UDC 539.32

APPLICATION OF NANOINDENTATION FOR INVESTIGATION OF RADIATION DAMAGE IN SS316 STAINLESS STEEL

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The possibility of application of nanoindentation technique for investigation of mechanical properties (hardness and modulus of elasticity) of SS316 steel irradiated with 1.4 MeV Ar^+ ions to a dose of $1\cdot10^{17}$ cm^{-2} at 900 K has been considered. Methods of experimental data processing for extracting of physical characteristics of materials, in particular, hardening at irradiation to 25 dpa are analyzed. The influence of different factors on hardening is discussed involving results of transmission and scanning electron microscopy.

INTRODUCTION

Currently, nanoindentation techniques are extremely popular among the specialists involved in the development and use of nanostructured materials. The intense progress of nano technologies required the advance of methods for investigation and characterizing of physical-mechanical properties of materials in nano-level.

Nanoindentation (depth sensing testing, ultra-low-load indentation, instrumented indentation) is the absolute leader of such testing, suitable for solution of different physical problems and clarify the fundamental laws of behavior of nanometer near-surface layers and submicron volumes of various materials. The term nanoindentation (NI) means all methods with the use of precise local load effect on material and simultaneous registration of deformation response with nano meter resolution [1–3].

Operation mode of nanoindenters is realized by penetration of geometrically certified indenter under the action of prescribed profile of normal force $P(t)$ and simultaneous registration of the depth of its penetration into the material $h(t)$.

It is noted in the review [4] that one of the attractive distinction of NI is the possibility to obtain the different quantitative characteristics of material. Approximately for a half of these characteristics there are well accepted determinations, standards, algorithms and programs of realization. These are: Young`s modulus E, hardness H, fracture toughness K1c, loading diagram $\sigma = f(\varepsilon)$, determined by diagram $P = f(h)$ and more than two tens of characteristics.

Another group of characteristics have not the accepted ways of description and methods of experimental characterization. They may be estimated by different methods having comparative or semi-quantitative character. These are: a) parameters of different scale effects, conditions of their manifestation, boundaries of mechanical properties invariance respectively to variation of specimen size and morphological units of its structure; b) conditions of polymorph transformation induced by high contact pressure under indenter; c) dependence of properties on depth in high-gradient materials; d) position of boundaries and separate determination of properties of the film and substrate in fine-film structures without preparation of transverse metallographic specimen and surface etching; e) adhesion, parameters of peeling and fracture of film coatings [5].

Promising is the possibility to use nanoindentation during investigation of radiation resistance and mechanisms of radiation damage on charged particles accelerators since experiments with ion irradiation has an essential disadvantage – the low depth of damaged layer, that make difficult the correct description of radiation phenomena and study of mechanical properties of material. Nanoindentation is practically the one experimental technique for obtaining quantitative rather than estimated values characterizing the set of mechanical and operational properties of surface layers in nano- and sub- microvolumes.

In [6] the first results on hardening of 316 steel irradiated with ions of deuterium, helium and argon were obtained. These results showed an increase in steel hardness of approximately two times after irradiation but did not allow to fully extract quantitative characteristics and to specify structural aspects of the mechanisms of radiation hardening.

The goal of present paper is the use of the nanoindentation method for investigation of hardness and modulus of elasticity of SS316 steel in initial state and after irradiation with 1.4 MeV Ar^+ ions to a dose of 25 dpa and to develop the methods for obtaining of macroscopic characteristics of materials from results of nano meter scale.

EXPERIMENTAL TECHNIQUE

Specimens of SS316 steels with dimensions of 27x7x0.1 mm were preliminary solution annealed at temperature 1340 K in vacuum 10^{-4} Pa during one hour. To clean the steel surface from impurities specimens were electropolished in electrolyte composed of 530 ml of glycerin, 300 ml H_3PO_4 and 80 ml H_2O. The polished surface quality was inspected with "MIM-2P" microscope.

After electropolishing before irradiation and investigation specimens were short-term annealed to 1200 K in experimental chamber. Composition of steel is presented in Table.

Composition of steel 316, weigh %

C	Si	Mn	P	S	Cr	Ni	Mo	Ti	Fe
0,06	0,67	1,77	0,035	0,015	16,68	12,01	2,39	0,01	Bal.

