structural state of the samples was performed on the DRON-2 diffractometer, equipped with the coordinate x-ray detector and using a goniometer console GP-2. Monochromatization radiation Cr Ka ($\lambda = 2.29 \cdot 10^{-10}$ m) was applied. Data registration was carried out using $\theta - 2\theta$. The X-ray diffraction studies were also conducted on the DRON-3 diffractometer. The X-ray tube BSWU-27 was used in the device.

In some studies, the formation of amorphous nanocrystalline structure is described as a result of the collapse of the original amorphous phase of amorphous metal alloy of grade 82K3XCP [1, 6]. In [6] a study of the formation of amorphous-nanocrystalline structure by heat treatment of alloy $Co_{71.66}B_{4.73}Fe_{3.38}Cr_{3.14}Si_{17.09}$ was conducted and described. In this work, investigations were carried out according to the methods described in [1, 6]. The resulting radiographs were identical (within measurement accuracy) with radiographs obtained previously in [6]. This suggests that heat treatment of the samples made it was possible to obtain an amorphous nanocrystalline structure similar to that described in [6].

The samples subjected to heat treatment were applied to a polymer substrate with a metal base. A polymer solution with a hardener was applied to a metal base placed in a $35 \times 25 \times 5$ mm mold. The metal plate was used as the base had dimensions of $35 \times 25 \times 3$ mm. A sample was placed on the polymer composite, while the distance to the mold walls was equal on all sides. On the top, the composite was covered with glass with a defatted surface and held until the polymer solidified completely.

The polymer used for the preparation of the substrate was selected on the basis of the parameters of adhesion and viscosity consistency to liquid state. After hardening, the polyester composite had a hardness of $\approx 3.6 \times 10^{-6}$ PA, which was significantly lower than the microhardness of the investigated material. The classification of the used composite (BodiFiber) was 67/548/EEC. The feneral composition was as follows: CAS 100-42-5, EINECS no 202-851-5 (Index number: 601-026-00-0, styrene Xn R20; Xi R36/38, R10 Flam. Liq. 3, H226; Acute Tox. 4, H332; Skin Irrit. 2, H315; EyeIrrit. 2, H319 20–25 %). This composite was chosen according to the results of experimental testing out of 20 polymer composites.

The used polymer composite had good adhesion to the samples and did not delaminate during indentation in the central regions of the sample.

Local loading was carried out on PMT-3 microhardness meter. Vickers and Berkovich pyramids, as well as steel balls of various sizes were used as indenters.

Experimental results and discussion

Among all the methods of mechanical testing, one of the most common is microindentation using the Vickers pyramid. It was noted in [4, 7, 8] that microindentation of sufficiently thin samples (micrometers – dozens of micrometers) often requires the use of a substrate, and it is necessary to take into account its contribution to the results obtained [4, 9, 10]. When using mechanical testing by microindentation, it is necessary to take into account the correlation between the thickness of the sample and the diameter of the mold. Thus, for microindentation with the Vickers pyramid, a number of requirements must be met. In particular, it is necessary to ensure that the maximum allowable penetration depth of the indenter does not exceed 10% of the thickness of the sample. This, in turn, limits the possibilities of using this method for testing thin samples [9, 10]. In [4 – 6] (and references therein), the dependences of the influence of various substrates on the results obtained were established and the choice of substrates with certain mechanical properties was recommended.

The methods of mechanical testing based on the local loading of a sample placed on the substrate with the Vickers or Berkovich pyramids, or a steel ball, are sufficiently informative. It was found that depending on the type of indenter used, the magnitude of the indenter load, the magnitude of the elastic deflections of the substrate and the mechanical properties of the material, as well as a number of other factors, it is possible to form a complex symmetric system of cracks.

In this case, it is correct to speak not about indentation, but about local loading. This is due to the fact that in this case the requirement noted above is not met, due to the fact that the indenter does not exceed the maximum permissible depth of indentation. In [4, 7, 8], local loading of thin, thermally brittle samples of 82K3XSR alloy with loads up to 3.92 N leads to a deep penetration of the indenter into the sample and provokes the formation of the system of cracks, the analysis of their location (including the analysis of the average distances between groups parallel cracks) and allows drawing a conclusion about some mechanical properties of the material.

