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Effect of High-Pressure Treatment on Structure and Properties of Invar Fe–35% Ni Alloy

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The effect of uniform high-pressure (HP) treatment on change of the internal structure hierarchy of polycrystalline Invar f.c.c. Fe–35.0% Ni alloy in quenched state and after hydroextrusion (HE) is studied. As shown, the treatment of the quenched alloy by pressure of 2 GPa is accompanied by the relief formation on the cross-section surface that is caused by reorientation of available grains and by a change of grain-boundaries' state. The increase of density of twins of deformation and the saturation of structural elements by vacancies and dislocations promoting the grain-boundary slipping is revealed. The HP processing of the previously HE treated samples of the Invar alloy ($\varepsilon = 0.96$ and 1.12) is not accompanied by notable distinctions in the structures' hierarchy with the exception of both the decrease of texture and the increase of twins' density that is caused by partial stress relaxation.

Досліджено вплив оброблення всебічним тиском (ВТ) на зміну структурної ієрархії полікристалічного інварного стопу ГЦК-Fe–35,0% Ni в загартованому стані, а також після оброблення гідроекструзією (ГЕ). Показано, що оброблення тиском у 2 ГПа стопу в загартованому стані супроводжується формуванням на поверхнях шліфа рельєфу, що обумовлено переорієнтацією в просторі зерен і зміною стану їх меж. Виявлено збільшення густини двійників деформації і насичення структурних елементів вакансіями та дислокаціями, які сприяють зерномежовому проковзуванню. Оброблення ВТ попередньо деформованих гідроекструзією зразків інварного стопу ($\varepsilon = 0{,}96$ і 1,12) не супроводжується помітними відмінностями в ієрархії структури, за винятком зменшення текстури і збільшення густини двійників, що є результатом часткової релаксації мікронапружень.

Исследовано влияние обработки всесторонним давлением (ВД) на изменение структурной иерархии поликристаллического инварного сплава ГЦК-Fe–35,0% Ni в закалённом состоянии, а также после обработки гидроэкструзией (ГЭ). Показано, что обработка давлением в 2 ГПа сплава в закалённом состоянии сопровождается формированием на поверхностях шлифов рельефа, обусловленного переориентацией в пространстве зёрен и изменением состояния их границ. Обнаружено увеличение плотности двойников деформации и насыщение структурных элементов вакансиями и дислокациями, способствующими зернограничному проскальзыванию. Обработка ВД предварительно обработанных гидроэкструзией образцов инварного сплава ($\varepsilon = 0{,}96$ и 1,12) не сопровождается заметными различиями в иерархии структуры, за исключением уменьшения текстуры и увеличения плотности двойников, являющихся результатом частичной релаксации микронапряжений.

Key words: high-pressure treatment, invar alloy, microstructure, stress concentrators, microhardness.

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1. INTRODUCTION

The Invar f.c.c. alloys based on Fe–(29–36)% Ni with a low thermal expansion coefficient (TEC) of the order of $1.2 \cdot 10^{-6}\,K^{-1}$ in relatively wide temperature range are used as a precision material in the production of highly precise instruments with the thermally stable parameters.

To increase the structural stability state and properties, such products are subjected to various influences: ageing, annealing, deformation, including the applied high pressure (HP). The HP treatment is one of the most effective methods for impact on the structural state of alloys. However, its effect on the structure and properties of Invar alloys was not enough studied although the main notions of the HP influence are set out both in theoretical and experimental studies [1–13]. Their analysis reveals the following. Theoreticians considering the problem from the spherical deformation tensor point to the necessity to consider the type and properties of crystal lattices of the processed materials and the changes in their elastic modulus [1–6]. In so doing, attention is paid to peculiarities of the existing structural defects behaviour as well as the generation, interaction and the spatial distribution of new ones both under the HP processing and when combining it with various scheme of cold plastic deformation [5, 7–11].

It was revealed in experimenters that the HP treatment can have a plasticizing effect on metals and their alloys by suppressing the formation and development of cracks [1, 2, 12]. In addition, under the strain, it contributes to easier formation of cellular (grains) structures with the different dispersion and the degree of crystallographic disorientation between adjacent elements in both single crystals [5] and polycrystalline states of metals with f.c.c. [13] and b.c.c. [14] lattices.

