

Evaluation of Embrittlement of Construction Steels by Microindentation

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Received February 21, 2023; revised March 19, 2023; accepted April 28, 2023

Abstract—The mechanical characteristics of a metal are determined by a combination of three groups of factors: the chemical composition, the structural features, and the deformation ability of the structure, i.e., the ability of elements to relax internal stresses during deformation through dislocation sliding which does not lead to the crack formation and destruction. The possibility of using microindentation to assess the deformation ability of the structure of structural steels with a relatively high ductility is the goal of the study. The theoretical analysis revealed that an increase in the stiffness and a decrease in the plasticity of a metal leads to a change in the deformation model during indentation and, in particular, to the occurrence of deformation effects of various morphologies on the surface near the imprint, which can be indicative of the metal plasticity. Experimental studies performed on pipe steels of various strength and types of structure confirmed that, as the deformation ability of the metal decreases (primarily as a result of deformation hardening), a system of localized shears is formed near the imprint along the lines of action of maximum tangential stresses. A scale for ranking data of localized shears is proposed, and the optimal load value and shape of the indenter which provide gaining maximum information by microindentation are determined. A methodology for assessing the embrittlement of plastic construction steels on the basis of the results of microindentation has been developed, which can form a basis for creating an effective technology of nondestructive evaluation of the metal state.

Keywords: microindentation, microhardness, brittleness, embrittlement, construction steels, localized shear, deformation ability

DOI: 10.1134/S0020168524700262

INTRODUCTION

One of the key tasks that organizations operating hazardous production facilities face is to ensure their reliability and safety. During their service, structures are exposed to a wide range of factors and loads, which can lead, in particular, to a change in the performance properties of steel, the main structural material. As a result, the accuracy and reliability of predicting the performance of such a structure is sharply reduced, and the risk of its unexpected destruction increases [1]. To effectively solve this problem, nondestructive methods of assessing the state of the metal of structures at the stage of their operation are used.

One of these methods, the development of which can lead to the creation of an effective technology for nondestructive assessment of the state of metal structures, is microindentation.

This method, known since the 1950s, at one time was widely developed for the physicochemical analysis of metals [2]. However, interest in it has recently decreased greatly, since it required cutting samples

from metal and using stationary equipment. At the same time, modern destructive methods for studying metal at the micro- and mesostructural level, such as electron microscopy and X-ray diffraction analysis, allow for more accurate analysis than the microindentation method.

At the same time, modern equipment is capable of implementing the microindentation method directly on the structure under study [3]. Therefore, the use of the microindentation method as a nondestructive method for assessing the performance of structural steels is of great practical importance.

The mechanical characteristics of a metal are determined by the interaction of factors such as

- (i) chemical composition (content of alloying elements, harmful impurities, etc.);
- (ii) features of the structure (type, shape, size of structural elements, parameters of nonmetallic inclusions, etc.);
- (iii) deformability of a structure.

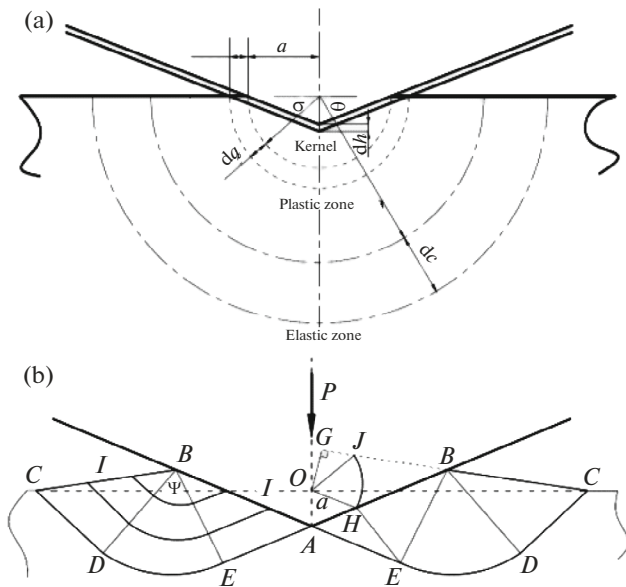


Fig. 1. Model of indentation of a blunt indenter into [6] (a) elastoplastic medium and (b) rigid-plastic volume.

