ELASTICITY MODULES AND ACOUSTIC PROPERTIES OF METAL CERAMICS ON THE BASIS OF TUNGSTEN MONOCARBIDE

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The results of measurements of acoustic wave speeds and elasticity modules of metal ceramic alloys on the basis of tungsten monocarbide in temperature range of 100...295 K as well as amplitude dependences of internal friction at temperatures 140...295 K and after γ-irradiation (^{60}Co, 1,25 MeV, $5 \cdot 10^3$ and 10^5 Gy) have been given. It was shown that elasticity modules of alloys decrease monotonously at increase of cobalt concentration. Radiation influence stimulates the processes complicating movement of grain boundary dislocations in alloys. Representation of these alloys in the form of isotropic mixes of anisotropic phases describes qualitatively correctly the dependence of elasticity modules on structure. The resulted experimental and calculated values of elasticity modules may be used for estimation of elastic and strength properties of these alloys.

Hard alloys on the basis of tungsten monocarbide are used for producing metal-cutting tool. Alloys are obtained by powder metallurgy method. They are composites by their structure. Alloy cutting properties are determined by WC. Co supports high plasticity. Alloys possess branch interface boundaries for which high dislocation density is typical. These boundaries serve as a drain for point defects. Dislocation interaction with point defects appears through amplitude-dependent internal friction occurring at acoustic vibration excitation in material [1–4].

Experimental data by Young modules and hard alloy shear are known for temperatures higher than 295 K. Data by coefficients of elasticity and internal friction in alloys on the basis of tungsten monocarbide at temperatures lower than 295 K which could be obtained by other authors wee not revealed [1–5]. In [6] it was determined that γ-radiation of hard alloys results in structural change which may appear at initial stages of plastic deformation [1]. Amplitude dependences of internal friction may be used for ascertaining this. They allow observing effects which could not be practically noticed at static single-axis material loading and, besides, internal friction being nondestructive inspection method allows studying repeatedly one and the same sample. Internal friction is efficient at investigation of temperature dependences as well as at estimation of changes occurring in materials at various actions [1–4].

Alloy chemical composition (in wt. %): VK 6 (94 % WC, 6 % Co), VK 8 (92 % WC, 8 % Co), VK 10 (90 % WC, 10 % Co) [5]. Phase composition: solid solution of tungsten monocarbide WC in cobalt Co. WC grains have different size in the range of 2,5...5,0 mkm, crystal lattice is hexagonal with parameters $a=(2,905...2,907)\cdot10^{-10}$ m, $c=(2,837...2,838)\cdot10^{-10}$ m, cobalt bundle has cubic face-centered structure with constant lattice $a=(3,562...3,565)\cdot10^{-10}$ m [1]. Excitation of mechanical oscillations generates periodic deformations in materials. Decay of oscillations (internal friction) depends on their frequency and amplitude as well as material structure. Internal friction is accepted to be measured in the wide range of frequencies ($1...10^7$ Hz) and amplitudes of deformation ($10^{-7}...10^{-3}$). There are various techniques for this purpose as it is impossible to cover the whole range with

the only one. The applied technique of compound piezoelectric vibrator allows measuring in frequency range $\sim10^5$ Hz at amplitudes of periodic deformation $10^{-6}...10^{-4}$. These deformations cause dislocation motion in crystalline bodies resulting in ultrasonic decay. Contribution of various sources into internal friction is determined by frequency of excited oscillations in a sample.

Other mechanisms except dislocation ones such as thermoelasticity and scattering at point centers appear in the range of frequencies $f>1$ MHz. They may be ignored at lower frequencies in the used frequency range $\sim10^5$ Hz [1–3]. Dislocations do not reproduce at excited periodic loading in the sample as mechanical stress is lower than yield point. Measurement of ultrasonic propagation velocity simultaneously with internal friction allows determining coefficients of elasticity of studied materials. Occurrence of dislocation inelasticity in frequency range $\sim10^5$ Hz is estimated in terms of various modifications of string model of internal friction when dislocation is considered in the form of string fixed in centers of inhibition [1–4].