Currently, there are no works, which study the peculiarities and regularities of the behaviour of metals and alloys under the HP treatment and consider the processes occurring in them in terms of modern concepts about stress concentrators [15, 16]. They are always available in real metallic materials and responsible for peculiarities of the plastic deformation development through changing the value of strains occurred in their tops and determine the conditions of generation (absorption) of defects by corresponding relaxation mechanisms: the vacancy mechanism, dislocation one or brittle cracks formation mechanism. In so doing, if for high-symmetry crystal systems of single-crystal lattices states the spherical deformation tensor can be conditionally implemented (excluding the impact of technological concentrators) for the anisotropic material (the isotropic state, $A = 1$), in polycrystalline samples the presence of the grain-form structural hierarchy and their boundaries, on the strength of specific character of their properties manifestation at the boundary slipping under external forces, create unattainable conditions for uniform hydrostatic pressure at the initial stage of HP processing [17–19].

Thus, the aim of this study is to investigate the effect of HP treatment on change of the hierarchy of the internal structure of polycrystalline Invar f.c.c. Fe–35.0% Ni–0.49% Mn alloy in the hardened state and after pre-treatment with hydroextrusion in the context of modern conceptions about the features of participation of currently known mechanisms of stress relaxation in the tops of always present concentrators in polycrystalline materials and real metal products.

2. MATERIAL AND STUDY METHODS

The industrial Invar alloy of the chemical composition Fe–35.0% Ni, 0.07% Cu, 0.03% Co, 0.49% Mn, 0.03% C (wt.%) (GOST 10994-74; hereafter, Fe–35.0% Ni) with the f.c.c. crystal lattice is studied. The cylindrical shaped samples with the 21 mm in diameter and 100 mm in length were annealed in vacuum at 1373 K for 30 min and subsequently quenched in oil at room temperature. The rectangular shaped plates of 200 μm in thickness were in the initial quenched state as one part of the samples. Another annealed part was deformed by hydroextrusion (HE) that implies in bursting out a sample from a closed container through a die under the fluid pressure of 1.5 GPa as described in [20]. The standard expression $\varepsilon = 2\ln d_0/d$ was used to determine the strain degree of 0.96 and 1.62, where d_0 and d are the sample diameters before and after HE. The procedure was repeated with 2 and 4 passes using a smaller die diameter for the strain accumulation.

Each sample was finally HP processed in a working chamber under

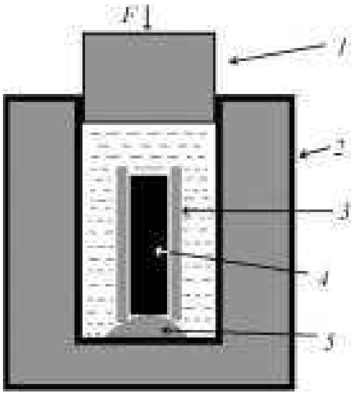


Fig. 1. The scheme of the sample *4* location under the HP processing in working chamber *2*: *1* is the plug, *3* is the sample holder, *5* is the rubber pad. Liquid is industrial mineral oil.

the 2 GPa for 3 min in accordance with the scheme in Fig. 1. The relevant pressure level was reached by the periodically switching of press pump with the loading and unloading rate of 0.1 $\mathrm{GPa \cdot s^{-1}}$. The reduction of pressure to atmospheric one was carried out for 20 sec. There are no reasons to assume that hydrostatic loading conditions were provided due to peculiarities of a sample shape and its location in the chamber and because the solidification of the working liquid (mineral oil) at high pressure was possible. Therefore, hereafter, the case in point is the high pressure (HP) instead of hydrostatic pressure in the text.

The flat sample covered by the 100 μm rubber film was maintained in holder 3 between two plastic plates in order to fix it in the same position and to provide close treatment conditions. One surface of a sample before HP processing was chemically polished to remove a cold worked layer and to subsequent study of probable structural changes.

The X-ray diffraction, metallography, transmission electron microscopy and durometry were used to study structure and properties of the alloy before and after the HP treatment. The X-ray DRON-3M diffractometer with the CoK_α radiation, the optical 'NEOPHOT-32' and electron 'JEM-2000FX' microscopes were used. The distribution of the average diameters of structure elements were estimated by the computer technology. The diffraction patterns were taken as a rule from the entire area of an image. The finish treatment of foils for electron microscopy was the electropolishing in teflon holder in the electrolyte containing the 75 g of chromic anhydride, the 130 ml of glacial acetic acid, the 20 ml of water at the 90 V of power and the current of 0.5–1.5 A. Microhardness was measured by PMT-3 under the 50 g loading according to the requirements of GOST [21].