Specimens were irradiated in electrostatic accelerator “ESU-2” with 1.4 MeV Ar^+ ions to a dose of $1\cdot10^{17}\,cm^{-2}$. The current density of argon ions was $10^{13}\,cm^{-2}\cdot s^{-1}$. The temperature of irradiation was chosen to be 900 K and was registered by chromel-alumel thermocouple.

Microstructure of irradiated specimens was studied by transmission electron microscopy at room temperature using standard bright field technique. Surface structure transformations were studied using scanning electron microscope JEOLJSM-7001F 00.

Nanohardness was measured by Nanoindenter G200 with the use of tripod Berkovich pyramid. The first data about the mechanical properties was obtained from simple loading-unloading test, which gives general information and basic characteristics such as hardness and modulus of elasticity and also reports about irreversible processes. Fig. 1 shows the diagram of indenter penetration obtained during nanoindentation of irradiated specimen of SS316 steel.

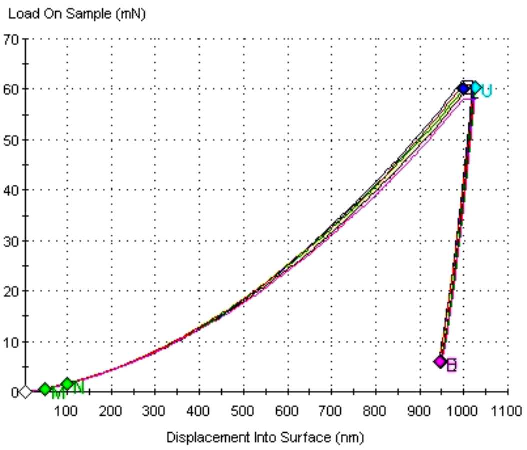
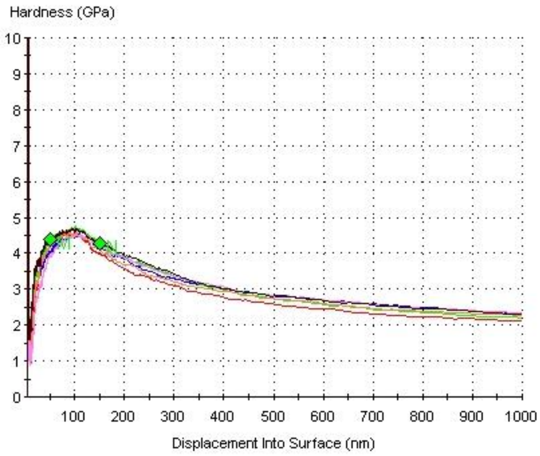


Fig. 1. Curve loading-unloading

The most common method of data analysis in nanoindentation is a method of Oliver and Pharr allowing determination of hardness and elastic modulus of specimen without measurement of its dimensions by direct methods [7]. However, a significant disadvantage of the classical method of Oliver and Pharr is that the hardness and elastic modulus of specimen ~~of~~ in such tests can be determined only with maximal load on indenter which is not always convenient and needs long-time tests.

We have realized tests using the method of continuous stiffness measurement with continuous registration of the curve of loading and unloading [8]. The method consist in superposition of harmonic disturbance $F=F_0\sin\omega_{0t}$ with small amplitude on the slowly varying test load and in measurement of amplitude and phase of indenter displacement on frequency ω_0 (usually from 1 to tens of Hz) using synchronous detector. Due to such modulation the areas of short-time load decrease appear periodically on the curve of indenter penetration.

As a result, continuous dependence of hardness and elastic modulus of the sample from the depth of indentation is obtained according to the data of one test (Fig. 2). Wherein, the rate of deformation in the contact remains constant.



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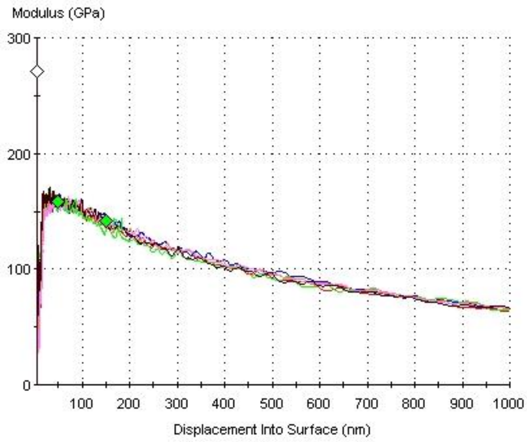

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Fig. 2. Hardness (a) and elastic modulus (b) of SS316 steel irradiated with 1.4 MeV Ar^+ ions

RESULTS AND DISCUSSION

Fig. 3 shows the dependence of initial SS316 steel nanohardness from the depth of indenter penetration. Measurements of nanohardness were performed up to the depth of 1000 nm. Imprints were made on distance 15 μm one from another. The figure shows the data of 8 imprints.

