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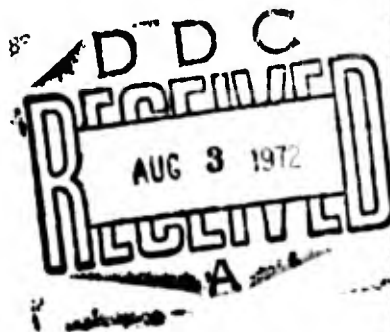
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THERMAL EXPANSION COEFFICIENTS AND
ELASTIC MODULI OF IRON SILICIDES

by

L. P. Andreyeva and P. V. Gel'd



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13. ABSTRACT Thermal expansion coefficients and elastic moduli of solid solutions of silicon in given solutions are studied. Data is given on the chemical and phase compositions and density of the specimens.			

14.	KEY WORDS	LINK A		LINK B		LINK C	
		ROLE	WT	ROLE	WT	ROLE	WT
	Thermal Expansion Thermal Expansion Coefficients Modulas of Elasticity Thermodynamic Stability Temperature Dependence						

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

* ye initially, after vowels, and after ъ, Ъ; e elsewhere.
 When written as ѣ in Russian, transliterate as yѣ or ѣ.
 The use of discritical marks is preferred, but such marks may be omitted when expediency dictates.

THERMAL EXPANSION COEFFICIENTS AND ELASTIC MODULI OF IRON SILICIDES

L. P. Andreyeva and P. V. Gel'd

The subjects of the test were solid solutions of silicon in α -Fe, as well as Fe_3Si , Fe_5Si_3 , FeSi , β -leboite (FeSi_2), α -leboite ($\text{FeSi}_{2.33}$), and Si. The raw materials used in their preparation were chips of monocrystalline silicon (99.99% Si) and reduced iron (99.95% Fe).

The melts were produced in a high-frequency induction furnace in an atmosphere of purified argon.

The test specimens - rods about 3 mm in diameter and about 160 mm long - were obtained by drawing the melt into quartz capillaries. For the purpose of homogenization the specimens were subjected to a 100-h annealing at temperatures which would assure stable test phases: α - and α' -solid solutions, Fe_3Si , FeSi , and α -leboite - at 1100°C , Fe_5Si_3 - at 1000°C , and β -leboite - at 940°C . In this case all specimens were polycrystalline and had the same grain composition and a crystallization texture which was marked to a certain degree. Their chemical and phase compositions were monitored for weight accuracy of the system during the fusion process by chemical, metallographic (in ordinary

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and polarized light), and X-ray (FeK_α and CrK_α -radiation, [VRS] (BPC) camera, [IZA-2] (ИЗА-2) comparator) methods. The data obtained has been generalized in the table.

Chemical and phase compositions of specimens and their density.

(1) Наименование	(2) Образцы						
	Fe	Fe_3Si	Fe_2Si_5	FeSi	FeSi_2	$\text{FeSi}_{2.33}$	Si
(3) Содержание кремния, % (вс.)	0	14,50	23,18	33,80	50,10	53,50	100
(4) Обозначение фазы	α	α'	η	ϵ	ϵ_β	ϵ_α	—
(5) Плотность, г/см ³ :							
(6) по данным авторов	7,80	7,19	6,60	6,10	4,83	4,65	2,330
(7) по литературным данным	7,85	7,23	6,66	6,13	4,86	4,67	2,328

KEY: (1) Name; (2) Specimens; (3) Silicon content, % (wt.); (4) Phase symbol; (5) Density, g/cm³; (6) from data of authors; (7) from reference data.

The temperature dependence of the modulus of normal elasticity was studied by a method of transverse flexural vibrations on a unit, whose basic structural features are similar to that described by Köster [1]. The results of the measurements were processed by standard methods with thermal expansion of the specimens considered. The data were reproducible within a range of 1%.

The thermal expansion of the specimens in a temperature range of 20-1000°C was observed by means of a differential Chevenard dilatometer with photorecording. The coefficient of linear expansion was calculated from the dilatograms by ordinary methods.