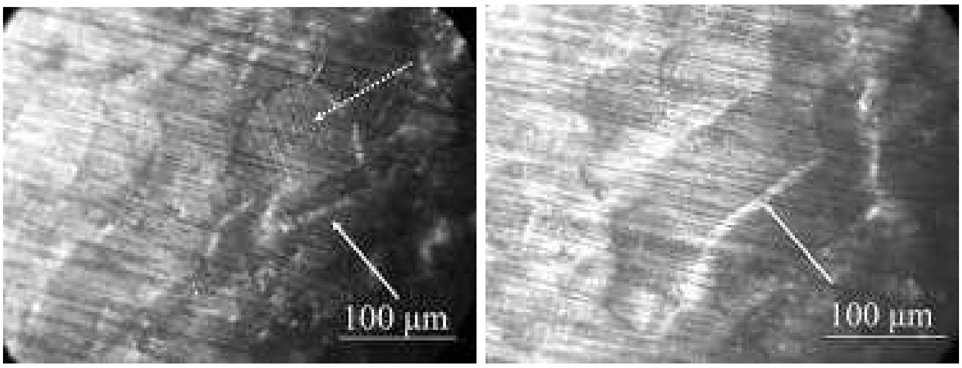
3. RESULTS AND DISCUSSION

Trying to revealed high-angle grain boundaries in metallographic analysis, the effect of grains displacement relative to each other that is accompanied by appropriate relief on a polycrystalline sample surface after the HP treatment of hardened state was found out (Fig. 2). The analysis of the images derived from a sample surface indicates that as its centre is approached (the diagonals intersection point), the relief and the grain boundaries appearance gradually weakens, but does not entirely disappear.

The parallel stripes inside individual grains in areas of the strongest relief exhibition (Fig. 2, *a*, indicated by dotted arrow) as well as the banding effect nearby the straight boundaries (Fig. 2, *a*, *b*, indicated by solid arrows) are observed rather frequently. This effect does not take place before HP treatment. Its appearance may be connected either with the changes in the position (width) of boundaries during the HP processing or with the twinning effect.

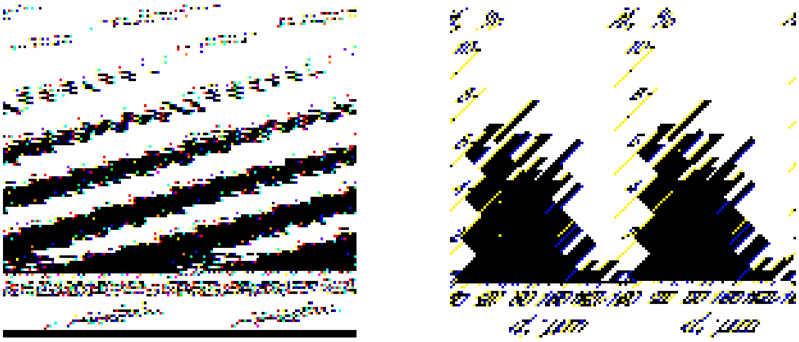
The appearance of the relief is apparently caused by both a deviation from conditions of the spherical deformation tensor, the effect of possible plastic deformation, which are associated with the shape of a sample, some differences of the pressure level in hardened worked liquid and the different relaxation processes at the grain-boundary sliding, in the interior volumes of grains, caused by the generation of vacancies and dislocations.

The results of comparison of the average grain diameters in initial state of the Fe–35.0% Ni alloy and after the HP treatment are presented in Fig. 3. Their analysis shows little changes in distribution of grains on average diameters indicating a decrease of more dispersive fraction with weak reduction in the distribution height due to increas-