Amplitude dependence occurs at increase of periodic deformation beginning with some amplitudes called critical ones. Ultrasonic decay is conditioned at this stage by overcoming by mobile dislocations of total resistance created by friction force, elastic tension of dislocation string, detachment of mobile dislocations from point centers of inhibition, break through dislocation forest. Dislocation segments distribution over the length and structure of fixing centers may be judged by the character of experimental amplitude dependences [1, 2]. Amplitude dependence slope characterizes fixing center density and critical amplitude – fixing strength. It allows estimating various factor influence on their structure and plastic properties without destroying materials.

Measurement of coefficients of elasticity by ultrasonic techniques is the inspection method of material mechanical properties. Estimation of strength properties on their basis is possible only on the basis of correlations determined experimentally. Calculation of coefficients of elasticity of practically important materials by the existing theoretical models and comparison of the obtained calculated values with those found experimentally is the required stage.

The aim of the work consisted in determining dependences of coefficients of elasticity of powder metallurgical alloys on composition, experimental check of possibilities of averaging method by Voigt, Royce and Hill for estimating alloy elastic response, experimental estimation of temperature and γ-radiation influence on dislocation inelasticity of materials.

Objects and methods of investigation

Acoustic and elastic properties of alloys VK 6, VK 8, VK 10 were studied. Velocity of propagation and decay of ultrasonic waves were measured. Young and shear modules were calculated by the obtained values of velocities, the results were compared with calculations obtained from alloys examination in the form of isotropic mixture of anisotropic components. Velocities of propagation and decay (internal friction) of elastic waves were determined by resonance method using two-part piezoelectric vibrator [1–3]. Resonance frequencies of rod piezoquartz converters for longitudinal vibrations (X-section) and torsional ones (Y-section) were respectively equal to 100 and 75 kHz. The method allows measuring not only ultrasonic propagation velocity in samples but its decay (internal friction) as well in wide range of strain amplitudes and temperatures. The used samples have the form of rods in which acoustic vibrations excite on their own frequency. High sensitivity of this method is conditioned by the fact that electric quantities are recorded at measurement.

Temperature stability of piezoquartz converter allows measuring at different temperatures. Adjusting resonance in two-part vibrator the proper frequency of a sample is determined from the ratio $f_0=f+(m_{\text{к}}/m_0)\cdot(f-f_{\text{к}})$. Here f, $f_{\text{к}}$, are the proper frequencies of compound vibrator and piezoquartz, $m_{\text{к}}$, m_0 are the masses of quartz and sample respectively. Acoustic wave length $\lambda=C_l f_0$ and $l_0=n\cdot\lambda/2$ (n is a number of half-waves placed on sample length l_0) therefore elastic wave velocity in a sample is determined as $C_l=l_0 f_0/n$. In a half-wave vibrator $n=1$. Velocity C_l of elastic longitudinal waves in a thin rod is connected with Young module E by the ratio $C_l=(E/\rho)^{1/2}$, velocity of transverse waves is connected with a similar ratio $C_t=(G/\rho)^{1/2}$ with shear module G, where ρ is the density of the sample. Measurements by this technique allow determining velocities of elastic wave propagation with relative error not exceeding 0,5 %. Studying internal friction a logarithmic decrement of vibration decay δ_n in a compound vibrator being a measure of internal friction is found from the ratio $\delta_n=(2hd_{11}/S_{22})\cdot(R_n/fm_n)$. Here h is the thickness of piezoelectric vibrator, m_n is the total mass of all its elements, f, R_n is the resonance frequency and electric resistance of a compound vibrator which are found at measurements; $d_{11}=1,279\cdot10^{-11}$ m²/N is piezoelectric module of quartz, $S_{22}=2,31\cdot10^{-12}$ C/N is its constant of elastic compliance. Relative amplitude of periodic sample deformation (ratio of longitudinal vibration amplitude to sample length) is found from the expression $\varepsilon=[S_{22}/(2hd_{11}l_0 f)]\cdot I_n$, where I_n is the intensity of current flowing through vibrator.