The density of the alloys was determined by two independent pycnometric methods using a gas or liquid as the working medium. The results (see the table) are in good agreement with each other and with the reference data.

Experimental Data

Modulus of normal elasticity E. As we see from Fig. 1, with a change in the composition and, particularly, temperature of the specimens their normal elastic modulus changes over a very broad range (about 3 times).

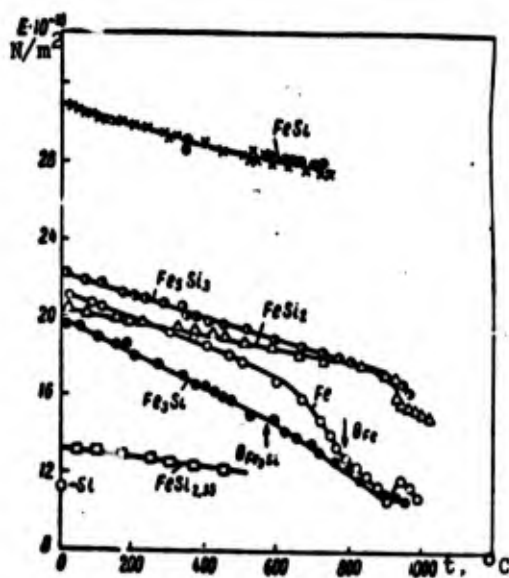


Fig. 1. Effect of temperature on modulus of normal elasticity of iron and its silicides.

The magnitude of the modulus of normal elasticity for iron at 20°C was equal to $21.2 \cdot 10^{10} \text{ N/m}^2$ and declined quite rapidly with an increase in temperature. In a range of $20\text{--}500^{\circ}\text{C}$ the value dE/dT is small and almost constant; yet as it approaches the Curie point it rises significantly, and when $T \approx \theta_c$ reaches a maximum (in absolute magnitude). The polymorphic $\alpha \rightarrow \gamma$ -conversion of iron was accompanied by a sudden increase in E from approximately $10.8 \cdot 10^{10}$ to $12.7 \cdot 10^{10} \text{ N/m}^2$ and a subsequent rapid decrease with a temperature rise.

The established characteristics for iron are in rather good agreement with the reference data [1], which confirms the reliability of the experimental unit which was used.

When silicon is dissolved in α -iron the curve representing the change in the elastic modulus becomes complex (Fig. 2). The extremely insignificant rise in the modulus with a silicon concentration of less than 2% is replaced by a rapid decrease (2-7% Si), characteristic of solid solutions. The transition from the unordered α -solution to the ordered α' -phase changes sign dE/dN_{Si} . Beginning from approximately 6-7% Si the increase in the silicon concentration, accompanied by a growing degree of order in the α' -phase, greatly changes the value of the concentration coefficient, and in a range of from 8-10% Si causes a rise in E. In particular the ordered superstructure of Fe_3Si at $20^\circ C$ is characterized completely by the elasticity modulus ($E_{Fe_3Si} \approx 20 \cdot 10^{10} \text{ N/m}^2$), which differs little from characteristic E for iron. However, in a 20-500°C range the absolute value of dE/dT for Fe_3Si is noticeably greater than for iron, which is seen in the following relationships:

$$E_{0\%Si} = 21,2 \cdot 10^{10} - 7,0 \cdot 10^7 t;$$

$$E_{7\%Si} = 14,5 \cdot 10^{10} - 7,0 \cdot 10^7 t;$$

$$E_{14,5\%Si} = 10 \cdot 10^{10} - 10 \cdot 10^7 t.$$

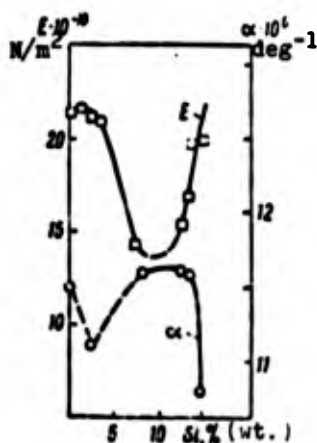


Fig. 2. Isotherms of modulus of normal elasticity E and the coefficient of thermal expansion α of solid solutions at $20^\circ C$.