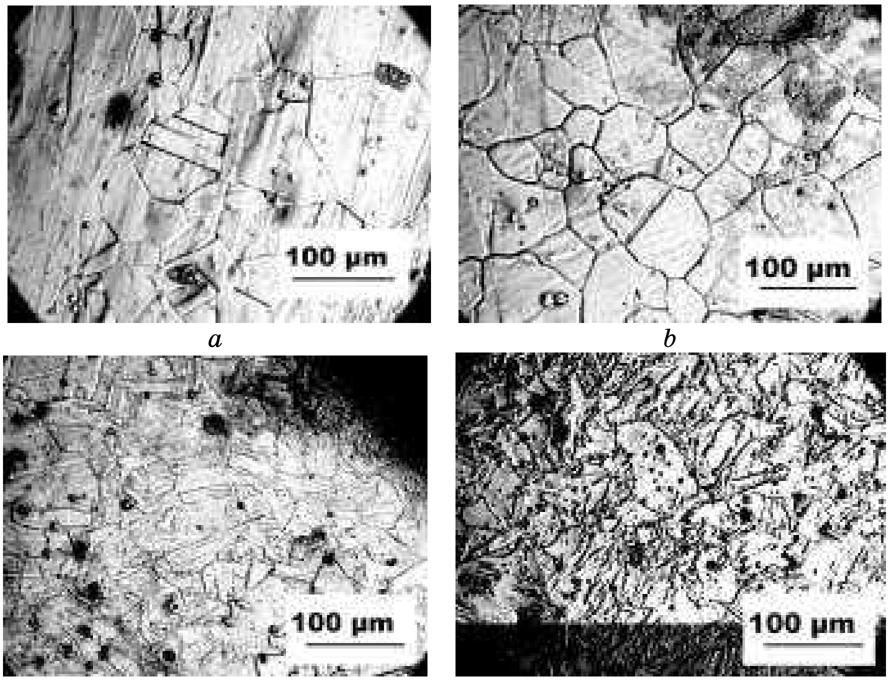
a *b*

Fig. 2. The grain structure relief on polished surface of the quenched Fe–35.0% Ni alloy after the HP processing (2 GPa).



a *b*

Fig. 3. Distribution of the average grain dimensions in the Fe–35.0% Ni alloy in initial hardened state (*a*) and after HP processing (*b*).



a *b*

c *d*

Fig. 4. The cross section microstructures of the Fe–35.0% Ni alloy after homogenization at 1100°C and subsequent quenching (*a*), the HP processing of initial quenched (*b*) and extruded with the compressions of $\varepsilon = 0.96$ (*c*) and 1.62 (*d*) samples.

ing the probability of the larger grains appearance.

The density of the planar formations on the etched surfaces of the cross sections after the HP treatment of the initial and hydroextruded samples was evaluated by means of standard metallographic technique (Fig. 4).

The analysis of the images shows that except growing etching of high-angle boundaries after the HP, influence of the frequency of meetings of the formations extended along the boundaries grows relating to initial and pre-strain states at common grinding of structural elements of the extruded samples.

The behaviour of the size of structural elements revealed in the Fe–35.0% Ni alloy by etching, which are formed under the above mentioned treatments was evaluated as well (Fig. 6).

The analysis of the obtained data shows insignificant increase (up to

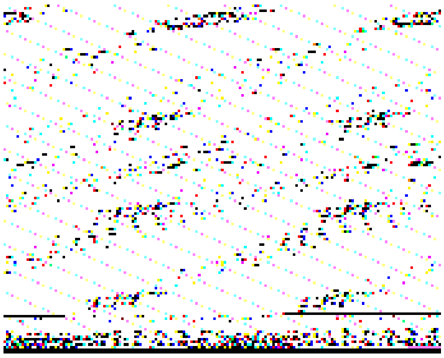


Fig. 5. The distribution of density ρ_d of the fine structural formations (twins), which are collinear to the grain boundaries *vs.* the degree ε of the HE deformation (□) and the subsequent HP treatment with 2 GPa (■).