Depending on the shape of the elements acting on the sample (indenters), as well as the experimental conditions, the nature of the microdefects of the sample can vary considerably. Thus, micro-patterns consisting of triangles embedded in each other (loading with the Berkovich pyramid), spiral cracks (loading with a ball), squares (loading with the Vickers pyramid) can be formed. At the same time, these geometric figures can have different degrees of manifestation / clarity: from clear and easily analyzed, to partially visible and not amenable to analysis. It should be noted that these micro-pqatterns of destruction can undergo changes (including those associated with the peculiarities of crack growth) in the process of mechanical action. At a certain stage, splits and radial cracks may occur, which can make it difficult to form a symmetrical system of cracks. Determination of the mechanical properties of cracks is carried out through the analysis of precisely symmetrical cracks. Thus, the formation of radial cracks under local loading is an undesirable factor. Accordingly, one of the problems of choosing the geometry of the indenter and the mechanical properties of the substrate is related to the task of reducing the number of radial cracks.

It is noteworthy that the deflection of the substrate increases with the increasing load on the indenter, and it can reach 50 μm. In other words, the value of the elastic deflection of the substrate can exceed the thickness of the test sample. However, the residual deformation after the removal of the load, with a symmetrical pattern of failure, does not exceed a few micrometers. In conditions of such a significant deflection, the properties of both the substrate and the quality of adhesion between the sample and the substrate are important. The deterioration of the quality of the substrate leads to a decrease in the symmetry of the micro-patterns of destruction.

A method for determining the mechanical properties of thin ribbons of an amorphous-nanocrystalline alloy based on an analysis of micro-patterns of fracture formed when the Vickers pyramid is pressed was proposed earlier. This method was practically tested and showed its effectiveness. The theoretical substantiation of the method of mechanical tests was considered in a number of works [4, 5, 7, 8] (and references therein). In addition, the methods of mechanical testing were protected by two patents of the Russian Federation earlier.

The results of the experimental application of mechanical testing methods for thin nanocrystalline samples, with local regions subjected to laser treatment, were described in [4, 7, 8]. The process of local loading was considered in detail. The specificity of the bending of a sample of an amorphous-crystalline metal alloy tape was considered. For the first time, the term “viscosity of microfracture” was introduced, which has a different physical meaning than the term “plasticity”. In the same works, the authors completed the development of some of the listed methods, which made it possible to partially eliminate their shortcomings. In particular, a new formula for calculating the plastic characteristic of a material under local loading on substrates was proposed, a method of mechanical testing of brittle bands of a nanocrystalline material loaded with a steel ball was proposed. It was shown that it is expedient to carry out the local loading of the most fragile samples with a ball. The use of this method contributes to the increase of accuracy and allows studying more fragile materials [8].

It should be noted that the proposed methods of mechanical testing have certain shortcomings. For mechanical loads, the loading process and the nature of failure should be monitored to avoid obtaining erroneous data. This is due to the fact that indentation during the initiation of fracture is accompanied by a substantial elastic deflection of the composite – "sample-substrate", which can provoke the detachment of the sample from the substrate. In the case of detachment of the sample from the substrate, the analysis of the microstructure of the destruction will result in the incorrect results. The detachment of the sample from the substrate can also be observed when local loading occurs near the sample boundary or near a group of macrocracks initiated by the previous test.

Thus, when finding the viscosity of microfracture and microhardness, it is necessary to control the quality of the substrate and the absence of pores and delaminations under the local loading place. If under the area of local loading there is a pore of a size of 25 – 50 μm or more, then instead of the system of cracks oriented parallel to the faces of the indenter, a characteristic dip is formed. In case the local loading is carried out near the sample boundary (the distance to the sample boundary is less than 1 mm), it is possible that the part of the sample is peeled off or inelastically pressed into the substrate (Fig. 1). All this distorts micro-patterns of destruction. Instead of the system of embedded squares, radial cracks and significant chipped sections of the material are formed. Fig. 2 shows an example of the characteristic distortion of microstructure destruction under local loading of a fragile sample with micropores located above a cluster. In these cases, the distortion of the symmetry of the microstructure of destruction is so great that the analysis of microcracks becomes impossible.