Measured values of hardness have the high discordance for the depth up to 50 nm due to the inaccuracy in determination of geometry of indenter head and effects of deformation rates round the pyramid head and also due to other surface artifacts such as the film contamination, for instance.

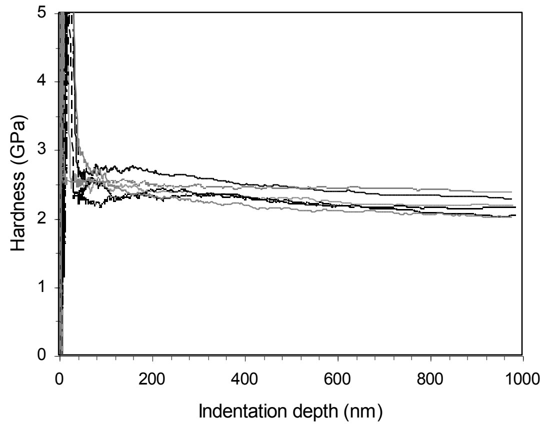


Fig. 3. Dependence of hardness of SS316 steel in initial state from the depth of indenter penetration

In general case several areas may be distinguish on the curve. In first area (0…30 nm) a rapid increase of pressure is observed that may be explained by the fact that Berkovich indenter is not ideally sharp. On indenter vertex there is a blunting in the shape of sphere (in our case the sphere has the radius ~ 230 nm according to data of atom-force microscopy). Spherical blunting induces the formation of initial elastic area.

In second area from 30 to 60 nm elastic-plastic transition is observed; the last is induced by that with the increase of indenter penetration depth the fraction of spherical peak in contact decreases and transition to pyramidal indenter is observed. Such smooth transition is observed during heterogeneous nucleation of dislocations in contact (another reason – multiplication of existing dislocations) [9]. Plastic flow in specimen starts only from depth ~ 60 nm when indentation is realized just by pyramid. The control of hardness became possible from this depth.

For initial non-irradiated specimens the value of nanohardness decreases slowly with the depth of penetration. Such behavior is due to methodological distinctions: with the increase of nanoindentation depth the measured hardness decreases and reaches gradually the value of macro hardness.

As it is seen from Fig. 3 on depths 150…1000 nm the tendency of dependencies is the same, but some spread of data (~ 15%) is observed. In the present paper 10 measurements were carried out on each specimen and then results were averaged. The averaged value of nanohardness in non-irradiated specimens of steel was ~ (2,3±0,3) GPa on depth up to 1000 nm.

Fig. 4 shows the dependencies of hardness versus depth of indenter penetration for specimen irradiated at room temperature with 1.4 MeV argon ions to a dose of $1\cdot10^{17}\,cm^{-2}$. Similarly to initial specimens profiles of hardness after irradiation show the effect of hardness increase on low depth of nanoindentation, so called ISE (indentation size effect).

According to the methodology adopted by international community the first parts of curves (depth 0…50 nm) having the artifact origin are not shown. Data are presented for two imprints showing the highest data spread. Structure of specimen surface with indentations is shown on Fig. 4,b,c.

Relative data spread for irradiated specimen exceeds slightly the data spread obtained from initial specimen. But the maximal variation in hardness obtained on depths 100…500 nm was 0.8 GPa and approximately twice exceeds the mean spread for initial specimens. Indentations on irradiated specimen fall on boundary and body of the grain (see Fig. 4,b,c). It may be supposed that higher value of hardness obtained for indentation #1 is due to the effect of grain boundary.