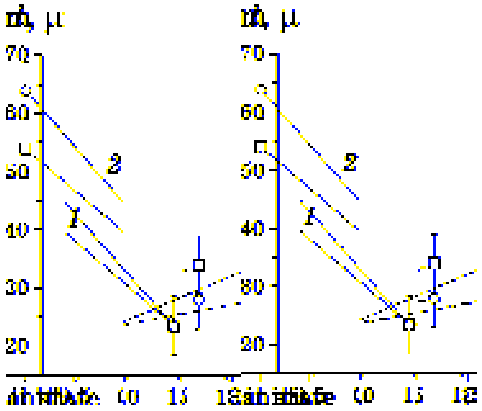
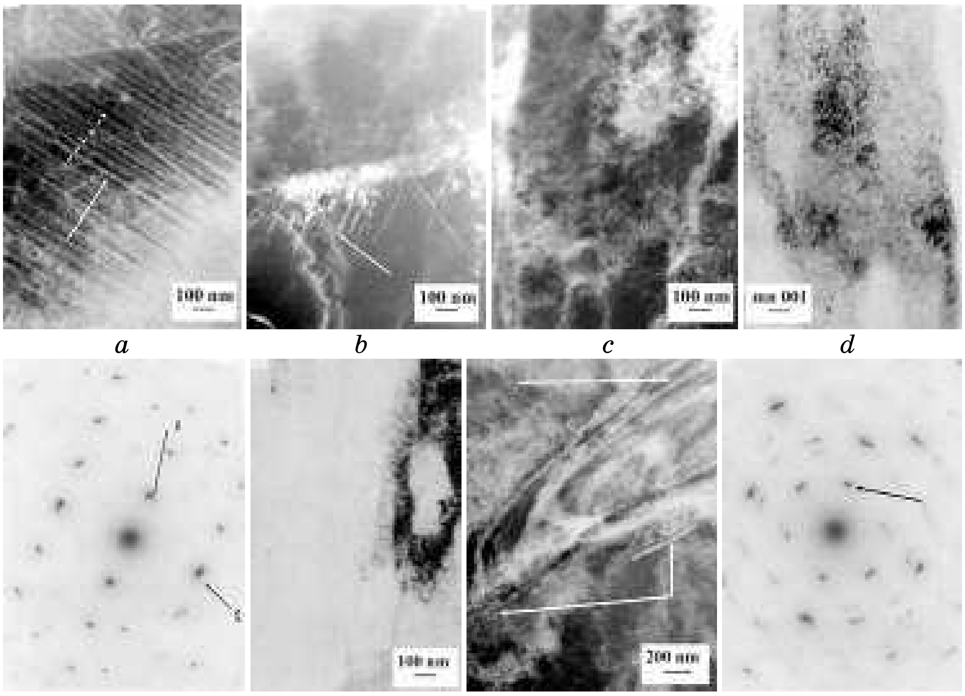


Fig. 6. The average size (d) of structural elements in the Fe–35.0% Ni alloy *vs.* the HE strain degree of $\varepsilon = 0.96$ and 1.62 (*1*) and after subsequent HP treatment (*2*).

16%) of the most probable average size of structural elements of the alloy after the HP treatment as compared to the quenched state, whereas the HP treatment of the extruded sample ($\varepsilon = 1.62$) is accompanied by the reverse effect (18%), indicating a reduction in mean diameter of the revealing structural elements at elevated etching susceptibility of their boundaries. The d value after the HE to $\varepsilon = 0.96$ coincides with this one after the subsequent HP processing.

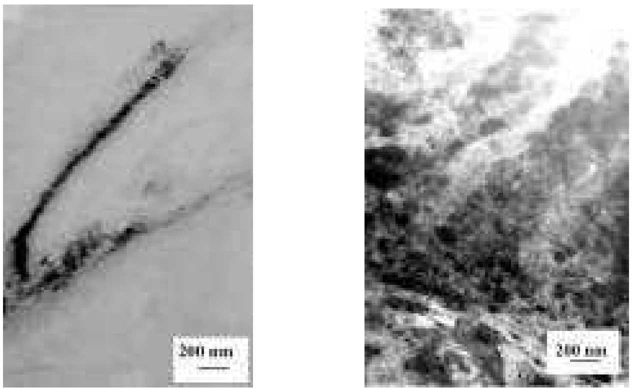
The results of the TEM studies of the HP treated samples are presented in Fig. 7. The distinctive features of annealed sample of the alloy after the HP processing are observed. The grain boundaries and the boundary zones, particularly at triple junctions, (Fig. 7, *b*) are the saturated ones by the crystal structure defects including linear ones (dislocations) of high density (approximately $5 \cdot 10^9$ cm^{-2}) indicated by solid arrows (Fig. 7, *a*, *b*), which alternate with the twins of deformation indicated by dashed arrow with the slightly expressed cross sliding (Fig. 7, *a*). All features of the structures presented in Fig. 2, Fig. 4, and Fig.



a *b* *c* *d*

e *f* *g* *h*

Fig. 7. Structure in cross-section of the Fe–35.0% Ni alloy after the HP treatment of initial quenched (*a*, *b*) and extruded sample with the degree of deformation of $\varepsilon = 0.96$ (*c*) and 1.62 (*g*, *j*). The dark field image (*d*, *f*, *i*) obtained from the areas presented on microstructures (*c*, *g*) in the reflexes indicated on electron diffraction patterns (*e*, *h*) by arrows, respectively.



i *j*

Fig. 7 *(continued)*.