Measuring temperature and amplitude dependences of internal friction and elasticity modules the compound vibrator is placed in a measuring cell [3]. Liquid nitrogen is used for cooling. Temperature may be controlled by a heating element; copper-constant thermocouple is used for measuring temperature. Samples for measuring are made in the form of rectangular rods with section 2,5×2,5 mm², length is chosen so that a half of length of excited acoustic wave may be placed there. Values of density $\rho(T)$ of studied materials required for calculating coefficients of elasticity are found from the ratio

$$\rho(T) = \frac{\rho(295)\cdot[1+3\alpha\cdot295]}{[1+3\alpha\cdot T]},$$

where $\rho(T)$ and $\rho(295)$ are the density at temperature T and 295 K, α is the temperature coefficient of linear expansion (TCLE). Sample density at temperature 295 K was determined by hydrostatic weighing, temperature coefficient α was measured by the technique [7], Table 1.

Table 1. *Density and temperature coefficient of linear expansion of alloys on the basis of tungsten monocarbide at 295 K*

Alloy	ρ, 10³ kg/m³	α, 10⁻⁶ K⁻¹
VK 6	14,87	3,61
VK 8	14,70	4,20
VK 10	14,45	4,82

Results and discussion

Experimental temperature dependences of propagation velocities of elastic longitudinal vibrations and internal friction in thin rods of alloys on the basis of tungsten monocarbide are given in Fig. 1.
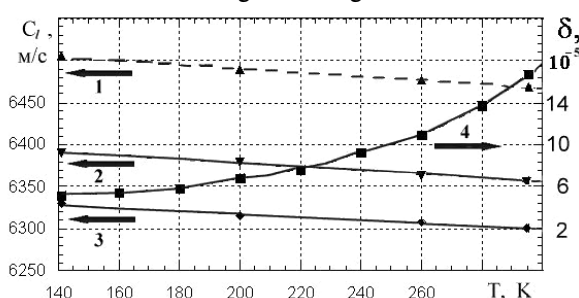

Fig. 1. *Temperature dependences of propagation velocities of longitudinal acoustic waves in alloys: 1) VK 6, 2) VK 8, 3) VK 10 and internal friction 4) in alloy VK 8*

It follows from the given results that at increase of cobalt content in alloy the ultrasonic velocity decreases and therefore Young module decreases. It is known that it is typical also for mechanical characteristics: ultimate stress at compression, bending and extension [1]. In alloys tungsten monocarbide grains are connected by cobalt phase and at mechanical loads plastic flow starts just in it. Processes preceding its beginning may be connected with occurrence of amplitude dependence of internal friction in material. Internal friction measured simultaneously with measurement of ultrasonic velocity

decreases at temperature lowering that coincides well with supposition about its dislocation character [1, 2]. The same conclusion follows from observations of amplitude dependences of internal friction at different temperatures. The results of calculations of coefficient of elasticity of studied sintered powder materials by the data on their components are given as well in the paper. Modules of polyphase polycrystalline solids were estimated by elastic parameters of monocrystal. Values obtained for 295 K are compared with experimentally determined values.

The widespread method of averaging coefficients of elasticity and elastic compliance constants by Voigt, Royce and Hill was used for calculations [1, 8]. Alloys were considered as two-phase compounds. Each phase has its crystal structure. Average values of alloy coefficients of elasticity were found on the basis of isotropic mixture model of anisotropic phases. Representation about phase chaotic propagation by orientations and random uniform distribution by their volume is in its basis. Calculations suppose averaging by orientations and determination of average modules of uniform compression $<B_i>$ and shear $<G_i>$ of separate i phases. To find hard alloys coefficients of elasticity average values of coefficient of elasticity of each phase are determined previously. Taking into account chaotic phase interlacing by volume compression $<B>$ and shear $<G>$ modulus of aggregate of n isotropic phases are found for this purpose ratios are used [1, 8]:

$$< B >_\phi = \sum_{i=1}^n c_i \cdot < B_i >_\phi ; < G >_\phi = \sum_{i=1}^n c_i \cdot < G_i >_\phi ;$$

$$< B^{-1} >_p = \sum_{i=1}^n c_i \cdot < B_i^{-1} >_p ; < G^{-1} >_p = \sum_{i=1}^n c_i \cdot < G_i^{-1} >_p .$$

