Plasticity Characteristic Obtained by Indentation Technique for Crystalline and Noncrystalline Materials in the Wide Temperature Range

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ABSTRACT

The majority of advanced structural and functional materials (ceramics, quasicrystals, metallic glasses, intermetallics, semiconductors, etc.) are brittle at standard mechanical tests. But indentation technique has made it possible to determine the plasticity characteristic δ_H of these materials and to compare these characteristics for different materials.

In the present paper the method for determination of δ_H is described and the value of δ_H for different materials is discussed for a wide temperature range.

1. INTRODUCTION

In paper /1/ the authors introduced a characteristic of plasticity δ_H which can be determined in specific conditions of local loading with the indenter. The index H is used to show that the parameter δ is determined by indentation.

In accordance with these considerations,

$$\delta_H = \frac{\varepsilon_p}{\varepsilon_t} = 1-\frac{\varepsilon_e}{\varepsilon_t}, \qquad (1)$$

where ε_p, ε_e and ε_t are the values of the plastic, elastic and total deformation in the direction of loading, averaged over the area of contact of the indenter with specimen.

Plasticity characteristic δ_H is the dimensionless parameter – the fraction of plastic deformation in the total elastoplastic deformation under the indenter.

The physical meaning of the parameter δ_H is determined by the fact that a part of plastic deformation in the total deformation of the material characterizes the ability of this material to change its shape during deformation, i.e. plasticity in a wide interpretation of this term.

The value δ_H varies from 0 for the absolutely elastic penetration of the indenter to unity for completely plastic deformation. In the practice these limiting cases are not observed and $0 < \delta_H < 1$. The presence of critical value of plasticity was found as $\delta_H \approx 0.9$ which achievement is required for the appearance of plasticity at standard mechanical tests of materials at tension or bending.

In this paper temperature dependence of the plasticity characteristic was investigated and discussed for a number of low-ductile advanced materials with high specific strength (ceramics, intermetallics, metallic glasses, quasicrystals and semiconductors).

The theoretical background for determination of the plasticity characteristic by the indentation technique, accounting the incompressibility of deformation core under the indenter for the calculation of the plastic share of deformation only, has been developed in /2/.

In the developed technique /2/ degree of the plastic deformation ε_P is determined by the equation:

$$\varepsilon_p = -\ln\sqrt{1+\left(\mathrm{ctg}\gamma_1 - \frac{HM}{kE^*}\right)^2}, \quad (2)$$

where γ_1 is the angle between the face and axis of indenter, HM is the Meyer hardness, E^* is the effective modulus:

$$\frac{1}{E^*} = \frac{1-\nu_1^2}{E_1} + \frac{1-\nu_2^2}{E_2}, \quad (3)$$

where ν is Poisson's ratio, E is the Young's modulus, indexes 1 and 2 are related to sample and indenter, respectively. Here k is a constant calculated for indenters of various shape. In the case of pyramidal indenter k = 0.565.

The degree of the elastic deformation is:

$$\varepsilon_e = \frac{HM}{E_1}\left(1-\nu_1-2\nu_1^2\right). \quad (4)$$

After that δ_H is calculated by Eq. 1.

Previously elaborated technique /1/ allows determination of the parameter δ_H upon indentation by Vickers indenter using the equation:

$$\delta_H = 1-14.3\left(1-\nu_1-2\nu_1^2\right)\frac{HV}{E_1}. \quad (5)$$

It was found that taking into account a core compressibility effect on plasticity δ_H is necessary only in the case of superhard materials for which δ_H < 0.3-0.4, for example diamond and BN (Table 1).

Generally, the method developed in /2/, that enables one to take into account compressibility of superhard materials which have a large share of the elastic deformation at indentation, should be preferred.

2. PLASTICITY CHARACTERISTIC AT ROOM TEMPERATURE

Typical values of plasticity characteristic δ_H for different materials are given in Table 1.

It is seen from Table 1 that diamonds have the minimum value of δ_H.

Table 1

Plasticity characteristic δ_H for different materials at room temperature.