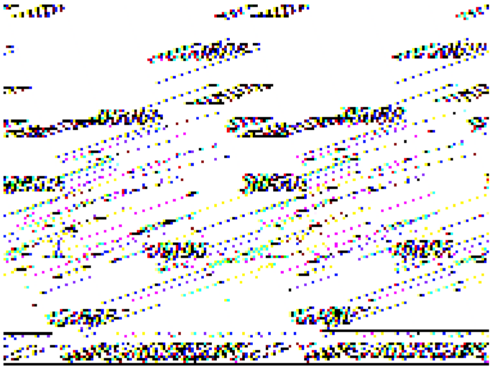


Fig. 8. The lattice parameter *a* of the Fe–35.0% Ni alloy vs. degree of deformation ε by HE [20] (□) and the subsequent HP processing (○).

7 show the development of several relaxation mechanisms of the occurring field stresses in a polycrystalline sample at the HP treatment: the grain-boundary sliding at vacancy and dislocation saturation as well as twinning.

As shown by X-ray diffraction studies, the HP treatment of initially quenched state is accompanied by the detecting of the residual compressive stresses as evidenced by decrease of the lattice parameter with respect to that for a quenched sample (Fig. 8). The TEM study results of samples pre-treated by HE and the subsequent HP treatment are shown in Fig. 7, *c*–*j*. One indicates that HP treatment with 2 GPa does not lead to significant changes in organization of the substructures and their dimensional parameters as compared to pure extruded state on retention of extremely high saturation of near-boundary zones of the individual grains with the defects of crystal structures of 10^{12}

cm^{-2}. By peculiarities of their location in a sample with respect to the extrusion direction, they are obviously the most active participants in the slipping process, which is going on with the formation of hydrodynamic state (Fig. 7, *g*). This state results from high transience HE deformation that demonstrates equiaxial structural elements including the most dispersed ones with the 10–40 nm in diameters (Fig. 7, *j*) at presence of system of thin elements (40 nm in thick) extending along boundaries remaining the deformation twins indicated by arrows in Fig. 7, *g*. The denoted structural changes are accompanied by recovery to the initial values of the alloy lattice parameter after the HP treatment with the increasing degree of the preliminary HE deformation (Fig. 8).

The comparison of the obtained TEM results that concerns studies of the extruded and then the HP treated samples with the results in [20] on structure analysis of the HE states has shown that the HP processing does not lead to any significant changes in organization of substructures and their dimensional parameters on retention of high density of defects up to 10^{12} cm^{-2}. A distinctive feature is only the increase in density of twins of different thicknesses with respect to those in the extruded state. The absolute majority of high-angle boundaries of the initial sample are transformed into states shown in Fig. 7, *g*, *i*, *j* pointing to the presence of a wider range of the dimensional parameters of structural elements both the equiaxed (more dispersed) and the larger ones, which extended in various directions and consist of smaller ones. The obtained results are in good agreement with the available literature data [22, 23], which point to the monotonic hardening in the grain boundary zones of pure iron at the pressure treatment up to 50 GPa after which the softening is observed. This is accompanied within the range of 5–15 GPa by nonmonotonic variation of density of dislocations and of their distribution.

The last one was not confirmed on the Invar alloy after the HP treatment of a pre-extruded samples, in which the pressure effect on the level of structural changes associated only with the increasing density of twins is accompanied by decreasing microhardness (Fig. 9) at more rapid increasing the crystal lattice parameter with the strain degree (Fig. 8, the curve HE + HP). The microhardness of the Invar sample after the quenching and reinforcing hydrostatic compression at 2 GPa increases in average by 200 MPa. At the same time, some decrease of microhardness of the HP treated pre-deformed samples is observed that is followed by growing scatter of its values relatively to those for the HE states (Fig. 9). The last one is possibly caused by changes in the stress state in the HE pre-deformed samples resulting from the HP treatment due to additional participation of twinning mechanism in its relaxation.