In some cases, the use of the Berkovich pyramid is preferable before using the Vickers pyramid. As a result of local loading with the Berkovich pyramid of the embrittled sample, a characteristic microstructure of destruction is formed, resembling a system of embedded triangles. However, the presence of a pore beneath the region also leads to distortion of the symmetry of the system of cracks.

In order to exclude such distortions, it is necessary to monitor the quality of the surface layer of the substrate, especially the presence of pores, in the microindentation of thin samples on a substrate. This is due to the fact that the pores in the surface layer of the substrate, as well as the peeling of the test sample from the substrate during laser treatment of the local region lead to errors in finding both the microhardness of the sample and the viscosity of the microfracture.

In case the sample is more plastic, the local loading leads not only to the formation of a symmetric microstructure of destruction, but also to the characteristic deformation bands. If the pore is located under the region of local loading, the formation of an annular crack along the boundaries of the pore leads to local elastic (or inelastic) subsidence of the sample.

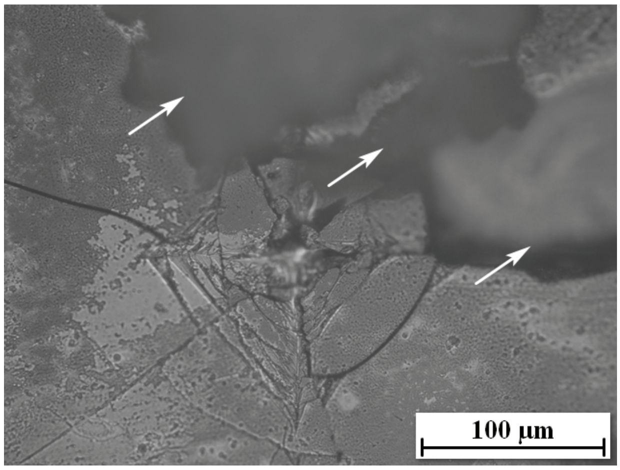


Fig. 1. The micro-pattern of fracture formed when the Vickers pyramid was pressed in at a distance of 200 μm from the edge of the sample
(The annealing temperature of the sample is 730 K. The load on the indenter is 3.92 N. The arrows indicate the sections of material that have been cut off and pressed into the substrate)

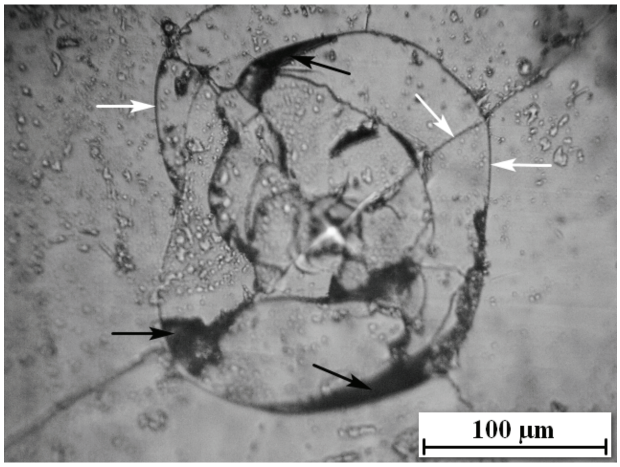


Fig. 2. Micro-pattern of destruction occurred when the sample was loaded with the Vickers pyramid (load 3.92 N)
(The loading region was located above the accumulation of micropores. A symmetric microprint was not formed due to the effects of micropores of provoking dips (shown by black arrows), detachments and fissures of sample material, as well as the formation of radial and arc macrocracks (shown by white arrows))

