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DISPERSION HARDENING OF SOME THERMOELECTRIC MATERIALS, (U)
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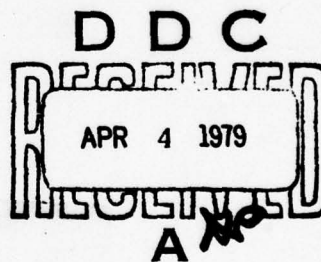
FOREIGN TECHNOLOGY DIVISION



DISPERSION HARDENING OF SOME THERMOELECTRIC
MATERIALS

by

N. S. Lidorenko, O. M. Narva,
et al.



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U. S. BOARD ON GEOGRAPHIC NAMES TRANSLITERATION SYSTEM

Block	Italic	Transliteration	Block	Italic	Transliteration
А а	А а	A, a	Р р	Р р	R, r
Б б	Б б	B, b	С с	С с	S, s
В в	В в	V, v	Т т	Т т	T, t
Г г	Г г	G, g	У у	У у	U, u
Д д	Д д	D, d	Ф ф	Ф ф	F, f
Е е	Е е	Ye, ye; E, e*	Х х	Х х	Kh, kh
Ж ж	Ж ж	Zh, zh	Ц ц	Ц ц	Ts, ts
З з	З з	Z, z	Ч ч	Ч ч	Ch, ch
И и	И и	I, i	Ш ш	Ш ш	Sh, sh
Й й	Й й	Y, y	Щ щ	Щ щ	Shch, shch
К к	К к	K, k	Ъ ъ	Ъ ъ	"
Л л	Л л	L, l	Ы ы	Ы ы	Y, y
М м	М м	M, m	Ь ь	Ь ь	'
Н н	Н н	N, n	Э э	Э э	E, e
О о	О о	O, o	Ю ю	Ю ю	Yu, yu
П п	П п	P, p	Я я	Я я	Ya, ya

*ye initially, after vowels, and after ъ, ы; e elsewhere.
When written as ё in Russian, transliterate as yě or ě.

RUSSIAN AND ENGLISH TRIGONOMETRIC FUNCTIONS

Russian	English	Russian	English	Russian	English
sin	sin	sh	sinh	arc sh	sinh ⁻¹
cos	cos	ch	cosh	arc ch	cosh ⁻¹
tg	tan	th	tanh	arc th	tanh ⁻¹
ctg	cot	cth	coth	arc cth	coth ⁻¹
sec	sec	sch	sech	arc sch	sech ⁻¹
cosec	csc	csch	csch	arc csch	csch ⁻¹

Russian English

rot curl
lg log

1848

Hardening and Shaping

Dispersion Hardening of Some Thermoelectric Materials

Lidorenko N. S., Narva O. M., Mazur V. A., Dudkin L. D., Yerofeyev R. S.

(Moscow)

Experimental data on the dispersion hardening of thermoelectric materials are obtained and compared with existing theories. It is noted that one of the most expedient means of the substantial improvement of strength and plastic properties of thermoelectric materials with allowable reduction of effectiveness is dispersion hardening with inclusions of nonreacting phase. The character and peculiarities of change of the effectiveness of heterophase thermoelectric materials are considered.

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Fundamental positions on the dispersion hardening of thermoelectric materials are presented.

The wide use of thermoelectric converters is slowed to a certain degree by the low mechanical properties of the semiconductor materials used. The role of the strength characteristics of thermoelectric materials is manifested the most clearly on the stage of manufacture of thermoelements and batteries and in the period of operation of converters. One of the obvious reasons for the appearance of mechanical stresses is the difference in the coefficients of thermal expansion (KTR) of the separate layers of the thermoelement. The thermal stresses in this case appear during cooling on the most dangerous in the region of temperatures at which the plasticity of materials is quite low, and the yield point is close to the ultimate strength. The presence of residual stresses, reaching values of the ultimate strength, can lead to substantial weakening of the materials due to the appearance of micro- and macrocracks.