The deformability of a structure is understood as the ability of its elements to relax internal stresses during deformation through dislocation sliding, which does not lead to the formation of cracks and destruction. In this case, various strengthening mechanisms are implemented that prevent the movement of dislocations.

The deformability of the structure does not completely determine the plasticity of the metal, but is its basis, which can only be reduced by other factors.

Obviously, microindentation does not allow one to evaluate most of the factors that determine mechanical properties of a metal, but this can be done by additional, including nondestructive, methods [4, 5]. This paper examines the possibility of using the microindentation method to assess the deformability of a structure, in particular, reducing this ability—embrittlement.

THEORETICAL RATIONALE FOR USING THE METHOD

The theory of mechanics of contact interaction is described in detail in [6]. In relation to solving the posed problem, two boundary cases described in it—elastoplastic and rigid-plastic media of indenter penetration—were considered.

In an elastoplastic medium, the displacement of material by an indenter at the initial stage is completely compensated by elastic displacements of the surrounding medium, the contours of the plastic zone are almost entirely located under the contact area, and the surfaces of equal deformations have an approximately hemispherical shape (Fig. 1a).

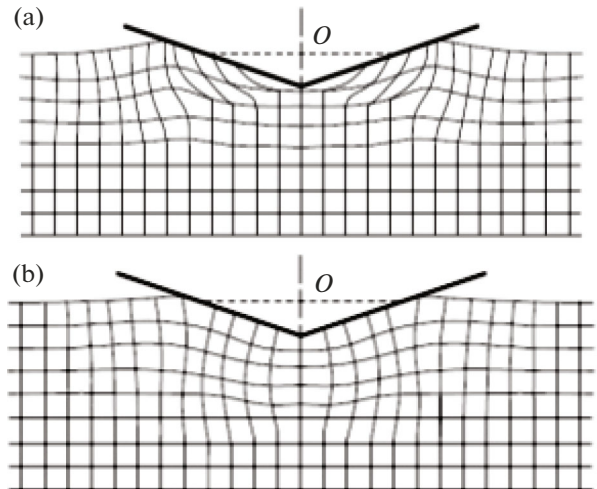


Fig. 2. Deformation upon the indentation of a blunt indenter [6] in the absence of (a) friction and (b) slippage between the material and the indenter.

A rigid-plastic medium under loading includes regions of plastic flow and regions of a rigid state where deformation is absent. The stressed state in flow regions is described using a field of slip lines passing parallel to the direction of the main tangential stresses at each point of the field, i.e., at an angle of 45° to the directions of the main normal stresses. In this case, plastic flow is localized in two symmetrically located regions, indicated in Fig. 1b with letters $ABCDE$. The material surrounding these areas is considered rigid and does not deform.

The indentation process is significantly influenced by the occurrence of friction between the indenter face and the material. In this case, the deformation develops below the top of the wedge, and displacements of the corner points of the mesh from their initial positions occur approximately in the radial direction from point O . Deformations of the body during the introduction of a blunt indenter for the cases of absence of friction and slippage between the materials and indenter calculated using a hodograph are presented in Fig. 2 [6].

On the basis of the given data, it is possible to conclude the following:

When a material has high elastoplastic characteristics, the main part of the volume displaced during indentation of the material is displaced in the radial direction owing to elastic and then plastic expansion of the environment.

If the material has low elasticity and high rigidity, the material displaced by the indenter moves to the side zone, rising along the edges of the hole. Moreover, in the case of low plasticity, its localization is observed, expressed in the appearance of deformation

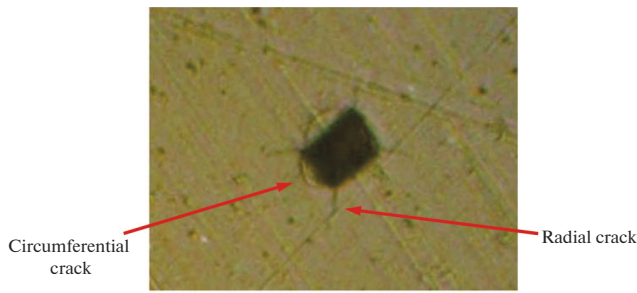


Fig. 3. Indenter imprint on the glass surface at a load of 50 gf.

effects of various morphologies on the surface, including destruction.