Materials		δ_H
Covalent crystals	C, diamond	0.05
	Si	0.37
	Ge	0.46
Ceramics	SiC	0.35
	Al_2O_3	0.43
	TiB_2	0.44
	TiC	0.46
	ZrC	0.52
	NbC	0.56
	ZrN	0.64
	WC	0.82
BCC metals (Fe, Mo, Nb, Cr etc.)		0.92 – 0.97
FCC metals (Cu, Al, Au, Ni)		0.97 – 0.99
HCP metals (Co, Re, Ti etc.)		0.95 – 0.97
Intermetallics	Al_3Ti	0.68
	$Al_{61}Cr_{12}Ti_{27}$	0.81
	$Al_{66}Mn_{11}Ti_{23}$	0.87
MG	ribbons on the base Fe	0.60
	bulk materials on the Zr and Ti base	0.7 – 0.75
Quasicrystals	$Al_{63}Cu_{25}Fe_{12}$	0.69
	$Al_{70}Pd_{20}Mn_{10}$	0.71

For other covalent crystals (silicon and germanium) δ_H is also low. The same is true for SiC and Al_2O_3 where covalent component of interatomic bond is high. Further, with the growth of δ_H in Table 1 borides, carbides and nitrides of transition metals are situated. In carbides of IV-A group metals (TiC, ZrC), as well as in carbide NbC and borides $\delta_H \approx 0.5$, i.e. plastic deformation is about 50 % from total deformation under the indenter. At the same time tungsten carbide has a considerably higher value δ_H = 0.82 which is a great advantage in comparison with other refractory compounds. Note that the high value of δ_H in tungsten carbide is the consequence both of the very high value of Young's modulus E ≈ 700 GPa and the low hardness (in comparison with other carbides). For nitrides the $\delta_H \approx 0.6$, i.e. rather higher than for borides and carbides (excluding tungsten carbide). For

investigated intermetallics on the base of Al_3Ti $\delta_H <$ 0.9 as well. But transition from tetragonal DO_{22} structure with low symmetry for Al_3Ti to the cubic $L1_2$ structure (by alloying Al_3Ti with Cr or Mn) increases δ_H essentially.

For metallic glasses (MG) on an iron base, which are produced by spinning technique as ribbons with thickness 30-50 μm, $\delta_H \approx 0.60$. There is slightly higher plasticity characteristic for bulk MG on the base of Zr and Ti, where $\delta_H = 0.7 - 0.75$.

For the new material, quasicrystals, $\delta_H < 0.9$ at room temperature as well.

For all pure metals $0.9 < \delta_H < 1.0$. The δ_H value is higher for metals with FCC lattice (Ni, Al, Cu, Au) than for metals with BCC lattice (Mo, Cr, Fe, etc.) and HCP lattice (Co, Re, Be, Mg, Ti).

The δ_H values given in Table 1 are consistent with the concept of relative plasticity of materials as determined by the type of interatomic bonds and by measurements at standard mechanical tests. However, the measurement of the δ_H parameter is probably the only way for direct comparison of plasticity for a number of materials, which are usually considered to be brittle, but during indentation elastoplastic deformation without substantial macroscopic fracture takes place in them.

3. TEMPERATURE DEPENDENCE OF PLASTICITY CHARACTERISTIC δ_H

The theoretical basis of the dependence $\delta_H(T)$ has been elaborated in /1/ for covalent crystals and BCC metals with a strong dependence of yield stress σ_s (and hardness) on the temperature.

It was shown /1/ that the dependence $\delta_H(T)$ is different for various temperature ranges. In the curve $\delta_H(T)$ three temperature regions with different deformation mechanisms are distinguished. There are regions of cold, warm and hot deformation. The boundary between temperature intervals of cold and warm deformation is the characteristic deformation temperature **T*** /3/ and the boundary between the regions of warm and hot deformation is the recrystallization temperature T_r.

In the wide temperature range at $0K < T < T^*$ yield stress is described by the following expression /4/:

$$\sigma_s = \sigma_s(0) - \frac{kT}{V}\ln\frac{M}{\dot{\varepsilon}}. \qquad (6)$$

In conformity with Tabor we can assume:

$$HV = C\sigma_s, \qquad (7)$$

where C = const. Here U is the activation energy of dislocation motion, V is the activation volume, M is a material constant, $\dot{\varepsilon}$ is the strain rate, T is temperature, k is the Boltzmann constant and $\sigma_s(0)$ is the flow stress at 0 K.

With the use of Eqs. 5-7 and neglecting the temperature dependence of Young's modulus in comparison with the dependence $\sigma_s(T)$ we obtain

$$\delta_H = \delta_H(0) + \frac{AT}{VE}, \qquad (8)$$

where $A = 14.3\left(1 - \nu_1 - 2\nu_1^2\right)Ck \cdot \ln\frac{M}{\dot{\varepsilon}}$; $\delta_H(0)$ is the plasticity parameter at 0 K. Thus, in the low temperature region δ_H has to increase linearly with the growth of temperature and this is observed in practice.