Another important factor, causing "technological" stresses in the thermoelements, can be the reverse elastic aftereffect (OUP) of heterogeneous materials, connected with the action of pressure and

temperature. Depending upon the specific conditions of operation of thermoelectric converters and the mechanical actions connected with them the reliability and the efficiency of the generator will in many respects be determined by the mechanical properties of the thermoelectric materials. The role of the mechanical characteristics is the most clearly visible on an example of hole lead telluride: while possessing quite good thermoelectrical characteristics in conjunction with high heat resistance, it did not find wide practical use (in contrast to n-PbTe) because of low plasticity (~ 0.1 o/o). All this determines the urgency and practical importance of the searches for improvement of the mechanical properties of thermoelectric materials.

1. Selection of the optimum method of the strengthening of thermoelectric materials. Since thermoelectric materials are used mainly in the form of polycrystals, then as applied to them the most expedient method is hardening due to intensification of the defectiveness of the crystal structure [1]. The main practical means of realization of this method can be: strain hardening, quench or vacancy mechanism of hardening, irradiation hardening, hardening by means of the introduction of dispersed crystals of the second phase into the initial material. As applied to thermoelectric materials the last of the methods is the most expedient, effective and technological. This is determined by the fact that precipitation

hardening with the aid of phase stable at elevated temperatures can be easily realized in the general technological cycle of manufacture of thermoelements, the fundamental methods of which are based on the methods of powder metallurgy. Furthermore, this method in principle provides prolonged thermal stability of the effect of hardening both during the manufacture of thermoelectric modules and during their subsequent operation.

In the theoretical works dedicated to the calculation of the change of thermoelectric characteristics (α , σ , κ) of heterophase systems depending on the properties, volume portion and distribution of each of the phases [2, 3] the conclusion is made that the thermoelectric effectiveness of multiphase system can be only lower than Σ of the initial matrix. However, in the indicated works it is clearly or indirectly inferred that the dimensions of particles participating in the formation of heterophase systems are quite great, i.e., each of them preserves volume properties, and that the boundaries between grains of the system are negligibly thin and do not have any effect on the properties of the system. At the same time, with decrease of the size of particles the volume properties should undergo substantial changes in connection with the fact that the surface energy becomes commensurate with volume. In this case, even with use for the creation of a system of particles of one phase, changes of separate characteristics should be observed depending on

the size of particles. Furthermore, with ^usufficiently small dimensions of heterophase particles, when the system is characterized by large area of contacts, the last can have a decisive effect on the properties of material. In this case it cannot be considered that the contacts can have only a negative effect on the properties. A case is also possible where the interfaces of two phases completely or partially reflect the phonons and are "transparent" for electrons (for example, due to "tunnel" effect) [4].

On the other hand, equilibrium of the various phases in the system, which designates alignment of the levels of chemical potentials in the contact region, with the presence of contact differences of potentials is achieved by the transition of some quantity of free carriers from one substance to another. With rather small dimensions of particles this can lead to a quantitative change of the conductivities of heterogeneous particles and, due to this, to a change of the electrophysical properties of the heterophase system as a whole.

Consequently, the account of the phenomena on contacts in dispersed heterophase systems can in principle lead to accomplishment of change of the thermoelectric quality factor to the opposite character than ^afollows from [2, 3]. Recently in a number of works [4, 5] is examined the possibility of some increase of quality factor in

heterophase thermoelectric materials¹.

Footnote: ¹ Dudkin L. D., Lidorenko N. S. and others. Branch of thermoelement. Auth. certificate No. 186539, cl. 21v, 27/03. Bull. No. 19, 3 Oct. 1966.

From that discussed above it follows that the problem of increase of the quality factor with the use of heterophase heterogeneity directly touches the problem of precipitation hardening of thermoelectric materials. The second problem is not specially discussed in this report. Below are provided the generalized results of the experimental investigations on precipitation hardening of thermoelectric materials on the basis of monotellurides of the IV group of elements of periodic system with inclusions of second phase depending on its nature, size of particles and volume quantity.

2. Experimental data on the precipitation hardening of thermoelectric materials. Matrix materials were used in the form of powders with dispersity ~ 0.25 μ m, inclusions in the form of powders with dispersity from 5 μ to (-0.50 ± 0.25) μ m. The content of admixture was basically 5-7 o/o by volume, since with large content the thermoelectric parameters of the materials substantially deteriorate.