The analysis of the X-ray diffraction data indicates that the HP pro-

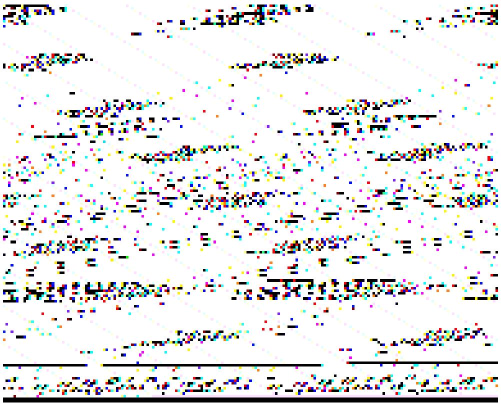


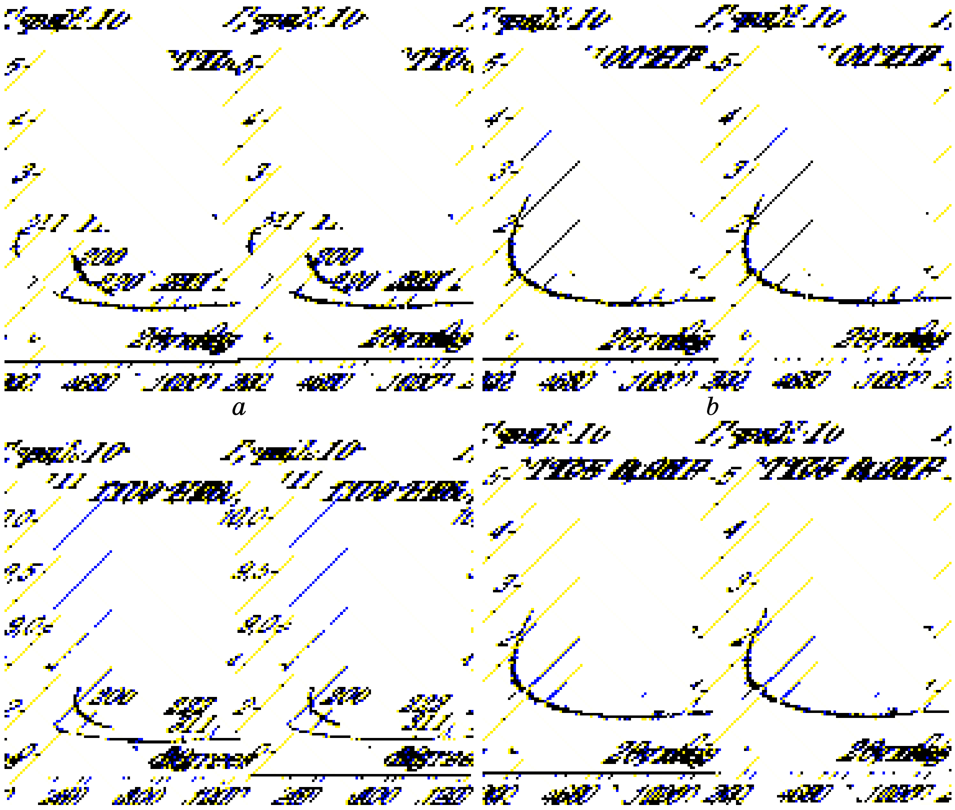
Fig. 9. The change of microhardness (H_μ) of the Fe–35.0% Ni alloy after the different degrees of plastic deformation (ε) by HE (□) and the subsequent HP treatment (○).

cessing of initial state is accompanied by decreasing the ratio of integral intensities of the diffraction lines J_{200}/J_{111} from 0.56 to 0.12 (Fig. 10, *a*, *b*) (for reference the ratio is 0.35). It points to the fact that the HP treatment of the annealed and subsequently quenched sample is accompanied by residual (inelastic) phenomena.

The significant increase in the integral X-ray diffraction line (111) intensity and the reduction of the J_{200}/J_{111} ratio to 0.16 was revealed on the HE treated sample for both strain degrees of 0.96 and 1.62. The example for $\varepsilon = 0.96$ is shown in Fig. 10, *c*. The intensity of the (311) line is significantly reduced and the (220) line completely disappears that indicates appearance of the crystallographic texture, which was also detected after the HE of the Invar alloy by TEM and X-ray diffraction techniques [20].

After the HP treatment of the extruded sample, the (111) line intensity decreases at $\varepsilon = 0.96$ and increases at $\varepsilon = 1.62$. In this case, the (220) line was not revealed as well (Fig. 10, *d*). The ratio J_{200}/J_{111} increases to 0.29 for $\varepsilon = 0.96$, and it was not considerably changed (0.16) for $\varepsilon = 1.62$, remaining abnormally low in comparison to the value for reference and indicating the texture in the alloy.

Thus, the obtained data show that the HP treatment of Invar Fe–35.0% Ni alloy changes its structure, however the degree of impact is determined by its prehistory either annealing at high temperature or plastic deformation by HE. In particular, unlike the results of [24], the treatment of the Invar Fe–35.0% Ni alloy by HP with 2 GPa does not completely remove texture formed by HE, giving rise an appearance of twin formations in both cases. The observed difference is apparently caused by a significantly higher level of pressure in [24] (up to 12



a b

c d

Fig. 10. The diffraction patterns of the Fe–35.0% Ni alloy after annealing (1100°C) (*a*) and subsequent processing by HP (*b*), after HE with $\varepsilon = 0.96$ (*c*) and subsequent HP treatment ($\varepsilon = 0.96$) (*d*).