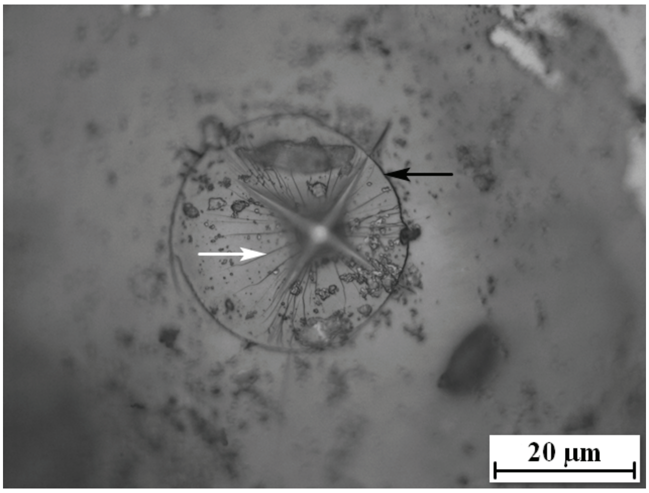


Fig. 3. Micro-pattern of fracture and deformation after pressing the Vickers pyramid into samples subjected to annealing at 703 K, load on indenter 3.92 N
(Samples were deposited on a substrate with artificially created voids and subjected to local loading. Local loading was carried out at times with a diameter of ≈ 20 μm. The black arrow shows an annular crack, a white arrow shows one of the deformation cracks. The deformation cracks appeared after the formation of an annular crack. As a result, the annular crack limited their spreading)

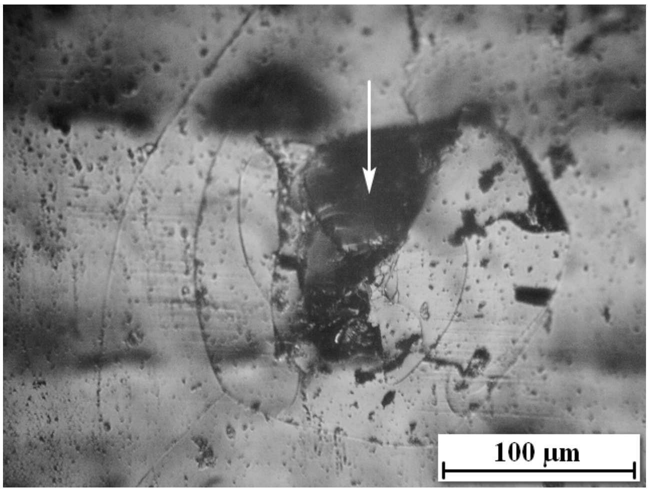


Fig. 4. Loading of the sample area located above the pore
(The annealing temperature of the sample was 850 K, the loading on the indenter was 3.43 N. The arrow shows the sample region located above the pore and pressed onto the substrate)

As a result, the possibility of forming a symmetrical system of cracks is blocked. Note that the formation of the splitting (annular crack) precedes the development of deformation bands. The deformation bands develop after the fractured part of the sample has subsided at the time and does not overcome the crack (Fig. 3).

Located under the place of local loading, a large single pore adjacent to the loading region strongly distorts the microstructure of destruction (Fig. 4). The arrows indicate the split and depressed region of the sample. The resulting distortions of the system of mechanical stresses did not allow forming a symmetrical system of cracks.

The presence of several pores in the vicinity of the loading can also greatly change the microstructure of the destruction. Radial cracks from the center of the loading region are formed around which one or several arc cracks appear (Fig. 5). In addition, because of the presence of a multitude of pores, a breakaway of the material was formed, limited by arc and radial cracks.

It was experimentally found that, in a number of cases, the formation of a dense network of cracks, splits and material sagging results in "mishmash" of broken pieces of the sample is formed. In this case, it is difficult or impossible to find the viscosity of the microfracture. In addition, cracks can form in the area of contact of the edges of the Vickers pyramid with the sample. They, in turn, block the formation of cracks oriented parallel to the faces of the indenter; the analysis of their location allows finding the viscosity of microfracture. The occurrence of cracks in the contact area of the edges of the Vickers Pyramid with the material alters the distribution of mechanical stresses and blocks the formation of complete and usable microcrack fracture toughness. Such fractures are characteristic for samples with high microhardness.