In some cases for evaluation of the mechanisms of the effect of inclusions on the properties of the compositions their content was expanded to 50 o/o by volume. Powders, taken in corresponding volumetric quantities, were mixed in drum-type mixers for a twenty-four hour period. From the obtained mixtures by technology, including hot pressing with annealing, cermet specimens were manufactured with cross section $2.5 \times 2.5 \text{ mm}^2$. All the tests at room temperature were conducted on an instrument for micromechanical tests [6] with speed of movement of punch $2.34 \times 10^{-2} \text{ mm/min}$. The height of the specimens, tested for compression, was 7 mm, the distance between supports during bending - 6.5 mm. The obtained data are presented on Figs. 1-4.

The conducted investigations showed that the introduction of iron into n-type lead telluride leads to increase of both strength (Fig. 1) and plasticity of this material. This growth is more considerable, the less iron powder that is used. The difference in hardening is especially noticable with increase of its content to 5 o/o by volume. These phenomena are easily explained during an account of the strengthening action of intergranular boundaries and the difference in the sizes of the basic and the admixture grains.

Compression tests showed that increase of the strength of the samples is accompanied by a rise of their plasticity. With 10 o/o

content of iron with dispersity -0.05 mm in n-type lead telluride the ultimate strengths of the last on compression, shear and bending are increased by 50-60 o/o, the magnitude of plastic deformation under compression to failure is raised approximately 2.2 times.

The character of change of the strength parameters of n-type lead telluride, and also tin and germanium telluride with the introduction of tungsten and quartz into them (Figs. 2, 3) substantially differs from such for lead telluride with iron. While in the last case there is observed only the growth of strength parameters in the range of iron contents up to 25 o/o by volume and with its dispersity up to $(-0.50 + 0.25) \text{ mm}$, compositions with SiO_2 and W along with hardening also exhibit softening, which is more considerable, the coarser the particles of inclusions and the higher their content.

The continuous growth of the strength of these compositions takes place only with dispersity of inclusions -0.05 mm . In this case the effect of hardening with quartz and tungsten content 5 o/o by volume reaches ~ 30 and ~ 25 o/o respectively.

With the introduction of tungsten with dispersity -0.1 mm into tin and lead tellurides (Fig. 3) there is observed growth of strength with rise of the tungsten content up to 19 and 22.5 o/o respectively.

In this case on tin telluride there is achieved approximately an 18 o/o effect, and on lead telluride - approximately 30 o/o. The difference in hardening is probably connected with the initial dispersity of the matrix powders, which is finer in the case of tin telluride. The observed effects of softening with the introduction of tungsten and quartz can be explained by the fact that both these materials have considerably smaller KTR than matrix materials: KTR of lead, tin and germanium tellurides in the temperature range 20-350° C (350° C - the temperature above which a sharp increase of plasticity of these materials is observed) is, according to the authors, respectively 21.0×10^{-6} , 22.0×10^{-6} and 15.0×10^{-6} 1/°C, and the KTR of tungsten and quartz is all of 4.5×10^{-6} [7] and 0.55×10^{-6} 1/°C respectively.

As a result during cooling in the process of the technological cycle around the inclusions are formed negative (with respect to external) stresses. These stresses are higher, the greater the difference in the KTR of the matrix and inclusions and the larger the size of inclusions. If the "coefficient" stresses around the inclusions or their accumulations, appearing due to unavoidable heterogeneity in their distribution, exceed the ultimate strength of the matrix, then crack formation, weakening the material, takes place.

For germanium telluride, the KTR of which is the closest to the KTR of tungsten and quartz, in accordance with that discussed above hardening is observed in those cases when for lead and tin tellurides it is already absent. In all cases with the absence of crack formation the introduction of quartz, the KTR of which is lower than the KTR of tungsten, causes much hardening. Crack formation appears earlier (with smaller contents) with the introduction of quartz and softening, caused by it, is more considerable. Similar phenomena take place, obviously, in compositions on a base of p-type lead telluride with Al_2O_3 , ZrN , TiC (Fig. 4), but to a different degree depending on the values of their KTR, which are respectively 6.8×10^{-6} [8], 8.1×10^{-6} [8] and $6.5 \times 10^{-6} \text{ } 1^\circ \text{C}$ [8]. It should be noted that compressive strength rises with the introduction of these generally softening admixtures. This is connected with insufficient rigidity, and thus the sensitivity of the given method of tests. The distribution of stresses under compression is such that it does not contribute to the opening of cracks, as with tension or bending; if the available cracks nevertheless weaken the material, then this is compensated or even covered in those places where the crack formation is absent. Of course, it is necessary to be oriented to the results of bending tests. Positive effects are achieved with the use of inclusions Y_2O_3 and TiC , covered with cobalt. With the introduction of Y_2O_3 in the quantity of 3 o/o by volume a 20 o/o hardening is achieved, connected apparently with interparticle linkage in