The polytherm of the modulus of normal elasticity of Fe_3Si (Fig. 1) at a temperature of about $550^\circ C$ undergoes a noticeable break, caused by the magnetic disorder of the superstructure, and from 550 to $900^\circ C$ a linear reduction in E is again observed.

In this range

$$E = 22.5 \cdot 10^{10} - 16.0 \cdot 10^7 t.$$

The reason for the anomaly around 900°C is not sufficiently clear. Possibly it is related to the substantial change at this temperature of the energy distribution of the electrons, which causes a simultaneous sharp change in its electric properties [2]. Here it is necessary to point out only the fact that this effect is not related to structural changes in Fe_3Si .

The modulus of normal elasticity of the η -phase (Fe_5Si_3) was studied under conditions of thermodynamic stability ($830^\circ\text{C} \leq t \leq 1030^\circ\text{C}$), as well as in a metastable state ($t \leq 830^\circ\text{C}$). We found (Fig. 1), that below 800°C

$$E_\eta \approx 22.5 \cdot 10^{10} - 6.0 \cdot 10^7 t.$$

Thus, at all temperatures $E_\eta > E_{\text{Fe}}$, while $(dE/dT)_\eta < (dE/dT)_{\text{Fe}}$. The magnetic disordering of Fe_5Si_3 ($\theta_c = 87^\circ\text{C}$) on the polytherm of the elasticity modulus, described by a number of authors, was not observed, probably because of the low energy of this process.

The E values of iron monosilicide are particularly great. In its homogeneous range in this case magnitude E is greatly diminished (by about 8%) with an increase in the silicon concentration (i.e., with an increase in the concentration of defects in the iron sub-lattice [3]) and diminishes slowly with an increase in temperature.

The elastic moduli of a specimen containing 33.8% Si change depending on temperature in relation to the following expressions: when $t < 400^\circ\text{C}$

$$E_{\text{FeSi}} = 31.2 \cdot 10^{10} - 5.5 \cdot 10^7 t;$$

when $t > 500^{\circ}\text{C}$.

$$E_{\text{FeSi}} = 30.2 \cdot 10^{10} - 3.5 \cdot 10^7 t.$$

For low-temperature β -leboite at 20°C $E \approx 20.5 \cdot 10^7 \text{ N/m}^2$. With an increase in temperature its magnitude is somewhat diminished:

$$E_{\text{FeSi}_2} = 20.5 \cdot 10^{10} - 3.5 \cdot 10^7 t.$$

The sudden change, about 960°C , seems to reflect the peritectoid decomposition: $\xi_{\beta} \rightarrow \xi_{\alpha} + \epsilon$.

The greatest difficulties arose in studying the properties of α -leboite, thermodynamically stable in a temperature range of 915 - 1220°C . To a great extent this was the result of the difficulty encountered in producing specimens in one piece. As a rule developed crack networks were revealed in them, rendering dependence $E_{\xi_{\alpha}} = 13 \cdot 10^{10} - 2.0 \cdot 10^7 t$ in Fig. 1 unreliable.

The coefficients of linear thermal expansion of α . For pure iron at 100°C $\alpha \approx 11.7 \cdot 10^{-6} \text{ 1/deg}$, which corresponds to existing data [4]. Above 200°C the increase in α with the increase in temperature is practically linear (Fig. 3):

$$\alpha_{\text{Fe}} = 11.6 \cdot 10^{-6} + 0.16 \cdot 10^{-8} t.$$

At room temperature the dissolution of silicon in iron changes the coefficient of expansion of the α -phase very little (Fig. 2), but causes a significant increase in its temperature coefficient in the transition from unordered solutions to ordered solutions. In particular, for Fe_3Si in a range of from 20 to 530°C

$$\alpha_{\text{Fe}_3\text{Si}} = 12.0 \cdot 10^{-6} + 1.5 \cdot 10^{-8} t, \text{ i.e.,} \\ (dz/dT)_{\text{Fe}_3\text{Si}} : (dz/dT)_{\text{Fe}} \approx 9.$$