Here c_i is the volume concentration of i phase. Voigt averaged by all lattice orientations supposing deformation homogeneity by crystal volume. Royce supposed that voltages are homogeneous. Formulas by which average values of compression modulus $<B>$ and shear modulus $<G>$ of polycrystalls are calculated for tungsten monocarbide have the form (hexagonal structure) [1]:

$$\left\langle B_\phi \right\rangle = \frac{1}{9} \cdot (2c_{11} + c_{33} + 4c_{13} + 2c_{12});$$

$$\left\langle G_\phi \right\rangle = \frac{1}{30} \cdot (7c_{11} + 2c_{33} - 5c_{12} - 4c_{13} + 12c_{44});$$

$$\left\langle \frac{1}{B_p} \right\rangle = (2S_{11} + S_{33} + 2S_{12} + 4S_{13});$$

$$\left\langle \frac{1}{G_p} \right\rangle = \frac{2}{15} \cdot (7S_{11} + 2S_{33} + 3S_{44} - 5S_{12} - 4S_{13});$$

for cobalt (cubic structure):

$$\left\langle B_\phi \right\rangle = \frac{1}{3} \cdot (c_{11} + 2c_{12}); \left\langle G_\phi \right\rangle = \frac{1}{5} \cdot (c_{11} - c_{12} + 3c_{44});$$

$$\left\langle \frac{1}{B_p} \right\rangle = 3 \cdot (S_{11} + 2S_{12}); \left\langle \frac{1}{G_p} \right\rangle = \frac{1}{5}[4(S_{11} - S_{12}) + S_{44}].$$

Here indices «ϕ» and «p» notice averaging by Voigt and Royce respectively. Values of coefficients of elasticity required for calculations are taken from reference book [9]. Constants of elastic compliance are calculated by transition formulas [1].

Hill showed that Voigt approximation results in conservative values of coefficients of elasticity and Royce approximation results in conservative values. Averaging method of coefficients of elasticity by Hill is based as well on idea about chaotic distribution of crystal grains on polycrystal volume. Average coefficients of elasticity $<B>_x$ and $<G>_x$ (index «x» means Hill averaging) are calculated as arithmetic or geometric mean values of proper coefficients of elasticity obtained by the methods of Voigt and Royce. We calculated by arithmetic mean value. The obtained shear and compression modulus allow calculating values of Young module E and Poisson coefficient v characterizing ratio of lateral contraction to longitudinal sample extension at its single-axis expansion. For isotropic solid elastic responses are connected by ratios [1]

$$E = \frac{3B \cdot G}{3B + G}; \ B = \frac{G \cdot E}{9G - 3E}; \ v = \frac{3B - 2G}{6B + 2G} = \frac{E}{2G} - 1.$$

The obtained experimental and calculated values are given in Table 2.

Table 2. *Elastic responses of hard alloys on the basis of tungsten monocarbide in temperature range 100...295 K*

Alloy	T, K	E, GPa	G, GPa	B, GPa	v
VK 6	100	632,5	262,7	356,0	0,204
	150	629,0	261,1	356,5	0,205
	200	627,4	259,9	356,9	0,207
	250	625,4	259,2	357,4	0,209
	295	622,5	257,2	357,9	0,210
	295*	641,4	258,1	415,2	0,242
VK 8	100	603,8	252,3	331,7	0,197
	150	601,4	251,0	332,2	0,198
	200	599,0	249,6	332,6	0,200
	250	596,5	248,3	332,7	0,201
	295	594,0	247,0	332,8	0,202
	295*	625,0	251,1	229,1	0,245
VK 10	100	581,7	242,4	323,0	0,200
	150	579,6	441,2	323,4	0,201
	200	577,4	240,2	322,8	0,202
	250	575,5	239,0	323,0	0,203
	295	573,5	238,1	123,3	0,205
	295*	609,5	244,5	400,6	0,246

* – calculation

It follows from the Table that at increase of cobalt content in alloy and temperature rise the coefficients of elasticity decrease steadily that indicates the absence of polymorphic transformations in studied materials. The values of Poisson coefficients depend slightly on temperature and ceramics composition they are in the range of 0,20...0,21. Values of Young modules and ceramic metal shifts exceed coefficients of elasticity of tool steel in в 2...5 times. Changes of coefficients of elasticity occur with temperature practically by linear law. It allows estimating the values of coefficients of elasticity at different

temperatures by linear extrapolation. Comparison of experimental and computed values of coefficients of elasticity shows that the model of ceramics as isotropic mixture of anisotropic phases describes qualitatively correctly their change depending on composition. Divergence between the calculation results and experimental data are in the range of 10 %. Probably, the reason is in the fact that the model does not take into account inhomogeneity of deformation and stress on material volume, besides, crystal grain shape is idealized; it is supposed to be spherical. The obtained results indicate acceptability of examining multiphase cermets in the form of isotropic mixtures of anisotropic phases. Such approach allows estimating technically important elastic features of materials and gives an opportunity of their forecasting by known elastic parameters of components and their concentrations.