connection with the smearing properties of powder. With further increase of addition the strength practically does not rise. The cause of this is the formation of accumulations of inclusions as a result of heterogeneity in their distribution and, as a consequence, crack formation around these accumulations, which is very probable, since the KTR of Y_2O_3 is all of $7.5 \times 10^{-6} \text{ } 1/^{\circ} \text{C}$ [9].

With the introduction of titanium carbide, covered with cobalt, into p-type lead telluride the greatest hardening effect is achieved: with 5 o/o content of addition the bending strength is increased by 50 o/o, and compressive strength - by 90 o/o. So substantial hardening was caused by the effect of thin interlayers of cobalt, separating the particles of the matrix and inclusions, and also by the fact that its KTR has an intermediate value between the KTR of titanium carbide and lead telluride. It is very important that the thermoelectric effectiveness of such composition practically negligibly differs from the effectiveness of the initial material. The achieved values of bending 4.9 kg/mm^2 and compressive strength 13.6 kg/mm^2 make this material in technological and constructional respects close to alloys on germanium telluride base applied at present as material of the positive branch.

The obtained experimental results on the measurement of thermoelectric properties (α , σ , κ) at room temperature of composite

materials depending on the percent by volume and the dispersity of second phase cannot be sufficiently fully explained only by the theoretical dependences, examined in [2, 3]. In those cases when a large difference does not exist in the KTR of the matrix and dispersed phase and the moduli of their elasticity under technological conditions differ insignificantly, close binding of particles is established. An aggregate structure is formed, which is naturally better than the state of interparticle contacts, which is essentially reflected on the conductivity of two-phase mixtures. For example, for system n-PbTe + Fe the change of conductivities is close to theoretical. The regression from theoretical dependences of specific electrical and thermal conductivities toward their decrease for system GeTe + Fe is caused by the interaction of germanium telluride with iron [10].

As a result of the different thermal expansion of phases, the appearing internal stresses lead to crack formation, the absence of interparticle contacts and, consequently, to decrease of conductivities. This is observed for systems n-PbTe + SiO₂, W, TiC, MoSi₂ and GeTe + SiO₂, W, ZrO₂ to a greater or lesser degree depending on the difference in KTR. Other possible causes of the lowering of conductivities of the investigated compositions can be additional scattering due to increase of the density of dislocations on the boundary of the matrix and dispersed phase and the appearance

of fastening layers in connection with the difference in the work of the output of electrons. The introduction of metal inclusions leads to a drop of the thermoelectromotive force due to the shorting action, especially noticeable in the case $n\text{-PbTe} + \text{Fe}$ in connection with the good state of interparticle contacts. The quantitative determination of the effect of the quality of interparticle contacts and contact phenomena on the thermoelectrical properties of two-phase mixtures is made difficult because of the absence of additional information, for example, about the work of the output of electrons of thermoelectric material, the thickness of the clearance between matrices and dispersed phase, the amount of interparticle contact.

Common for these compositions is a decrease of the σ/χ ratio with increase of the content of hardening phase (Fig. 5) in connection with the sharper drop of specific electric conductivity, and this, in turn, leads to lowering of the thermoelectric effectiveness. However, insignificantly reducing the quality factor, but raising the strength of material, it is possible to avoid considerable losses during the manufacture of thermoelements and batteries and to apply more effective materials, for example, $p\text{-PbTe}$.

3. Analysis of experimental data. On the whole the obtained results on dispersion hardening of thermoelectric materials cannot, of course, be explained by the action of any one of the mechanisms of

hardening. To the investigated compositions are peculiar high internal stresses, which according to Mott - Nabarro [11] slow the movement of dislocations. In view of the magnitude of internal stress, they probably often exceed the yield point of the matrix, which leads to the formation of dislocations close to the inclusions. Consequently, the moving dislocations are forced to cut through the "forest" of dislocations according to Ashby [12].