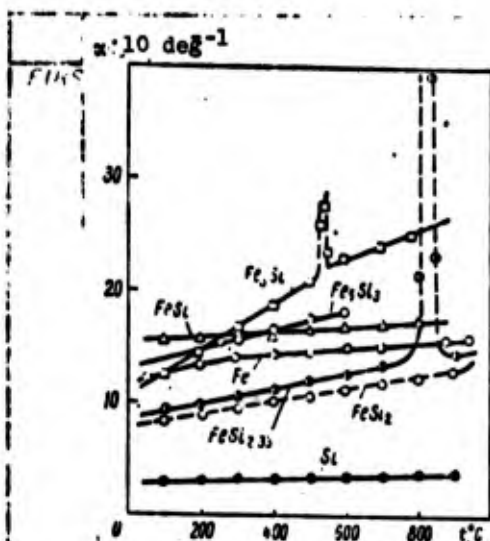


Fig. 3. Temperature dependence of thermal expansion coefficients of silicon, iron, and its silicides.

The anomaly of the dependence $\alpha = f(T)$ near 520°C (Fig. 3) reflects the magnetic disordering of the superstructure ($\theta_c = 530^\circ\text{C}$). If $t > \theta_c$, a linear rise in α is again revealed, but with a somewhat lower temperature coefficient:

$$\alpha = 15.5 \cdot 10^{-6} + 1.2 \cdot 10^{-8}t.$$

In a range of 20 to 600°C the coefficient of thermal expansion of the η -phase rises linearly:

$$\alpha_\eta = 13.0 \cdot 10^{-6} + 0.8 \cdot 10^{-8}t.$$

In the case of low temperatures the ϵ -phase ($\alpha_\epsilon^{20^\circ} = 15.7 \cdot 10^{-6} \text{ deg}^{-1}$) has the greatest value of α . The temperature increase results in a rise in α , which in the first approximation is described by the expression

$$\alpha_\epsilon = 15.7 \cdot 10^{-6} + 0.25 \cdot 10^{-8}t.$$

Here within the concentration stability range of the ϵ -phase only the slightest increase in $d\alpha/dT$ (from $0.25 \cdot 10^{-8}$ to $0.35 \cdot 10^{-8}$) can be observed with an increase in silicon.

Higher silicides and silicon are distinguished by lower coefficients of thermal expansion. In them the value of rigid, short-acting, homopolar bonds is particularly great. With an increase in temperature up to peritectoid ($t_p = 960^\circ\text{C}$) the coefficient of the low-temperature ξ_β -phase increases linearly:

$$\alpha_{\xi_\beta} = 7.7 \cdot 10^{-6} + 0.55 \cdot 10^{-8} t.$$

We find similar characteristics for α -leboite (Fig. 3). In a range of from 700 to 910°C the coefficient of thermal expansion of $\text{FeSi}_{2.33}$ undergoes anomalous change due to the intensive development of its eutectoid decomposition. Outside of these temperatures the experimental data are described fairly well by the expression

$$\alpha_{\xi_\alpha} = 8.3 \cdot 10^{-6} + 0.67 \cdot 10^{-8} t.$$

In the homogeneous region of the ξ_α -phase noticeable changes in α could not be established.

Silicon has the lowest coefficient of thermal expansion and $d\alpha/dT$. Right up to 900°C the experimental data are satisfactorily approximated by the expression

$$\alpha_{\text{Si}} = 2.7 \cdot 10^{-6} + 0.1 \cdot 10^{-8} t.$$

It should be mentioned that the data indicated above are in satisfactory agreement with the results obtained in testing technically pure specimens [5]: scatter did not exceed 10% and, probably, was caused by differences in the purity of the materials used as well as by more careful monitoring of the phase composition of the alloys in the present work.