At higher temperatures, close to the characteristic deformation temperature T* an exponential dependence $\sigma_s(T)$ is observed /3-5/

$$\sigma_s \approx \text{const} \cdot \exp\frac{U}{3kT} \qquad (9)$$

In this case

$$\delta_H = 1 - \frac{14.3\left(1 - \nu_1 - 2\nu_1^2\right)C \cdot \text{const}}{E_1}\exp\frac{U}{3kT}, \qquad (10)$$

i.e. δ_H falls exponentially with decreasing temperature lower than T*, and this exponential curve turns smoothly into a straight line (Eq. 8).

If the temperature $T > T_r$ (hot deformation range) materials have high plasticity and parameter δ_H increases and approaches 1. This characteristic of the temperature dependence δ_H is shown for polycrystalline molybdenum in Fig. 1.

Temperature dependence of plasticity characteristic for investigated ceramic materials is given in Fig. 2. For

these materials T* is very high and practically Fig. 2 gives dependence $\delta_H(T)$ in the temperature range of cold deformation. For majority materials given in Fig. 2 δ_H increases linearly with the growth of temperature. But for some materials (Si_3N_4 and SiC) δ_H does not depend on the temperature at low temperatures. It is the consequence of the existence of an athermal low-temperature sections on the dependence HV(T) for these materials. These athermal sections in covalent crystals and ceramics may be the result of phase transition or intensive cracking during indentation /6,7/. In these case δ_H parameter has another meaning – it characterizes "quasiplasticity" with microcracking or possibility of the phase transformation under high hydrostatic pressure during indentation. It is seen from Fig. 2 that all investigated materials are brittle at standard mechanical tests (δ_H < 0.9) in the wide temperature range. It is possible to determine ductile-brittle transition temperature for every material as the temperature at which δ_H = 0.9.

Fig. 2 makes it possible to compare the plasticity of different ceramics at various temperatures, e.g., it is seen that δ_H for WC is higher than that δ_H for TiC if T < 900 °C. But at T > 900 °C δ_H of TiC is higher. It is interesting that composite materials WC + Co have an δ_H lower than WC single crystal. However, increasing cobalt content in this composite enlarges the δ_H value.

It was found that temperature dependence of δ_H has an identical character for all BCC metals and covalent crystals in the temperature ranges of cold and warm deformation. Different crystals have almost constant and quite high (0.8 – 0.95) δ_H values in the range of warm deformation and even higher δ_H values (up to 0.98) in the range of hot deformation.

At the same time in the range of cold deformation, a sharp decrease in δ_H on temperature decrease is observed, which has a different intensity for different crystals.

An abrupt decrease in δ_H is observed for covalent crystals with high Paerls-Nabarro stress and low activation volume (see Eq. 8), for instance, for Si and Ge having the ductile-brittle temperature T_{db} of 0.7–0.9 T_m. For BCC metals (Mo, W, Cr, Ti, Nb) having significant covalent component of atomic bond T_{db} is 0.1–0.2 T_m. These materials have low plasticity characteristic δ_H only at low temperatures.

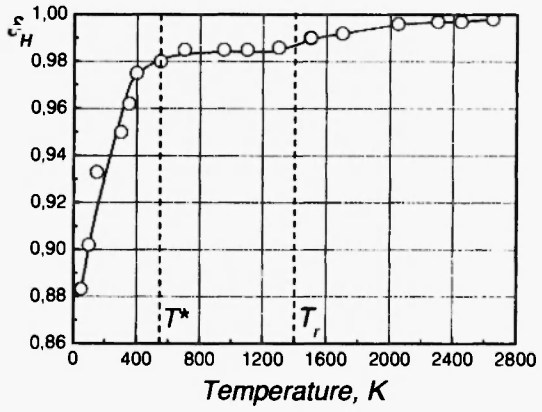


Fig. 1: Temperature dependence of the plasticity parameters δ_H for polycrystalline Mo.