It is known [2] that fracture during indentation occurs only when the indenter is pressed into very brittle bodies (glasses, refractory compounds, minerals, etc.). As an example, Fig. 3 shows the imprint when glass is indented. It can be seen that the formation of the imprint is accompanied by the appearance of both radial cracks, formed by tearing as a result of the action of normal stresses, and cracks and chips around the circumference of the imprint, repeating the contours of the maximum tangential stresses along which the material was extruded.

In bodies with higher plasticity, fracture does not occur. This is due to the fact that, compared to other types of loading (tension, compression, bending, etc.), indentation is characterized by “soft” deformation conditions, since the plastically deformed volume is surrounded by a mass of undeformed material, “supporting” the zone of plastic deformation and forming conditions similar to all-round compression. As a result, resistance to plastic deformation during indentation is more than four times higher than the resistance to axial compression [2].

However, even in this case, surface effects may be observed near the imprint. For example, when indenting amorphous alloys (Fig. 4) [7], which have a specific plasticity (extremely low in tension and high in bending), localized shear bands are formed. It can be seen that the formed localized shear lines are similar in their morphology to circumferential cracks (Fig. 3)

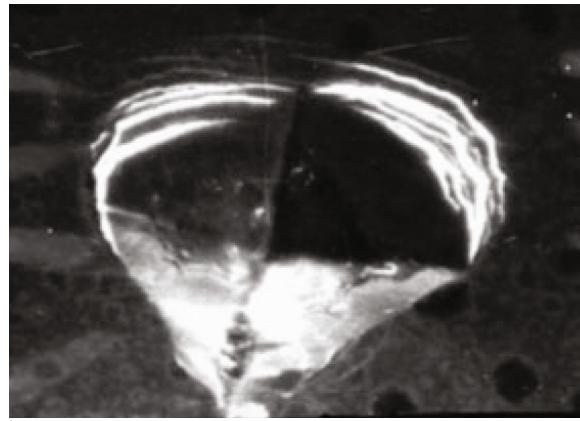


Fig. 4. Localized shifts near the imprint during microindentation of an amorphous Fe–Cr–Mo–Ni–C–Mg–Al alloy (load 150 gf) [7].

and also correspond to the contours of maximum tangential stresses.

The presented analysis shows that, as the plasticity decreases, the deformation scheme of the metal during indentation passes from elastoplastic (Fig. 1a) to rigid-plastic (Fig. 1b). The deformation that displaces the bulk of metal in the area of the imprint becomes more constrained and is realized in the form of individual localized shear bands near the imprint, which serve as sources of formation of cracks and chips when the plasticity is completely exhausted. Moreover, given the visual similarity, it is not so easy to clearly distinguish a localized shear from a circumferential crack. In this case, when studying structural steels with relatively high plasticity, the morphology of localized shear lines near the imprint can serve as an informative criterion characterizing the deformability of the structure. In order to confirm this assumption, experimental studies were carried out.

EXPERIMENTAL

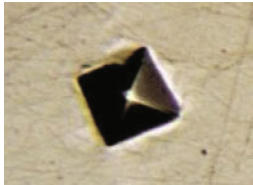
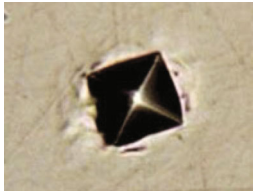
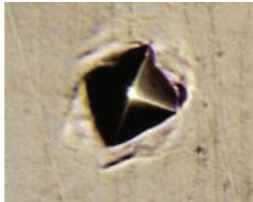
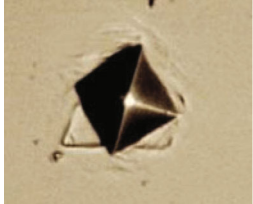
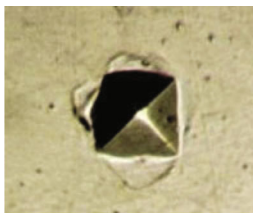
To carry out the tests, metal samples were made, which are listed in Table 1.