The damping of elastic wales in solid is accompanied by their damping, wich is characterized by a logarithmic decrement δ. Dependences of δ on relative amplitude of periodic deformation ε of a sample of alloy VK 8 were studied, Fig. 2.
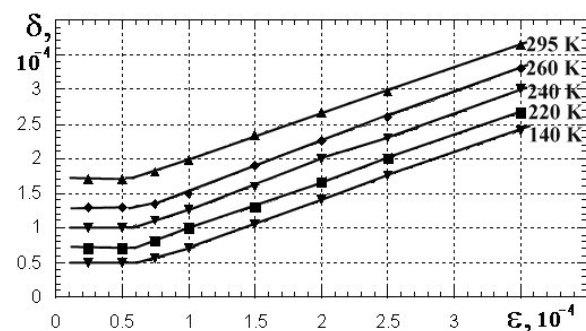
Fig. 2. *Amplitude dependences of internal friction in alloy VK 8 in temperature range 140...295 K*

Two ranges are detected: amplitude-independent δ_i and amplitude-dependent $\delta_h(\varepsilon)$. Level of internal friction lowers in the whole range of periodical strain amplitude at temperature decrease. Threshold amplitude of deformation ε_i corresponding to beginning of amplitude dependence $\delta_h(\varepsilon)$ does not practically depend on temperature. Critical stress $\sigma_i = E \cdot \varepsilon_i$ corresponding to critical amplitude is about two orders lower than yield point for this alloy. Occurrence of dependence $\delta_h(\varepsilon)$ in deformation region $\varepsilon \geq \varepsilon_i$ may be considered as initial stage of plastic deformation occurring in separate microvolumes of a sample. In the sample where standing elastic wave excites the linear dependence $\varepsilon \geq \varepsilon_i$ observed in amplitude region $\sigma_h = f(\varepsilon - \varepsilon_i)$ may be connected with microdeformations at interfaces owing to significant inhomogeneity of voltages.

Reversibility of amplitude dependences show that no structural changes occur in alloy under the influence of periodical deformation. The observed deformations occur in stress range «proportionality limit – elastic limit». Threshold stress corresponding to the beginning of amplitude dependence should be evidently considered as proportionality limit which can not be practically determined at experimental diagrams «stress – deformations». Amplitude dependence inclination does not depend on temperature therefore the conclusion may be made from the position of internal friction string model [1, 2, 4] on constancy of mobile dislocation density and their centers of inhibition in the studied temperature range and amplitudes of periodical deformation.

Influence of γ-irradiation (^{60}Co) by portions $D=5 \cdot 10^3$ and 10^5 Gy on alloy VK 8 resulted in decrease of ultrasonic waves decay in it. Amplitude dependences of decay of longitudinal acoustic waves (internal friction) at 295 K in the sample of alloy VK 8 before and after irradiation by portions $5 \cdot 10^3$ and 10^5 Gy are given in Fig. 3.
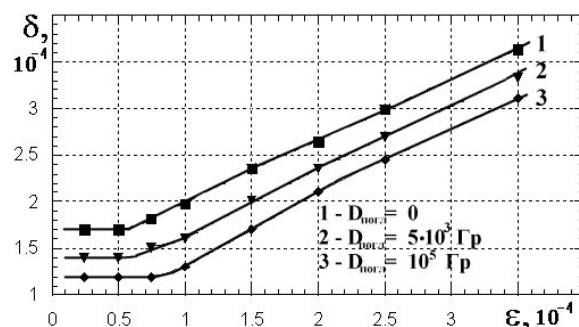
Fig. 3. *Amplitude dependences of internal friction at 295 K in alloy VK 8: 1) unirradiated sample, 2) D=5·10³ Gy, 3) D=10⁵ Gy*