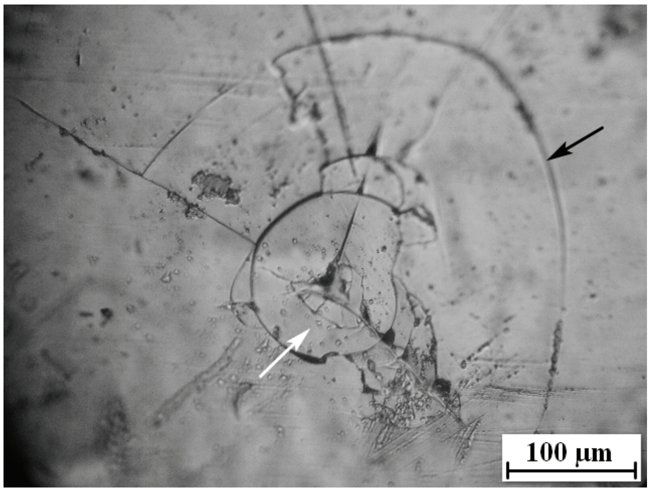


Fig. 5. Typical distortion of the microstructure of destruction, caused by the influence of several micropores located in the substrate (in the loading region and above)
(The annealing temperature of the sample was 700 K, the load on the indenter was 4.41 N. The white arrow shows the central loading region, separated from the rest of the material by an annular crack. The black arrow shows the splitting of the material, caused by the sample slipping into the micropore system. The splinter part is separated from the rest of the sample by a system of arc microcracks, annular microcracks, and also by radial cracks)

It was experimentally found that in a number of cases a system of embedded squares is formed in the center of the loading region, and only then, in the final stages, shocks and dips are formed due to excessive load on the indenter or the influence of micropores (Fig. 6). However, the distortions introduced by the junctions and gaps into the symmetrical picture of failure do not allow determining accurately the viscosity of the microfracture.

In a number of cases, radial cracks are formed before the appearance of an annular crack. At the same time, part of the radial cracks can reach a length of several millimeters. Radial cracks can be joined together and come out to the boundary of the sample.

Experimental studies were carried out to identify the optimal and permissible modes of mechanical testing. Based on the results obtained, an algorithm for performing mechanical tests was proposed. It is aimed at determining the conditions under which it is permissible to use the method of mechanical testing discussed in [7, 8].

It was experimentally established that the sample size should be at least 20 × 20 mm. The sample should be applied to a substrate with a thickness of at least 5 mm. Its recommended size is 30 × 40 × 5 mm. The sample is applied to the center of the substrate, and it is placed on a metal substrate 3 – 5 mm thick.

At the first stage of the experimental determination of the optimal conditions for detecting the viscosity of microdeficiency, the optimum load range for the indenter should be determined.

The variety of substrates, samples and indenters used poses the problem of correctly interpreting the requirements for the mechanical properties of the substrates. It is also necessary to establish the requirements for the permissible load on the indenter and the optimal distances between the loading regions. In this sense, preliminary mechanical tests and detection of optimal distances from the sample boundary and adjacent loading points are expedient.

In the central regions of the sample, at least 5 mm from the edge, the indenter is loaded using a microhardness meter (for example, PMT-3). The recommended initial loads are 0.2 N. In the event that their application leads to severe cracking, the loads should be reduced. However, as a rule, cracks are not formed under these loads. In this case, it is necessary to increase the load in steps to 0.1 N, until symmetrical micro-patterns of destruction are formed. If microcracks do not occur at loads of 3 – 5 N, it is recommended to use the Vickers or Berkovich pyramids instead of the spherical indenter.