Metallographic sections were made in the cross section of the samples in accordance with the require-

Table 1. Characteristics of metal samples under study

Sample number	Name of the structure from which the sample was cut	Grade of steel	State	Structure of metal
1	Tee Du 89/Du 89 mm, wall thickness of 12 mm	09G2S	New	Ferrite-pearlite
2	Pipe Du 1220 mm, wall thickness of 12 mm	K56	New	Ferrite-pearlite
3	Pipe Du 1220 mm, wall thickness of 12 mm	K56	After thermal treatment: $T_{\max} = 1100^{\circ}\text{C}$, $V_{\text{cool}} = 90^{\circ}\text{C/s}$	Bainitic
4	Pipe Du 1020 mm, wall thickness of 15.7 mm	Kh80	New	Bainitic

Table 2. Scale of the brittleness of structural steels according to the results of microindentation

Score	Description	Photo
1	Imprint without visible lines of localized shift and traces of nonuniform plastic deformation*	
2	Traces of inhomogeneous plastic deformation, short,** clear lines of localized shear at 1–3 edges of the imprint	
3	Clear lines of localized shift of average*** length on 1–2 imprint edges or short lines on 4 imprint edges	
4	Clear lines of localized shift of average*** length at 3–4 edges of the imprint or the formation of extended**** lines at 1–2 edges of the imprint	
5	Clear extended**** lines of localized shift on 3–4 edges of the imprint or on 2 edges with lines of medium*** length on 1–2 more edges	

* The features were analyzed at maximum sharpness of the imprint image.

** Short line of the localized shift: the line length is no more than a third of the length of the face of the imprint.

*** Line of localized shift of medium length: the line length is between one-third and two-thirds of the length of the imprint edge.

**** Extended line of localized shift: the line length is more than two-thirds of the length of the face of the imprint.

ments of GOST 5640-2020. Next, microindentation of the surface of the polished sections was carried out, followed by an analysis of the morphology of the deformation effects near the imprint and determination of microhardness values.

The imprints were made with an indenter to determine the Vickers microhardness (diamond pyramid with an apex angle of 136°) at a load of 50 gf on a PMT-3M microhardness tester in accordance with GOST 9450-76.

Sample no. 1 was etched in accordance with GOST 5640-2020, and the zones for indentation were selected so that the imprints were located in the center

of ferrite grains, the area of which in diameter significantly exceeded the indentation size.

The remaining samples were not etched because they had a finely dispersed structure, which made it difficult to analyze the deformation effects near the imprint.

The obtained results showed that the morphology of localized shifts that occur near the imprint is quite diverse. To rank it, a point scale has been introduced, shown in Table 2, by analogy with the well-known brittleness scale of nitrided steels [8].

At the first stage, sample no. 1 was examined. It was found that microindentation of ferrite grains in the internal volume does not lead to the occurrence of any

deformation effects near the indentation (1 point from Table 2), and the average microhardness value is 131 kgf/mm². At the same time, localized shifts and, at the same time, an increase in the microhardness value were recorded in the near-surface layer with a depth of about 0.5 mm. Moreover, both phenomena intensified as they approached the outer surface. In particular, the average microhardness of surface ferrite grains reached 214 kgf/mm², and the pattern of localized shifts in this zone corresponded to 4 and 5 points from Table 2.

The results indicate that the near-surface layer of the metal of the tee joint was subjected to strong strain hardening during production, which resulted in a significant increase in the microhardness of the metal and the occurrence of developed localized shifts near the imprints in this zone. This conclusion is confirmed by the nature of the structure of the near-surface layer of sample no. 1, shown in Fig. 5, which represents highly deformed elongated grains of ferrite and pearlite.

At the second stage, comparative studies of sample no. 2 cut from a normalized pipe in the original state and sample no. 3 cut from the same pipe and then subjected to quenching were carried out. It was established that the metal of sample no. 2 has microhardness values in the range of 162–180 kgf/mm² in the entire volume and 1 point of brittleness in accordance with Table 2, including in the near-surface layer. For sample no. 3, the microhardness values are 267–326 kgf/mm² in the entire volume, and the morphology of localized shifts near the imprint varies greatly—from 2 to 5 points (Table 2).

It is clear from the obtained results that the microhardness of sample no. 3 is higher than that of sample no. 2, which is due to its chemical composition and dispersity of the structure, as well as the high deformability of the structure achieved by normalizing heat treatment. As a result of quenching, a nonequilibrium structure with a high density of dislocations and low deformability was formed in sample no. 3. This led to both a sharp increase in the microhardness values relative to steel in the initial state and to the occurrence of localized shifts of various morphology. Moreover, the inhomogeneous pattern of localized shifts throughout the volume of the sample also indicates its inhomogeneous plasticity.