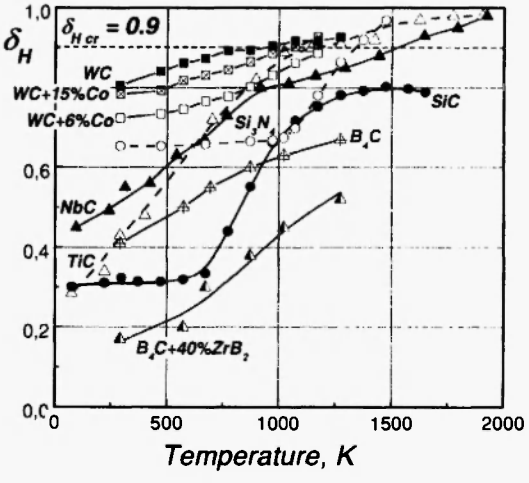


Fig. 2: Temperature dependence of δ_H for various ceramic materials.

For Si in the wide temperature range of 20 – 400 °C δ_H does not depend on the temperature (Fig. 3) that rises from phase transition upon indentation. When temperature exceeds 400 °C, δ_H grows sharply, and at temperatures above 750 °C Si single crystal possesses plasticity upon macroscopic test on bending.

It is necessary to emphasise that for SiC and B_4C in the temperature range of the investigations, δ_H value remains below the critical value of 0.9, confirming the absence of macroscopic plasticity for these materials.

Titanium carbide TiC has low plasticity at room

temperature (δ_H = 0.46), but when temperature grows, the δ_H increases significantly and reaches the critical value of 0.9 at 900 °C.

As mentioned above, δ_H for WC is much higher than for TiC. The values of δ_H for WC and TiC are almost similar only if the temperature is higher than 900 °C. But the working temperature of tools is usually lower than 700°C. These results have shown that WC has a better combination of hardness and plasticity than TiC, and from this point of view anybody can understand the advantage of WC over its rival TiC as the basis of hard alloys.

The temperature dependence of plasticity characteristic for materials with different types of interatomic bonds and different atomic structure is shown in Fig. 3 using a homological scale of temperature.

This generalization allowed comparison of the dependence $\delta_H(T)$ for such different materials like aluminum (δ_H > 0.98 in the all temperature range) and diamond (δ_H changes from 0.048 at room temperature to 0.7 at the temperature close to diamond intensive graphitization temperature) T_{graph} (see Fig. 3).

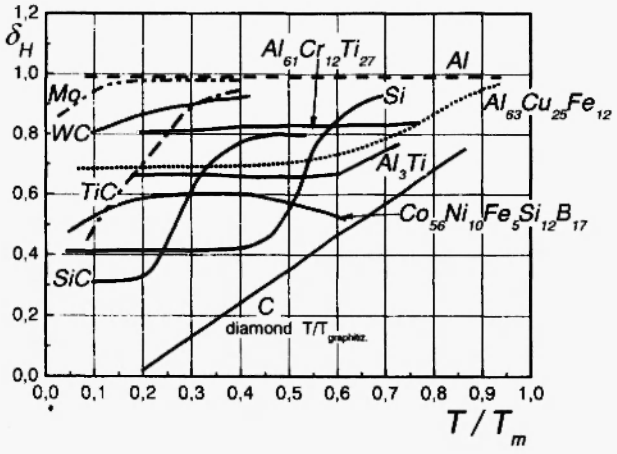


Fig. 3: Temperature dependence of δ_H for materials with different type of interatomic bonds and atomic structure.

In Fig. 3 we used instead of a melting point for carbon in the P-T scheme the temperature of intensive graphitization (2000 K), because it was shown /5/ that the strength characteristics of diamond in a metastable state depend on the temperature as if diamond would possess some effective melting temperature $T_{m\,cf} = T_{graph}$ which, in the calculations of strength temperature dependence, is equivalent to the melting temperature of other crystals which are free from polymorphous transformation.

In Fig. 3 the temperature dependence of plasticity characteristic for new noncrystalline materials – metallic glasses (MG) and quasicrystals – is given as well as $\delta_H(T)$ for some investigated intermetallics.