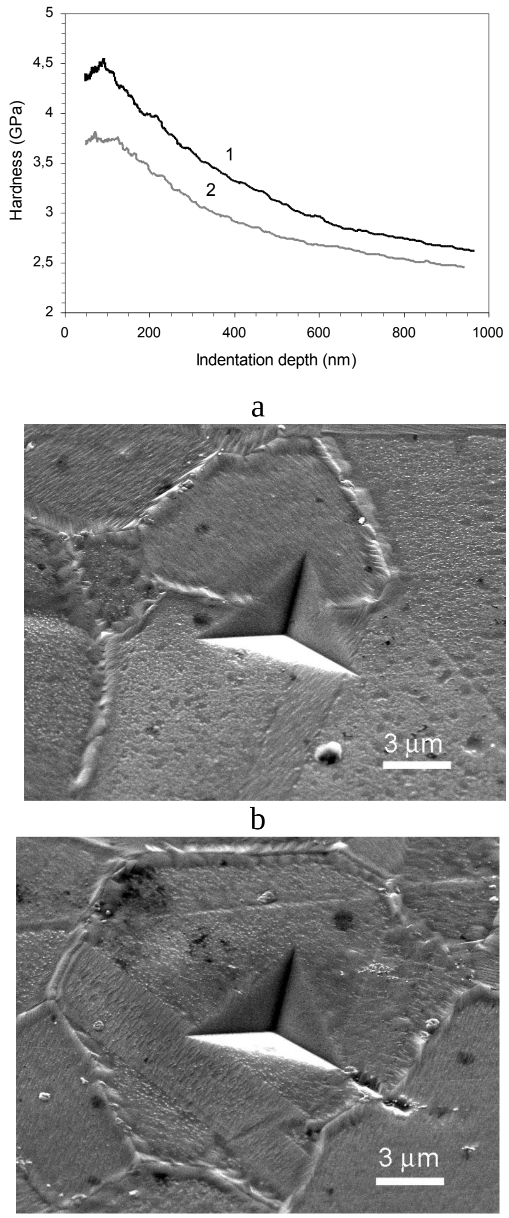
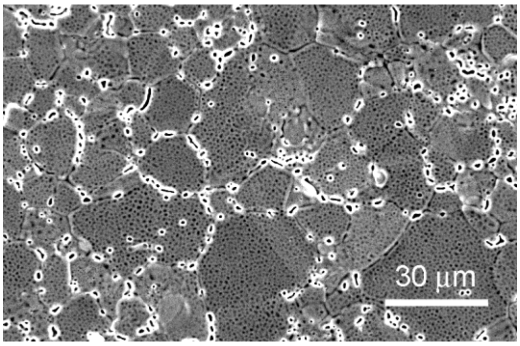


Fig. 4. Hardness of SS316 steel irradiated at 900 K with 1.4 MeV Ar^+ ions to a dose of $1\cdot.10^{17}$ cm^{-2} measured on boundary (1) and in grain body (2) (a). Surface of specimen with imprints of indentation # 1 (b) and # 2 (c)

There is a contradict information about the effect of grain boundaries on hardness [10–12]. Majority of authors have observed hardening with the indentation approaching to boundary. They have explained this effect by difficulty of gliding transfer into the neighboring grain. Decrease of hardening was rarely observed with the approaching to grain boundary. It was noted in [4] that on initial stage of Berkovich indenter penetration the grain boundaries are on large distance and can't participate into deformation.

On the other hand quantitative characteristics of hardening reflect the integral character of nucleation and movement of dislocations, their interaction with lattice, among themselves and other structural imperfection of crystals. Under irradiation the variety of different radiation defects contribute to hardening; these defects are: isolated point defects, clusters, vacancy and interstitial dislocation loops, precipitates of new phase, vacancy and gas-filled voids which make difficult the free movement of dislocation [13].

In present experiments SS316 steel was irradiated with ions of inert gas-argon at temperature 900 K, in conditions where gaseous bubbles are formed effectively. Swelled grain boundaries (see Fig. 4,b,c) indicate the accumulation of argon bubbles on them. However TEM and SEM studies of steel structure had showed that gaseous bubbles are formed not only on grain boundary but also into the grain body (Fig. 5).