The value of the expansion coefficient of silicon, equal to $4.15 \cdot 10^{-6} \text{ deg}^{-1}$, often mentioned in reference and monographic literature, is wrong. It is based on inaccurate X-ray measurements

[6], made in a very narrow (10-50°C) temperature range. This follows not only from the results of our measurements, but from those of other authors as well [7].

Discussion of Experimental Data

As we see from the material introduced above, the coefficient of linear thermal expansion and the modulus of normal elasticity of iron silicides vary as a function of composition and temperature in a very broad range.

For solid solid solutions of silicon in α -Fe (Fig. 4) these changes are very unique. Without analyzing them in detail, let us note that the high values of solution heat for silicon in iron, the decrease in the lattice parameters of the α -phase with an increase in silicon concentration, the brittleness of the solid solution, and also the results of magnetic, neutron-diffraction, and X-ray diffraction studies indicate the substantial participation of rigid, homopolar bonds in their mutual inter-particle reaction. At first glance the large values of α and da/dT , as well as the relatively high elastic moduli, contradict this. Agreement of the above-mentioned characteristics is possible, for example, when we assume an inequality of the distribution of the bonds and atoms in the lattice of the α' -phase which grows with an increase in temperature, promoted, in particular, by the coordination numbers and "valence" angles which are uncommon for silicon. This probably leads to the formation of several contiguous pairs of atoms of Fe and Si analogous to those existing in monosilicide and to a weakening of the bonds between similar groupings¹.

¹Small E values, as well as great values of α , and, in particular, da/dT , are determined this way.

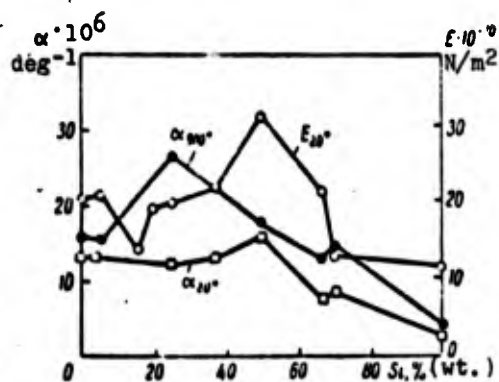


Fig. 4. Isotherms of elastic modulus (at 20°C) and the coefficient of thermal expansion (at 20 and 900°C) of the phase components of the iron-silicon system.

At room temperatures α and E of iron and Fe_3Si are close. However, in the case of a transition to higher silicides they reach a maximum in the case of equiatomic concentrations of the components (ϵ -phase), while leboite and silicon are distinguished by much lower values for these characteristics.

With a temperature increase the structure of the isotherm of the expansion coefficient changes substantially. According to the results of studying technically pure alloys [5] at temperatures above 400°C the α maximum moves in the direction of specimens close in composition to Fe_3Si . We must assume that this is the result of intensification of nonuniformity in mutual interatomic reactions and intensification in the number of weakened bonds in the lattice of the α' -phase [8, 5].

The extremely high values of the elastic modulus and of the expansion coefficient of monosilicide and their relatively slow change with a temperature increase can be explained by the heterodesmic nature of the mutual interparticle reaction, which in this case rests in the existence of other localized bonds in FeSi quasimolecules along with weak resonant ("metallic") bonds between the different types of atoms of neighboring quasimolecules. The relatively low quantities of $d\alpha/dT$ and dE/dT indicate the comparatively slight change in the interparticle reaction in the ϵ -phase in the studied temperature range.

There are different reasons for the much lower elastic modulus values of leboite and silicon. For silicon in particular, where covalent bonds predominate, this is probably caused by a low coordinate number ($k = 4$). The directed, localized nature of the bonds appears at the same time to be the reason for the small values of its thermal expansion coefficients ($\alpha_{20^\circ} = 2.7 \cdot 10^{-6} \text{ deg}^{-1}$). In the case of leboite we must consider not only the fact that the coordination numbers of atoms in it are on the average somewhat less than in the ϵ -phase, but also the fact that in this case overlapping of 3d- and 4s-wave functions of iron atoms and 3p-functions of silicon is not as strongly expressed. Actually, the strong spd-hybrid bonds in monosilicides cause particularly small interatomic distances ($r = 2.29 \text{ \AA}$), while in α -leboite they are not so intensive, and therefore $r = 2.35 \text{ \AA}$.