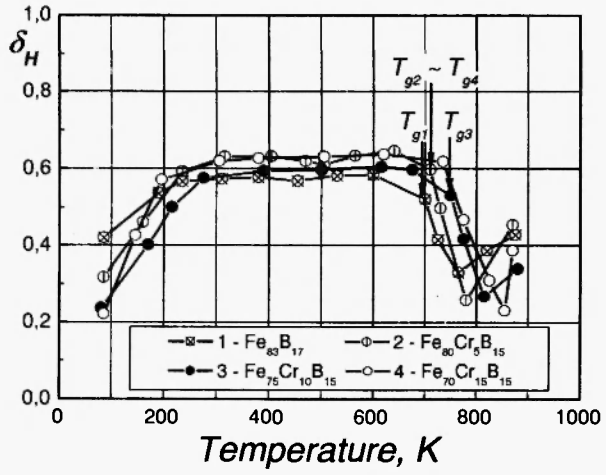
In conjunction with small plasticity of intermetallics it was effective to investigate mechanical properties by indentation method /8/.

This investigation was made using Al_3Ti and its alloys, which have brittle failure or have limited ductility upon standard mechanical tests.

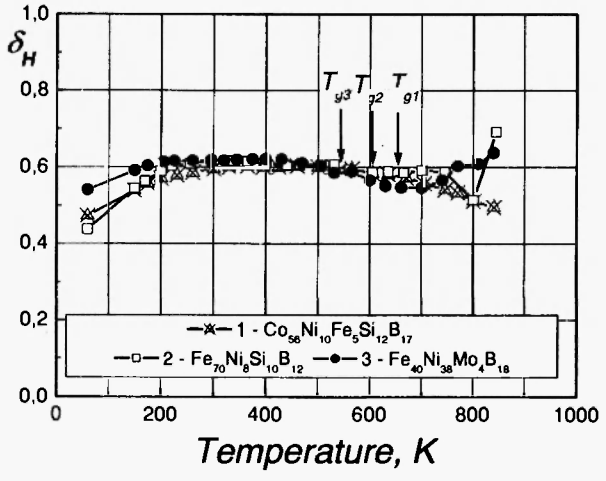
It is seen from Fig. 3 that $\delta_H \simeq 0{,}7$ for intermetallic Al_3Ti, i.e. a bit higher than for a majority of ceramic materials; however, it is lower than for tungsten carbide and hard alloys WC-Co. It is also seen that a transformation of Al_3Ti into $L1_2$-phase (alloys $Al_{61}Cr_{12}Ti_{27}$ and $Al_{66}Mn_{11}Ti_{23}$) brings δ_H to appreciable increase. Moreover, δ_H approaches the critical value δ_H = 0.9, that provides some macroscopic plasticity, but in compression tests only /8/. It is seen that temperature dependence $\delta_H(T)$ for intermetallics appears of an anomalous character – δ_H does not practically increase with a growth of temperature. This character of dependence $\delta_H(T)$ was observed for the first time and this is a consequence of anomalous temperature dependence of hardness and yield stress for these materials.

Employing results on temperature dependence of hardness for different MG /9/, the temperature dependence of the plasticity characteristic δ_H was calculated. Plasticity characteristic δ_H for MG on the iron and cobalt basic is approximately 0.6 in the wide temperature range (from room temperature up to 600°C) (Fig. 4). When temperature decreases below the room temperature, δ_H decreases because of difficulty of plastic deformation in the amorphous state. Significant decreasing of δ_H is also observed at high temperature due to crystallization. It was shown for the first time that plasticity characteristic δ_H for MG is low and comparable with that for ceramic materials. But it is known that brittleness of ceramics in a great extent is connected with intergranular fracture, which is absent in

MG because of the absence of grain boundaries. Bulk MG on the base of Ti and Zr were investigated as well. For these MG δ_H exceeds essentially 0.6. For example, for $Zr_{41}Ti_{14}Cu_{12.5}Ni_{10}Be_{22.5}$ amorphous alloy δ_H is 0.75.



(a)



(b)

Fig. 4: Temperature dependence of plasticity characteristic δ_H for MG:
(a) alloys $Co_{56}Ni_{10}Fe_5Si_{12}B_{17}$, $Fe_{70}Ni_8Si_{10}B_{12}$ and $Fe_{40}Ni_{38}Mo_4B_{18}$
(b) alloys $Fe_{83}B_{17}$, $Fe_{80}Cr_5B_{15}$, $Fe_{75}Cr_{10}B_{15}$ and $Fe_{70}Cr_{15}B_{15}$
T_g are the crystallization temperatures.

For the first time the temperature dependence of hardness of new class of materials – quasicrystal Al-Cu-Fe – was studied /10/. Employing results /10/, the temperature dependence of plasticity characteristic δ_H was calculated (Fig. 3).