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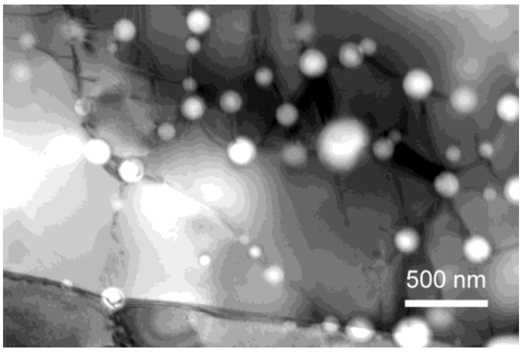

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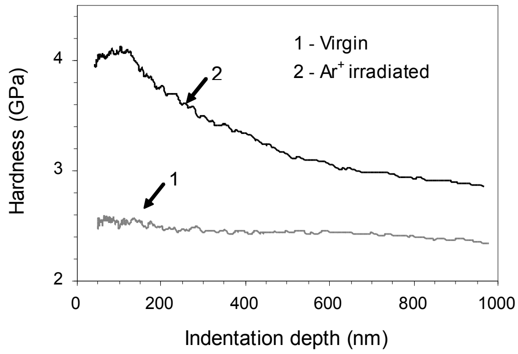
Fig. 5. Microstructure of SS316 steel irradiated at 900 K with 1.4 MeV Ar^+ ions to a dose of $1\cdot10^{17}cm^{-2}$ obtained by means of SEM (after electro polishing) (a) and TEM (b)

It may be supposed that gas bubbles located in the grain body or on its boundary influence differently on dislocation movement during nanoindentation. But this assumption must be confirmed experimentally.

Fig.6 shows profiles of hardness for initial and argon irradiated specimens of SS316 steel and also calculated profiles of radiation damage and argon concentrations for irradiation of steel with 1.4 MeV Ar^+ ions to a dose of $1\cdot10^{17}$ cm^{-2}. Maximal (calculated) value of damage is 60 dpa, concentration of implanted argon – 4.2 at.%. Depth of damage extends up to ~ 800 nm [14].

The comparison of the two hardness profiles shows that irradiation causes (leads to) an increase of hardness nearly 1.7 times in the depths of 50…125 nm. Decrease of the hardness with an increase of indentation depth is related to the effect of soft base (see Fig. 6,a, curve 2) [15]. During nanoindentation, as it is shown in [16–18], plastic zone around the indenter tip extends well below it on depth approximately 4…7 times larger than the depth of indentation. So, the hardness of softer non-irradiated part of material located beyond the ion ranges dominates at hardness measurement of irradiated area. In the same time the highest value of hardness on depth 100…150 nm is induced by maximum of damage on depth 500…600 nm (see Fig. 6,b).



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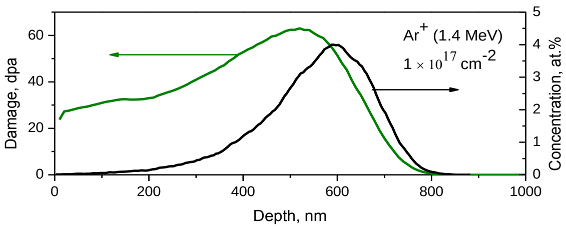

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Fig. 6. Profile of hardness of steel 316 for initial (1) and argon irradiated (2) specimens (a); designed profiles of damage and concentrations of argon with energy of irradiation 1.4 MeV to dose $1\cdot10^{17}cm^{-2}$ (b)

Method of determination of actual relative hardening due to radiation was proposed in [17]. It means the subtraction of the hardness measured in initial material from the hardness measured in irradiated material. For specimens irradiated with argon ions the maximum increase of nanohardness after irradiation was determined to be ≈ 1.7 GPa.

With the development of the method of nanoindentation, especially for its use in ion-implanted specimens other approaches were examined for determination of hardening not in nano-region, but also for macroscopic volume of material.

It was supposed that at continuous indentation when radius of contact spot varies from atomic to macroscopic one the scale of the problem passes successively the different structural level of deformation. In the result of such scanning each level having characteristic dimension will add its special features in scale effects.

One of most developed approaches to size effects is the use of concept of geometrically necessary dislocations [19]. According to this concept size effects arise in the case where the typical dimensions of deformation (specimen cross-section) became lower than the characteristic length $h*$. On the base of these representations Nix and Gao [20] have derived the relation

$$\left(\frac{H}{H_0}\right)^2 = 1 + \frac{h^*}{h},$$

where H – hardness; h – indenter penetration depth; H_0 – limited hardness on infinite depth. Experimental data rearrange as $H^2 = f(1/h)$, as it is shown on Fig. 7. Square root from H^2_0 obtained on intersection of the curve with axis H^2 will give the value of hardness for the bulk material.