We see in Fig. 3 that ξ_α - and ξ_β -phases differ in their close values of α and $d\alpha/dT$. The fact that α_{ξ_α} is somewhat greater than α_{ξ_β} and $(d\alpha/dT)_{\xi_\alpha} > (d\alpha/dT)_{\xi_\beta}$, is apparently the result of the following: when $t < 920^\circ\text{C}$ α -leboite is thermodynamically unstable and has a metallic type of conductivity, while β -leboite has clearly expressed semiconductor properties. This gives us reason to believe that in the first case the role of rigid, homopolar bonds is more significant than in the second. And, finally, judging from structural data, the ξ_β -phase has a lower symmetry and a greater dimension of its elementary cell than does the ξ_α -phase [9]. All this leads us to believe that compared with the high-temperature leboite low-temperature leboite should be distinguished by higher force constants (in particular $E_{\xi_\beta} > E_{\xi_\alpha}$) and by lower thermal expansion coefficients ($\alpha_{\xi_\beta} < \alpha_{\xi_\alpha}$), i.e., here the empirical rule on the inverse proportional bond of α and E is realized qualitatively.

It should be mentioned that this relationship is frequently generalized excessively. Thus, Novotny and Lunde [10] have

assumed that for a large group of high-melting carbides of greatly differing structures $E = \text{const.}$ It is difficult to agree with this. In fact, for a simplified model of linearly connected oscillators $\alpha E v = \text{const.}$ Probably v (molecular volume) is a structurally sensitive parameter. Thus in general $\alpha E \neq \text{const.}$ which is confirmed by the following data:

	Fe	Fe ₂ Si	Fe ₃ Si ₂	FeSi	FeSi ₂	Si
$\alpha E \cdot 10^{-3}$ npu 20° C . .	24,2	21,0	29,2	48,3	15,8	3,05
$\alpha E \cdot 10^{-3}$ npu 900° C . .	16,5	28,7	—	47,2	22,1	—

[npu = when]

Expression $ET_{\text{пл}} = \text{const}$ [10] is also not applicable.

Actually, even for metals of similar structure this relationship gives more or less satisfactory results only in the case of the densest packings [11]. In the case of [b.c.c.] (o.c.m.)-lattices possible deviations may exceed 100%.

Apparently conditions are even less favorable for silicides and other low-melting compounds with greatly differing structural and force characteristics (as seen in the material above). Thus, the fusion temperature of the test substances diminishes in the sequence $\text{Fe} \rightarrow \text{Si} \rightarrow \text{FeSi} \rightarrow \text{FeSi}_{2.33} \rightarrow \text{Fe}_3\text{Si}$, and their elastic moduli (at room temperature) $\text{FeSi} \rightarrow \text{Fe}_5\text{Si}_3 \rightarrow \text{Fe} \rightarrow \text{FeSi}_2 \rightarrow \text{Fe}_3\text{Si} \rightarrow \text{Si}$. Furthermore this relationship assumes the nearness of dE/dT , which, as mentioned above, has not actually been observed.

Finally, according to [12], in a range from -100 to $+100^\circ\text{C}$ for many metals $E = kr^m$, where $-m^{-1} = \alpha/\eta = 40.0 \cdot 10^{-3}$ (r - the interatomic distance, and η - the temperature coefficient of the elastic modulus).

Verification of the applicability of this dependence for the studied alloys also established its rough approximation. In particular, at 20°C for different phase components there is more

than a 3-fold increase in α/η :

	Fe	Fe ₃ Si	Fe ₃ Si ₂	FeSi	FeSi ₂
$\alpha/\eta \cdot 10^3$	39,0	22,0	44,8	81,0	67,0

Thus, the indicated empirical relationship cannot be used for substances which do not differ substantially from one another in their structure and the nature of their interparticle interaction.

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