GPa), which probably leads to dissociation of high-angle boundaries of the corresponding elements of texture.

Non-monotonic behaviour of the lattice parameter *vs.* the degree of deformation (ε) by HE [20] as well as the recovery stage induced by the HP treatment accompanying with the dramatic increase of the lattice parameter a (Fig. 8) point to a possible phasic nature of the change of material volume, which is consistent with the concepts of [25]. Considering the situation in the context of present of stress concentrators, which always present in metallic materials [26], we divide them on two types upon the contribution to the volume changing. The subtraction concentrators are conventionally related to the first one, the number of which is denoted as K_T^-. Their presence is accompanied by a decrease of the real volume of a sample material. As a rule, they are voids of various shapes and origin, the vacancies or their clusters. We refer to the second type the interstitial stress concentrators (K_T^+) associated with the particles of different origins the phases as well as the atoms of one sort or another one in the crystal lattice of the alloy.

The linear character of volume change of the NaCl and Sb single crystals at the HP treatment within the pressure range up to the 9 GPa was established in [27] experimentally and by calculation. Since the type and the number of concentrators as well as their behaviour in the elevating pressure conditions may be considerably different, one can expect the features on the pressure dependence of ΔV and the rate of the pressure change, which should be exhibited as the step changes with varying extension and slope of portions not only in mono- but in polycrystalline states.

The non-monotonic change in volume ΔV_P of a sample depending on the pressure P and its derivative dP/dt, the elastic change of the lattice parameter da/dP, the defects density at the stress concentrator top ρ can occur in polycrystalline materials at overlapping of several processes in the condition of the elevating HP pressure. This can be written in functional expression:

$$\Delta V_P = f\left(K_T, K_C, \rho, P, \frac{dP}{dt}, \frac{da}{dP}\right), \tag{1}$$

where $K_T = K_T^- + K_T^+$ и $K_C = K_C^- + K_C^+$ are the numbers of the technological and the structural subtraction and interstitial stress concentrators, caused by the presence of boundaries. The latter ones play the role of stress concentrators in polycrystals [17–19] and lead to the fragmentation of a macroscopic stress field in a sample into components, the growth and relaxation of which, as well as the change of their sign at all levels of the structural hierarchy can provide a non-monotonic behaviour of the volume.

Thus, the results of this work and their discussion as well as the available literature data allow us to assume that the behaviour of the sample volume of the Invar Fe–35.0% Ni alloy in the polycrystalline state on the pressure should be phasic one. In this case, the extension and the slope of each stage will be determined by the amount and the patterns of behaviour at the recombination of both existing and arising defects in conditions of changing magnitude and sign of the stresses level in the top of existing and arising stress concentrators, depending on their type and behaviour at HP. The HP, as it increases, should cause a transition from subtraction concentrators, which are healed in a sample, to the interstitial stress concentrators with the increasing their number. This should lead to an increase in tensile residual stresses after the pressure removal, which is observed on the strain dependence of the lattice parameter of the Fe–Ni austenite (Fig. 8). The reasons for the considered possible volume change of the Invar Fe–Ni alloy may be the subject of further studies.

4. CONCLUSIONS

1. The HP (2 GPa) treatment of the quenched polycrystalline Invar Fe–35.0% Ni alloy results in the relaxation of the elastic field stresses by appearance of twins of deformation and accumulation of the plastic deformation carriers on grain boundaries and pre-boundary zones.
2. The effect of HP is accompanied by the increase of fraction of large grains and the formation of the relief on a sample cross section surface resulting from reorientation of available grains by facilitation of their boundary slipping. It is the result of distinctive changes of grain boundaries states and the pre-boundary zones via their saturation by plastic deformation carriers, including dislocations of high density (approximately $5\cdot10^9$ cm^{-2}) and the twins of deformation.
3. The HP (2 GPa) treatment of the previously HE treated samples of the Invar Fe–35.0% Ni alloy with the degree of deformation $\varepsilon = 0.96$ and 1.62 is not accompanied by notable differences in the organization of the structures hierarchy with the exception of decrease of texture induced by HE and of increase of twins density that results from partial relaxation of the elastic stresses.
4. The HP treatment of the HE treated alloy slightly decreases microhardness resulting from recovery process, however remaining it on the relatively high level as compared to the quenched state.

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