At the third stage, sample no. 4 was examined. Throughout the entire volume of the sample, the microhardness values were 235–286 kgf/mm², and the morphology of localized shifts was estimated from 4–5 points in the near-surface layer of metal about 0.5 mm deep to 3–4 points in the central part.

It can be seen that, in terms of average microhardness values, sample no. 4 is close to quenched sample no. 3, and in terms of the development of the morphology of localized shifts near the imprint, it even surpasses it. The explanation for this is the production technology of Kh80 steel sheets—controlled rolling

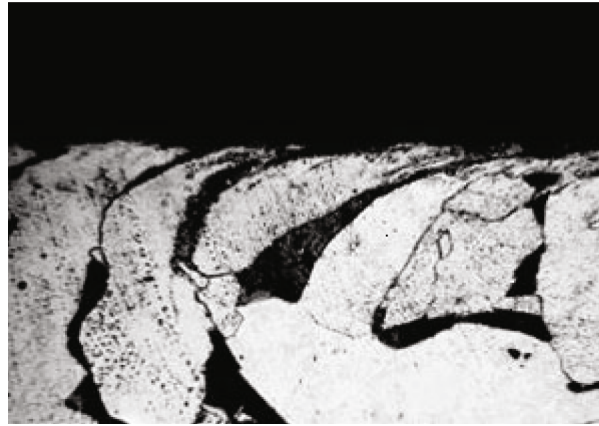


Fig. 5. Structure of the subsurface layer of sample no. 1.

with accelerated cooling. The implementation of this technology leads to the appearance of a bainitic structure in the metal, which itself is not completely equilibrium and is characterized by an increased density of dislocations. In this case, this structure is additionally subjected to high plastic deformations during mechanical processing, which as a result leads to very high dislocation density in the metal and almost complete exhaustion of the possibility of its strain hardening.

Analyzing the results, one can see that the morphology of localized shifts correlates with the deformability of the structure, in particular, with its ability to relax internal stresses through strain hardening.

As already noted, the morphology of localized shears does not directly determine the plasticity of the metal. Therefore, despite the low deformability of the structure, Kh80 steel has acceptable plasticity and impact strength owing to the optimal chemical composition, the low amount of harmful impurities, the shape and size of structural elements, and other factors. However, it should be expected that the emergence of the conditions leading to the appearance of a developed system of localized shifts in the metal during deformation will primarily have a negative impact on the crack resistance characteristics, since the latter are sources for the initiation and development of cracks. This assumption is confirmed by the studies [9], in which low characteristics of cold brittleness and crack resistance of Kh80 steel were established.

It is worth noting the relationship between the morphology of localized shifts near the imprint and microhardness values. On one hand, there is a general qualitative relationship between these characteristics—the development of localized shifts in all cases is accompanied by an increase in the average microhardness values. On the other hand, there is no quantitative relationship between these characteristics—at the same morphology of localized shifts, the microhardness values can differ significantly. Moreover, this dif-

ference is both systemic in nature (when comparing average fragility scores and average microhardness values for different samples) and particular (when comparing these characteristics for different imprints on one sample).

An explanation for these results can be found from the analysis of four well-known physical models of hardening of materials with a crystalline structure [10, 11]:

dislocation—an increase in the density of dislocations prevents their mutual movement and access to the surface;

grain boundary—restraining the movement of dislocations by grain boundaries;

solid solution—strengthening due to distortion of the crystal lattice of the material as a result of the introduction or replacement of its atoms with alloying elements;

dispersion—strengthening due to the release of particles of other phases that resist the movement of dislocations.

The implementation of each of these hardening models to an appropriate degree increases directly the resistance of the metal to plastic deformation, in particular, increasing the hardness. As for plasticity, i.e., the ability to change shape without destruction, such an inverse relationship does not exist. Only dislocation strengthening, directly proportional to the increase in hardness, reduces the plasticity of the structure; in other cases, this relationship is more complex and is determined by the peculiarities of the implementation of various types of strengthening [10, 11].