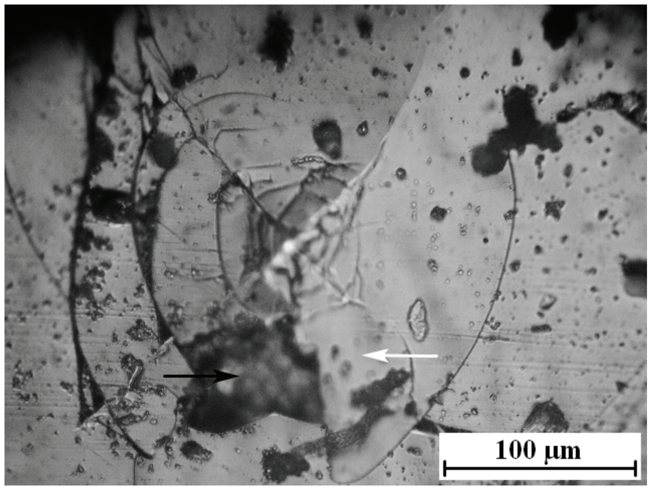
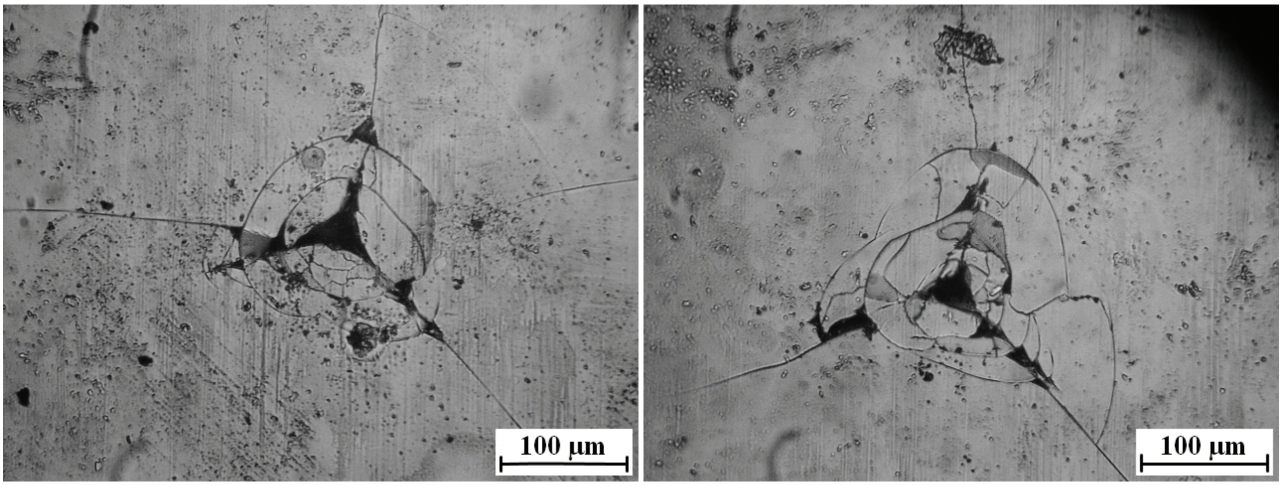


Fig. 6. Formation of a symmetric microstructure of destruction was interrupted by the formation of the gap of the sample material into the pore and inelastic subsidence of the material
(The black arrow shows the "gap" of the material. The white arrow shows the depressed region of the material bordering the gap. As a result of these processes, the formation of the system of cracks oriented parallel to the faces of the indenter was not completed. The annealing temperature of the sample was 850 K, the loading on the indenter was 3.92 N)

Thus, the choice of the optimum load on the indenter is limited by two factors. It is necessary that the probability of the formation of a standard microstructure of destruction tends to unity, which is achieved by increasing the load on the indenter. At the same time, if the load on the indenter increases, the probability of distortion of the standard picture increases. The distortion of the standard pattern almost always leads to the blocking of the development of a system of nested cracks or the formation of a crumb from the cleaved sections of the sample. Therefore, excessive load increase should be avoided. As a limit load, one can take such a load, at which the probability of distortion of the standard microstructure of destruction will not exceed 0.5. This is due to the fact that insufficient load does not allow to form a sufficient number of cracks and to obtain the necessary symmetry of the microstructure destruction (Fig. 7 *a*). At the same time, excessive loading leads to the formation of an indestructible pattern of failure due to the formation of a number of random splits of the sample (Fig. 7 *b*) and / or the growth of the radial crack system, blocking the formation of spiral and annular cracks. At the same time, the formation of main cracks is mainly characteristic when the Vickers or Berkovich pyramids are used.

It is important to ensure the maximum probability of formation of standard microstructures of destruction with the least possible load on the indenter. If this is not possible, it is expedient to replace the spherical



a) *b*)

Fig. 7. Microfracture when indenting an annealed sample at a temperature of 800 K:
a – Typical distortions of the “typical microfracture pattern” under local loading with the Berkovich pyramid of insufficient load (1.96 N);
b – Distortion of the microstructure of destruction under local loading by the Berkovich pyramid with the formation of a chaotic system of cracks and tiny bits from the broken sections of the sample under excessive load of 4.9 N

indenter with an indenter with a smaller radius, or change the substrate used. If it is not possible to achieve stable micro-patterns of destruction, indenters of another geometric form (the Vickers or Berkovich pyramids) should be used.