As a result of the successive movement of dislocations, accumulations of them are formed near the particles of inclusions in accordance with the Ansell model [13]. Stresses in the head of accumulation sharply rise until plastic deformation of the inclusion is started, which takes place, obviously, in the case of composition of n-type lead telluride with iron, since its plasticity rises in proportion to increase of the content of addition. In this case, since the dislocations are forced to move through the particle, they are slowed according to Fleischer [14] in connection with the difference in elastic moduli of the materials of the matrix and inclusions. In the case of rigid undeformed particles, which are in the investigated compositions, for example, tungsten and quartz, the growth of stresses probably leads to some plastic flow of matrix material by means of transverse slip according to Hirsch [15].

The interface between the matrix and inclusions should have such

a character that the field of dislocation stresses could be transferred from the lattice of the matrix through the interface to the lattice of the dispersed particle. For the fulfillment of this requirement the matrix should possess the property of "wetting", i.e., to stick to dispersed particles (by means of adhesion) [16]. The role of cobalt in the composition ρ -PbTe and titanium carbide, covered with cobalt, is partially connected with the "wetting" on the phase boundary.

The quantitative determination of the contribution of each of the mechanisms to the overall effect of hardening of the investigated compositions is made difficult because of the absence of necessary additional information, for example dislocation parameters, and the imperfection of theory.

On the basis of the obtained results and known theoretical positions it is possible to note that the main requirements for the hardening phase, which should be considered for the greatest effect of hardening of thermoelectric materials, are determined by the dislocation mechanism of hardening in dispersion-hardened compositions [11-15]. The specific nature of the problem of hardening of thermoelectric materials consists of the fact that the selection of the type, content and the distribution of particles of second phase should be accomplished in such a way that the greatest

hardening effect is achieved with minimum (allowable) lowering of the thermoelectric quality factor. First of all it is necessary to bear in mind that the lowering of the thermoelectric effectiveness will be the least if the dispersed phase will be chemically inert with respect to the entire working range of temperatures. The content of the hardening phase in the composition should not exceed 5-7 o/o by volume, since with large contents a sharp drop of thermoelectric quality factor takes place because of the shorting (isolation), the formation of fastening layers or crack formation.

Increase of the dispersity of inclusions probably leads to such a relationship of phases, when with a certain critical volume percent of inclusions the composition becomes matrix relative to the inclusions. For conformation of this conclusion there was conducted a theoretical calculation, based on the fact that with a given ratio of average dimensions of grains of the main phase (L) and inclusions (l) there is a critical volume content of second phase, at which the system becomes matrix relative to the inclusions. Two schemes were broken down: in the first were examined cubic particles of the matrix, surrounded by cubic particles of inclusions, in the second - spherical particles of matrix, surrounded by spherical particles of inclusions. Calculations in the assumption $l/L \approx 0.1$, which corresponds to the investigated compositions, show that the system becomes matrix relative to the inclusions for the first case with V_2^*

= 0.24 V, and for the second - with $V_2^* = 0.15$ V. The smaller the ratio of dimensions of inclusions and matrix, the smaller the volumetric portion of inclusions that this transition will be observed with. Considering the increase of the probability of heterogeneous distribution of the particles of inclusions with a rise of their volumetric portion with mechanical mixing, it is possible to explain the decrease of thermoelectric effectiveness due to shorting or the isolating action of inclusions.

The above-discussed shows that the structure of the composition plays a substantial value for the hardening and maintaining of the quality factor of thermoelectric materials. The introduction of hardening phase into grain of the matrix raises the strength of the grain itself, prevents intracrystalline failure, the formation of shorting or isolating layers. It is proposed to conduct a detailed examination of this question in further investigations.

Conclusions. 1. One of the most expedient means of the substantial improvement of the strength and plastic properties of thermoelectric materials with allowable lowering of the effectiveness is dispersion hardening by inclusions of nonreacting phase.

2. By the introduction into lead telluride of n-type conductivity of 10 o/o iron by volume with dispersity -0.05 μ m its

strength properties are raised by 50-60 o/o and the plasticity, determined as the value of plastic deformation with compression to failure, - 2.2 times.

3. With the introduction of titanium carbide, covered with cobalt, into p-type lead telluride with dispersity less than 5μ the strength parameters of the composition are raised 1.5-2 times.