At the absorbed portion $D=5 \cdot 10^3$ Gy internal friction decreased but thresholdamplitude since which there is amplitude dependense is changed unsidnificantly. Its significant increase occurred at the absorbed portion $D=10^5$ Gy. In work [6] it was stated that at portions ~$5 \cdot 10^3$ Gy the reflections of the new phase of alternate composition corresponding to chemical formula Co_xW_yC appear in alloy. It may be supposed that occurrence of new phase reflections impedes dislocation motion on grain boundaries without changing in this case dislocation segment distribution along lengths [1, 2, 4]. Identical inclination of amplitude dependences both in irradiated and in unirradiated samples indicates this. The occurred disperse particles may change alloy strength properties. But crystalline material irradiation stimulates as well the processes of their structure ordering [6].

Existence of short-range order reacts additionally to dislocation motion. Dislocations disturb this order moving in their sliding planes. Area covered by dislocation at motion gets surface energy gain W_r. It is equivalent to increase of stress of resistance to its motion by the value ~W_r/b, where b – Burgers vector [10]. For occurrence of amplitude dependence the value has dislocation motion close to interface. Crystal structure of tungsten monocarbide is partially constructed on covalent bonds which do not support evident motion of dislocations at temperature lower than 295 K. Dependence of internal friction on oscillation amplitude is not observed in the same temperature range in cobalt subjected to thermal annealing. It allows supposing that occurrence of amplitude dependences in hard alloys is caused with motion of grain boundary dislocations. Processes occurring on interfaces at γ-irradiation impede dislocation motion on these boundaries.

Principle conclusions

It was shown that in the studied metaloceramic alloys on the basis of tungsten monocarbide the coefficients of elasticity decrease steadily at increase of cobalt concentration. Alloy model in the form of isotropic mixture of anisotropic phases describes qualitatively correctly dependence of elastic characteristics of metal ceramics on composition. The computed values of Young modulus and shift coincide well with the measured ones. Gamma irradiation by portions to 10^5 Gy decreases the level of internal friction in metaloceramic alloys, increases boundary amplitudes of periodical deformation starting with which ultrasonic decay obtains amplitude-dependence character. The reason of this may be both formation of reflections of new carbide phase under the influence of irradiation and structural changes in alloy impeding motion of grain-boundary dislocations. Such character of internal friction change allows supposing that γ-irradiation changes conditions of plastic flow in materials at initial stages of deformation.

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STRUCTURAL–PHASE STATE AND MECHANICAL PROPERTIES OF SUBMICROCRYSTALLINE TITANIUM ALLOY Ti-6Al-4V OBTAINED WITH USE OF REVERSIBLE HYDROGEN ALLOYING

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Features of evolution of structural-phase state of titanium alloy Ti-6Al-4V at the process of submicrocrystalline structure formation using reversible hydrogen alloying have been investigated by methods of electron microscopic and X-ray diffraction analyses. Influence of hydrogen alloying on mechanical properties at stretching of submicrocrystalline titanium alloy Ti-6Al-4V in temperature interval of 293...1023 K was studied. Possible reasons of increase in ultimate and yield strength and reduction of deformation to destruction of submicrocrystalline alloy Ti-6Al-4V in temperature interval 873...1023 K at hydrogen alloying in quantity 0,08...0,33 mas. % were discussed.

1. Introduction

Formation of submicrocrystalline (SMC) structure (grain size is less than 0,5 mμm) in titanium and its alloys improves significantly their operating characteristics [1]. One of the ways of obtaining SMC structure in titanium alloys is the method combining preliminary hydrogen treatment and hot plastic deformation by pressing [2]. In scientific literature there data according to which use of this method allows obtaining homogeneous SMC structure with grain size $d<0,3$ mμm in titanium alloys [2, 3]. It is known that hydrogen may result in fragility of titanium alloys at maintenance therefore, it is practically fully removed from alloy after hot working by pressing by vacuum annealing at temperatures 873...973 K. However, such annealing may cause the change of phase composition, recrystallization and growth of grains of SMC structure formed in alloy doped with hydrogen at hot pressing [4, 5] and thereby result in decrease of its strength and plastic properties. In this connection it is of interest to study evolution of structural phase state at degassing by annealing and influence of hydrogen residual concentration on mechanical properties of titanium alloys in submicrocrystalline state obtained by use of reversible hydrogen alloying.