It is seen that the plasticity characteristic δ_H for this quasicrystal indeed has a value of $\delta_H \approx 0.7$ in a broad temperature interval i.e. at local loading of quasicrystal only 70 % of deformation is governed by plastic flow. If the temperature goes higher than 300 °C δ_H starts to grow and at 700 °C reaches critical value $\delta_H \sim 0.9$. At the same temperature plasticity appears at mechanical test by tension of these quasicrystals. Thus plasticity characteristic δ_H can be used to describe mechanical behavior and transition from brittle to plastic state for quasicrystals as well.

4. STRUCTURAL SENSITIVITY OF PLASTICITY CHARACTERISTIC δ_H

This problem was investigated in /1/. For description of the dependence of yield stress σ_s on the grain size, we will use the Hall-Petch equation

$$\sigma_s = \sigma_o + K_y d^{-1/2}, \qquad (11)$$

where σ_o is the mean yield stress of a single crystal and K_y is a Hall-Petch coefficient.

Using Eqs. 5, 7 and 11 we obtain:

$$\delta_H = \delta_{Ho} - \frac{14.3\left(1-\nu_1-2\nu_1^2\right)K_y C}{E_1} d^{-1/2}, \qquad (12)$$

where $\delta_{Ho} = 1 - \frac{14.3C\left(1-\nu_1-2\nu_1^2\right)}{E_1}\sigma_o$ is the plasticity characteristic of a single crystal.

The hardening due to increase of dislocation density ρ in metals as well as in covalent crystals is satisfactorily described by the relation

$$\sigma_s = \sigma_o + \alpha G b\sqrt{\rho}, \qquad (13)$$

where σ_o is the flow stress of a crystal without dislocations, α is a coefficient, b is the Burgers vector of dislocation. Using Eqs. 5, 7 and 13 we obtained in /1/

$$\delta_H = \delta_{Ho} - \frac{14.3\left(1-\nu_1-2\nu_1^2\right)\alpha\, G\, b C}{E}\sqrt{\rho} \qquad (14)$$

where

$\delta_{Ho} = 1 - \frac{14.3\left(1-\nu_1-2\nu_1^2\right)C\sigma_o}{E}$ is the plasticity characteristic of a crystal without dislocations.

It is seen that strengthening due to decreasing grain size and increasing dislocation density must increase plasticity characteristic. Some experimental results that confirm Eqs. 12 and 14 are obtained in /1/. But this problem must be investigated more completely, especially for materials with submicron and nano-size grains.

5. CONCLUSIONS

Taking into account the results which were obtained in the present work and in previous investigations of authors it is possible to note the following:

1. Theoretical ground is elaborated for the temperature dependence of plasticity characteristic δ_H in the temperature ranges of cold, warm and hot deformation of BCC metals, covalent crystals and ceramics on their base. Experimental results for BCC metals confirm this theory completely. Ceramic materials, for which characteristic deformation temperature is very high, are working usually only in the temperature range of cold deformation. In this temperature range δ_H decreases monotonically with the temperature decreases. There exist athermal sections of δ_H at low temperatures in some covalent crystals and ceramics, that is a consequence of phase transition or microcracking during indentation. The temperature of ductile-brittle transition for these materials can be estimated as a temperature at which $\delta_H = 0.9$.
2. Pure FCC metals are ductile in the whole temperature range and for these metals $\delta_H > 0.9$ and approaches to 1 at every temperature.
3. For metallic glasses on the base of iron and cobalt δ_H does not exceed 0.6 up to the crystallization temperature. For bulk metallic glasses on the Ti and Zr base δ_H is some higher, $\delta_H = 0.7 - 0.75$ at room temperature, and its behavior at elevated temperatures was not investigated.
4. The dependence $\delta_H(T)$ was for the first time defined for quasicrystal of system Al-Cu-Fe. It was shown that a strong increase of δ_H is observed for this quasicrystal at temperature higher than 300 °C and at temperature ~ 700 °C δ_H achieves a critical value and equals 0.9 that provides a macroscopic plasticity.
5. For intermetallics on the Al_3Ti base there exists a very wide temperature range in which $\delta_H \simeq$ const, that is the consequence of anomalous behavior of yield stress in these materials. δ_H of these intermetallics increases if alloying leads to phase transformation into cubic $L1_2$ phase.

The authors would like to note once more that plasticity characteristic δ_H obtained through hardness measurement is the effective method for characterization of plasticity for different crystalline and noncrystalline materials in the wide temperature range.

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