In the second stage of determining the optimum experimental conditions for detecting the viscosity of microfracture, it is necessary to determine the minimum distances to the edge of the sample and to cracks initiated by local loading. Local loading indenter is accompanied by a significant elastic deflection of the composite – “sample – substrate”. The elastic deflection of the substrate can reach several tens of micrometers. Under these conditions, when loading near the sample boundary, the sample is peeled off from the substrate or the sample is inelastically pressed into the substrate material. At the same time, there are distortions in the shape of the spiral-shaped crack, the distances between its turns, and the number and shape of the embedded cracks. The minimum distance to the boundary of the sample, which ensures the correct procedure for the mechanical tests, depends strongly on the properties of the substrate material. If the distance to the edge of the sample (or destroyed adjacent regions) is less than 500 μm, loading with a load of 3.92 N causes significant distortion of the micro-patterns of destruction. It has been experimentally established that the distance from which it is possible to begin determining the permissible remoteness (*S*) of the local loading point from the edge of the sample should be taken in one millimeter.

At a given distance S from the edge of the sample, a series of local loads is carried out (at least 10). The distance between neighboring loading points is also assumed in *S*.

Minor exfoliation of the sample from the substrate during testing is permissible and practically unavoidable for some substrate materials. However, the presence of such peeling at significant (more than 300 μm) distances from the loading region is unacceptable, especially if exfoliation is observed at neighboring fracture regions, fissures and sample boundaries. If, during local loads, the sample is peeled off from the substrate at the sample boundary, cracks appear on the sample boundary or the shape of the spiral (embedded) cracks is distorted, then the distance S from the sample boundary should be increased. In this case, respectively, we increase the distance *S* between neighboring regions of local loading.

By the above scheme, the optimal distances to the sample boundaries and between the loading regions are selected. Note that for qualitative analysis, it is necessary that standard micro-patterns of fracture are formed upon a single loading with a probability of at least 0.5.

Cases causing excessive damage should be excluded from the analysis. It is necessary to strive to minimize the number of "incorrect" micro-maps of destruction. Thus, before carrying out tests to determine the viscosity of microfracture, it is necessary to determine experimentally the optimum substrate. After selecting the substrate, the optimum geometric shape of the indenter should be determined experimentally: a spherical indenter, the Vickers or

Berkovich pyramid. The next step is to determine the permissible distances between the areas on which the loads are carried out, the permissible distances to the sample boundary and cracks. At the final stage of the preparatory tests, the optimum load interval for the indenter should be determined.

In the case when the determined value of the viscosity micro-disintegration differs significantly from the mean value, control loads (at a distance of 100–300 μm from the point of initial loading) should be made with a load of up to 4.9 N. If in the local loading region a material breakage occurs and a microprint is formed destruction with a print from the indenter surrounded by an annular crack, the measurement results should be deleted, since the high value of microhardness (as indicated by the insignificant size of the print from the pyramids Vickers) associated with the deflection section of the sample located above the pore.

Conclusion

1. Deviations in the symmetry of the arrangement of the system of cracks, their deviation from "standard" microfracture patterns are due to the presence of pores in the site of impact, poor adhesion between the sample and the substrate, and the influence of asymmetric stress fields when loaded near the sample boundary.

2. Increasing the accuracy of detecting the viscosity of microfracture can be achieved by reducing the porosity of the substrate and improving the quality of adhesion between the sample and the substrate.

3. The method of preparatory tests of the sample-substrate composite is proposed, which allows refining the parameters of mechanical tests when detecting the viscosity of microfracture. The proposed method includes the following steps: selection of the optimal material for substrate preparation; selection of the optimal form of the indenter, determination of permissible distances to the sample boundary and the neighboring fractures, and determination of the permissible range of loads on the indenter.

4. Incorrect execution of the experimental procedure leads to the impossibility of obtaining the required microstructure of destruction or leads to a violation of its symmetry. Asymmetric microbreaks of fracture cannot be used to detect the viscosity of microfracture.

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