Thus, the values of microhardness reflect the level of hardening of the metal from the processes discussed above, and the morphology of localized shifts near the imprint is the contribution of these processes to the reduction of plasticity and fracture resistance owing to changes in the mechanism of plastic deformation.

It would be fair to assume that the fracture resistance is affected by a combination of the level of hardening and the deformability of the metal; therefore, in relation to solving the problem of creating an effective technology for nondestructive assessment of the state of the metal, both of these characteristics are informative and should be evaluated in their entirety.

To use the results for practical purposes, it is of interest to assess the influence of the type of indenter and the magnitude of the load on the formed morphology of localized shifts near the imprint.

For this, in addition to an indenter in the shape of a regular tetrahedral diamond pyramid with an apex angle of 136° , we tested the following:

a Birbaum indenter (regular tetrahedral diamond pyramid with an apex angle of 90°);

a specially manufactured indenter in the shape of a regular tetrahedral diamond pyramid with an apex angle of 60° .

The loads on the indenter were varied in the range of 20–500 gf.

The studies were carried out on sample no. 1 (Table 1), since, as established, it contains both a zone in which developed localized shifts are formed near the imprint (near-surface layer) and a zone with a complete absence of such deformation effects (main volume of sample).

Additionally, electron microscopic analysis of the imprints was performed using a scanning electron microscope SU8000 (Hitachi).

The results showed that, as the indenter tip increases, pronounced piles of metal begin to form next to the indentation; i.e., regardless of elastoplastic properties, an increase in the volume of displaced metal under the imprint leads to its extrusion onto the free surface (Fig. 6a). This, in turn, interferes with the identification of localized shifts during visual analysis of imprints (Fig. 6b).

As the load increases, the development of localized shifts decreases and practically disappears at loads above 200 gf, even in the surface layer of sample no. 1, where imprints with a fragility score of 5 were recorded in accordance with Table 2.

This unexpected result can be explained by a change in the mechanism of plastic deformation in the imprint formation zone as the load increases. In particular, as shown in Fig. 2b, the occurrence of friction during the introduction of an indenter leads to the localization of a zone of plastic deformation under the imprint, even with a rigid-plastic deformation model. As a result, the localized shifts near the imprint formed at the initial stage of penetration are subsequently absorbed by the expanding surface of the contact of the indenter with the metal, and new deformation effects no longer appear on the surface.

Thus, the most informative pattern of localized shifts near the imprint is observed when using a less sharp indenter and a lower load. Taking into account that, starting from a certain size, the analysis of imprints is difficult using optical microscopy, it is optimal to use an indenter in the form of a regular tetrahedral diamond pyramid with an apex angle of 136° at a load of 50 gf.

RESULTS AND DISCUSSION

To date, there are many known methods for assessing the fragility of materials on the basis of the results of microindentation. The most popular of them is the calculation of the critical stress intensity factor on the basis of the maximum length of a radial crack [12–14] and the average length of radial cracks [15, 16], determination of the Palmquist parameter taking into account the length of all radial cracks [17, 18], and assessment of the microfragility index taking into account the number and type of cracks and chips [2].