4. The character and features of change of the effectiveness of heterophase materials are examined depending on the type, size and percent content of inclusions.

Submitted 16 Aug 1969.

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Fig. 1. Dependences of the ultimate compressive (σ_c), bending (σ_u) and shear (τ_c) strengths of composition n-PbTe + Fe on the percent content of iron of various dispersity. Numbers at the curves indicate the size of iron grain, μ .

Fig. 2. Dependence of the ultimate bending strength of compositions based on n-PbTe and GeTe on the type, dispersity and content of inclusions.

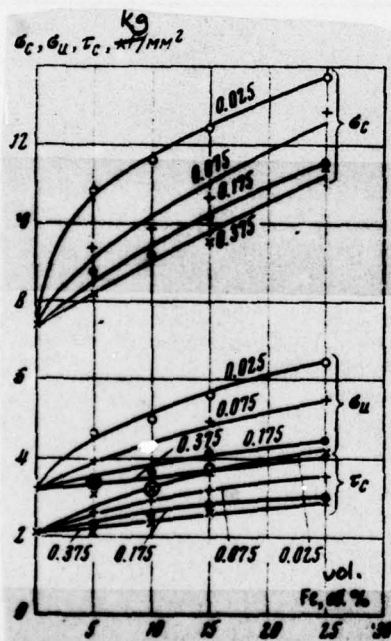


Fig. 1.

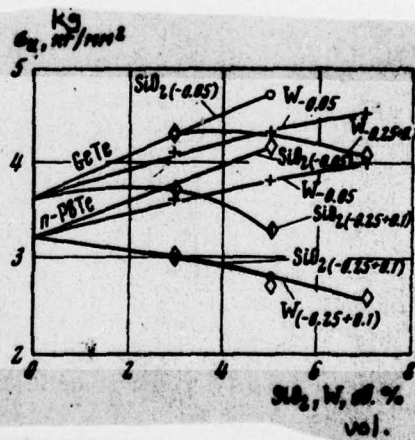


Fig. 2.

Fig. 3. Dependence of the ultimate bending strength of compositions based on n-PbTe and SnTe on the content of tungsten inclusions with dispersity -0.10 mm.

Fig. 4. Dependence of the ultimate compressive (σ_c) and bending (σ_u) strengths of compositions based on p-PbTe on the type and the content of inclusions.

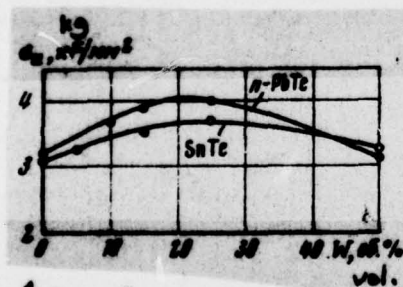


Fig. 3.

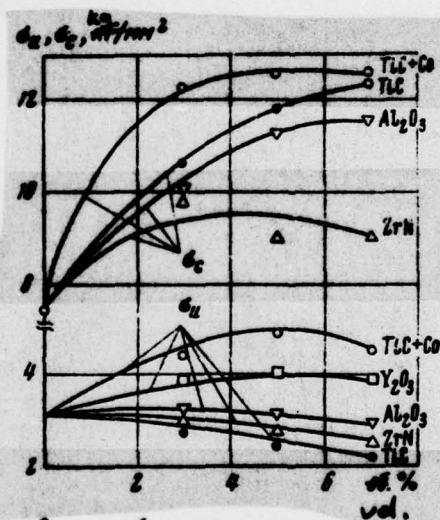
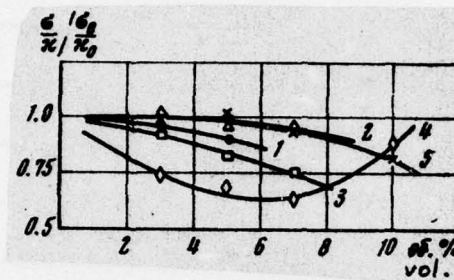


Fig. 4.

Fig. 5. Relative changes of $(\sigma/\kappa)/(\sigma_0/\kappa_0)$ at room temperature of compositions based on n-PbTe on the type and content of inclusions: 1 - SiO_2 , 2 - Fe, 3 - W, 4 - MoSi_2 , 5 - TiC.



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