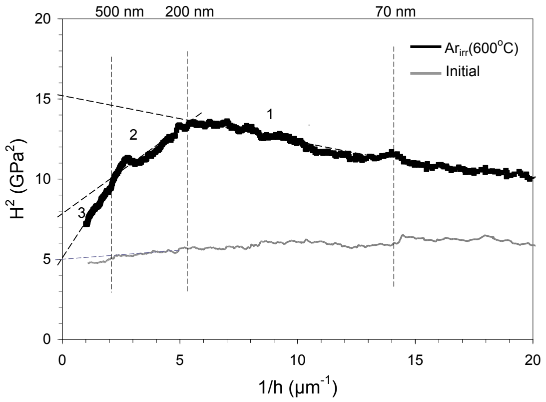

Fig. 7. Dependence of H^2=f(1/h) for initial and argon irradiated specimen of steel 316

For initial specimen practically linear dependence is observed while for irradiated specimen the curve has inflections in the region of 200 and 500 nm. The data over the inflection point, which is indicated by the arrow 1, could be interpreted as the hardness of the irradiated region. The bulk hardness H_0 estimated from this irradiated region was 3.9 GPa. The data below the inflection point indicated by arrow 2 included the hardness of both the irradiated and unirradiated regions and 3 – unirradiated region [18]. The hardness values H_0 obtained by such processing for the initial samples are 2.2 GPA. Obtained values of elastic modulus are: 181.7 GPa for initial steel and 183.9 GPa for argon irradiated steel.

CONCLUSION

Methodology of nanoindentation was tested during measurement of hardness and elastic modulus of SS316 steel in initial state and after irradiation with 1.4 MeV Ar^+ ions to a dose of 25 dpa at 900 K .

It is shown that this methodology is a suitable tool for determination of radiation-induced hardening in damaged layer on the depth of several hundred of nanometers.

Processing of experimental data for initial and irradiated specimen requires consideration of size effects when with the decrease of the depth of nanoindentation the hardness increases.

Relative hardening due to radiation can be obtained by subtracting the hardness measured in the initial material from the hardness measured in the irradiated material. To extract hardness values corresponding to the bulk specimen the treatment of data must be performed taking into account the theory of geometrically necessary dislocation and plotting of dependence H^2=f(1/h).

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Article received 19.08.2015

ПРИМЕНЕНИЕ МЕТОДА НАНОИНДЕНТИРОВАНИЯ ДЛЯ ИССЛЕДОВАНИЯ РАДИАЦИОННОЙ ПОВРЕЖДАЕМОСТИ СТАЛИ 316

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Рассмотрена возможность применения метода наноиндентирования при исследовании изменения механических свойств (твердости и модуля упругости) стали 316, облученной при 900 К ионами Ar^+ с энергией 1,4 МэВ до дозы $1\cdot10^{17}$ см$^{-2}$. Анализируются способы обработки экспериментальных данных с целью извлечения физических характеристик материала, в частности, его упрочнения при облучении до 25 сна. Обсуждается влияние различных факторов на упрочнение стали 316 с привлечением данных просвечивающей и сканирующей электронной микроскопии.

ЗАСТОСУВАННЯ МЕТОДУ НАНОІНДЕНТУВАННЯ ДЛЯ ДОСЛІДЖЕННЯ РАДІАЦІЙНОЇ ПОШКОДЖУВАНОСТІ СТАЛІ 316

Г.М. Толмачова, Г.Д. Толстолуцька, С.О. Карпов, Б. С. Сунгуров, Р.Л. Василенко

Розглянута можливість використання методу наноіндентування при дослідженні змін механічних властивостей (твердості і модуля пружності) сталі 316, опроміненої при 900 К іонами Ar^+ з енергією 1,4 МеВ до дози $1\cdot10^{17}$ см$^{-2}$. Аналізуються способи обробки експериментальних даних з метою вилучення фізичних характеристик матеріалу, зокрема, його зміцнення при опромінені до 25 зна. Обговорюється вплив різних чинників на зміцнення стали 316 з залученням даних просвічуючої і скануючої електронної мікроскопії.