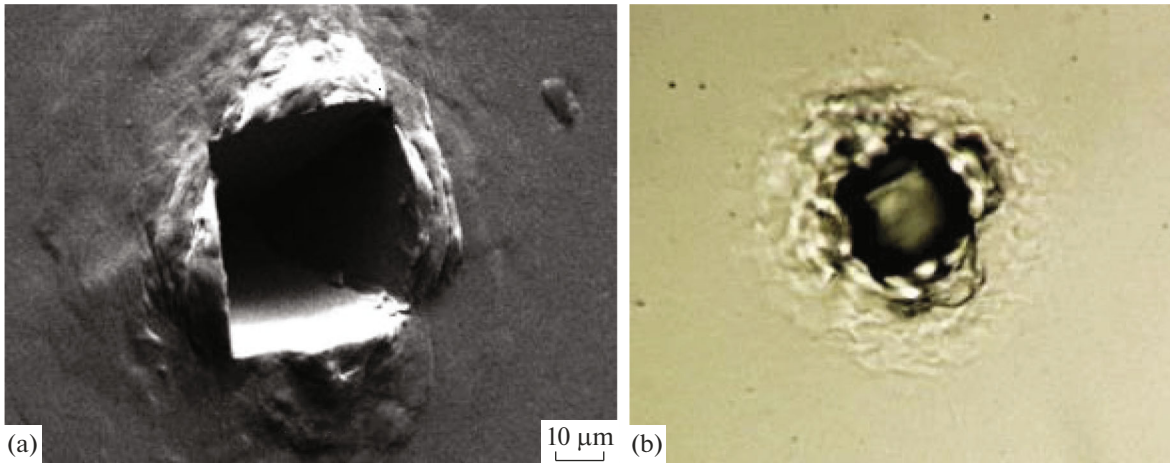


Fig. 6. The deformation pattern near the indenter imprint in the form of a regular four-sided diamond pyramid with an angle at the apex of 60° : (a) electron microscopy of the imprint ($\times 1000$); (b) optical microscopy of the imprint ($\times 600$).

However, all these methods are applicable only for the assessment of brittle materials, the indentation of which leads to the appearance of cracks and chips near the imprint.

There is also a well-known method for mathematical processing of the results of microhardness measurements to assess the accumulated damage in the metal of a structure and predict its residual life [19]. In this method, it is proposed to calculate the metal damage coefficient on the basis of a comparative analysis of the number of “hardened” and “softened” grains after a series of microhardness measurements in the metal in its original state. This approach cannot be called optimal, since the values of microhardness during random indentation are influenced by a large number of factors, both related to the occurrence of degradation processes (hardening, formation of microcracks, aging) and not related to them (falling into various metal phases, grain boundaries, chemical heterogeneity, etc.). At the same time, it is obviously difficult to correctly identify these processes and take into account the degree of their manifestation and influence on the properties of the metal only by changing the microhardness values.

From the results presented above, a method can be proposed for assessing the degree of embrittlement of structural steels that have relatively high plasticity on the basis of obtaining microhardness values and analyzing the morphology of localized shifts in the imprint zone. The method consists in determining the fragility index of the metal

$$K_h = \frac{0.01}{n} \sum_{i=1}^n HV_{\mu_{-i}(h)} Z_{p(h)}, \quad (1)$$

where K_h is the value of the metal fragility index at the depth h ; $HV_{\mu_{-i}(h)}$ is the i th value of the microhardness of the metal in a series of measurements at the depth h , kgf/mm^2 ; n is the number of imprints in a series of

measurements at the depth h ; and $Z_{p(h)}$ is the total fragility score of the metal at the depth h . The latter is calculated using the formula

$$Z_p = 0n_1 + 1n_2 + 2n_3 + 3n_4 + 4n_5, \quad (2)$$

where $n_1, n_2, n_3, n_4,$ and n_5 are the relative numbers of imprints from their total number in a series of measurements with a given fragility score in accordance with Table 2.

Microindentation is carried out using an indenter in the shape of a regular tetrahedral diamond pyramid with an apex angle of 136° under a load of 50 gf. In this case, it is advisable to evaluate the morphology of localized shifts from the imprints deposited on an unetched surface and the microhardness values from the imprints deposited into the main elements of the structure on the etched surface.

CONCLUSIONS

It has been established that the morphology of localized shears that appears near the imprint during microindentation of ductile structural steels reflects the deformability of the structure and can be used as an informative criterion for its brittleness.

A method has been developed for assessing the embrittlement of structural steels according to the results of microindentation, which can be recommended for nondestructive testing of assessing the state of the metal of structures.

Carrying out additional research to establish an empirical relationship between the proposed fragility indicator and mechanical characteristics of the metal (primarily crack resistance), taking into account the influence of structural factors and chemical composition, will make it possible to reliably predict the strength and durability of critical steel structures under various operating conditions.

ABBREVIATIONS AND NOTATION

K_h	value of the metal fragility index at the depth h
$HV_{\mu_i(h)}$	i th value of the microhardness of the metal in a series of measurements at the depth h , kgf/mm ²
n	number of imprints in a series of measurements at the depth h
$Z_{p(h)}$	total fragility score of the metal at the depth h
$n_1, n_2, n_3,$ n_4, n_5	relative numbers of imprints

FUNDING

This work was supported by ongoing institutional funding. No additional grants to carry out or direct this particular research were obtained.

CONFLICT OF INTEREST

The authors of this work declare that they have no conflicts of interest.

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Translated by Sh. Galyaltdinov

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