Electrical Resistance of Modified Thermo-exfoliated Graphite

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1 Introduction

The development of new composite materials based on different forms of nanocarbon and nanographite structure is quite a perspective direction in the creation of new materials with a wide range of functionality. Due to the unique combination of such properties as low density, high electrical conductivity, high thermal and corrosion resistance, these composite materials are used in the chemical industry, nuclear and thermal power, automotive and aircraft construction. The expansion of the areas of use of these materials can be achieved by creating new composites based on modified thermo-exfoliated graphite (TEG), i.e. composites formed by the deposition on the surface of TEG particles of metals, their salts and oxides.

The application of TEG as a basis for nanoscaled metal (or its compounds) deposition gives the possibility to create thermo-protective, rust-resisting materials possessing qualitatively new characteristics, which are specified by both the properties of metal and specific properties of graphite and do not inherent to the routine crystalline materials. So, the presence of a small amount of nanostructured phase (about 1–2%) could lead to an essential improving of electrical, magnetic and kinetic properties of composites. The application of TEG as supporter for nanoscaled metal particles is quite promising, due to the possibility to produce on the base of TEG-metal powders the products of different shape. Moreover, no binders are necessary for their production.

There are a number of methods for obtaining a modifying metal component on the surface of nanocarbon and graphite carriers, in particular, on the surface of the

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TEG particles. These modification methods, as well as the effect of the metal modification of graphite surface on the transport, magnetic, mechanical and thermal properties of the obtained composites are described in the literature. In particular, a two-step method of producing "graphite-metal" compositions through intercalated graphite compounds is used to obtain an atomically distributed metal in a graphite matrix. Graphite is intercalated with metal salts, which is then reduced with various reducing reagents [1, 2]. Another method of obtaining composites based on graphite with metals is the reduction of metal from metal halides by intercalated compounds of graphite with potassium in organic solutions [3–5]. Modification of graphite by such methods significantly changes the properties of the source graphite. Modified according to these methods, graphite revealed a new interest effects, in particular, the anomalous Hall effect and linear asymmetric magnetoresistance [6–9]. However, these modification methods are very complex and require significant time and specific chemical equipment. In addition, these methods lead to partial destruction of the graphite layers and the graphite π-system, which can impair the conductive properties of graphite.

Other large groups of modification methods are physical methods of applying metal on the surface of a graphite carrier [9, 10]. The bases of these methods are phase transformations of the first order in the absence of chemical reactions. Such phase transformations are observed in the condensation of atoms on the substrate, condensation of liquid droplets followed by solidification, crystallization from a melt or solution, as well as in the decomposition of solid solutions. However, the use of these methods for the deposition of metal nanoparticles on the surface of dispersed carbon and graphite particles is quite problematic.

One of the most common methods of obtaining metal nanosized particles on the surface of a graphite carrier is a method of reducing the metal from the corresponding metal salts, aqua solution of which impregnates a graphite carrier. Recovery occurs using different types of reducing agents in the presence of stabilizers (surfactants, polymers, etc.) [11–14].

In the presented paper, the relationship between the parameters of structure, as well as between methods of obtaining a modifying coating on graphite surfaces and the concentration of metal in graphite and the electrical resistance of bulk specimens of modified TEG is investigated.

2 Experimental

2.1 Preparation of Bulk Specimens of Modified Thermo-exfoliated Graphite

As a source material for the production of TEG natural monocrystalline dispersive graphite (crystallite size $L \sim 1$ mcm, interplanar spacing $d_{002} = 0.335$ nm, parameter of preferred orientation of crystallites $\eta = 10^4$) has been used. Initially, natural

dispersive graphite has been intercalated with sulfuric acid by the standard liquid-phase technique up to the first stage compound [12]. Then obtained intercalated graphite has been thermo-exfoliated in ascending flow furnace at the temperature 1100 °C (the density of TEG powder is ~0.002 g/cm^3).

The modification of thermo-exfoliated graphite with metals has been performed by the two methods that are completely different in the succession of thermoexfoliation and modification processes [12]. In the first method, the graphite powder has been previously treated with a substance which contained the modifier as an element of chemical composition of this substance. The powder was then placed into cyclone gas-fired furnace where the processes of modification and thermoexfoliation were going on simultaneously at the temperature 1100 °C. This method allows to produce the specimens with concentration of modifier up to 5% mass and density ~(0.002–0.005) g/cm^3.

In the second method (chemical modification), the graphite was previously thermoexfoliated, and then the modifier was deposited on the surface of TEG particles during several successive chemical reactions. The powders of modified TEG produced by this method had the density ~(0.03–0.07) g/cm^3 which considerably exceeded the density of initial TEG. One of the advantages of this modification method is the possibility to produce the modified TEG with desired concentration of modifier (from 5 to 50% mass). The specimens of modified TEG with Ni, Co Fe and Cu (metals concentrations are 10, 30 and 50% mass) have been obtained with this method.

Methods of TEG modification, as well as the structural and morphological peculiarities and phase composition of obtained modified graphite are described in detail in [12].

For measurements of electrical resistance, the bulk specimens of modified TEG have been prepared by method of cold pressing without binder on automatic press with the following parameters: the range of allowable load p is from 1 ton up to 10 tons, and the rate of load growth v_p is from 0.1 up to 10 kN/s. For manufacturing bulk TEG specimens, such moulds have been used: the round press mould with diameter $D = 14$ mm and the rectangular press mould with linear dimensions $a \times b = (15 \times 3)$ mm^2. Thus, specimens for resistivity investigations have been in the form of pallets or rectangles.

The production of the (3–10)–mm-high pallets from the powders of low bulk density requires the application of large-sized press moulds. To avoid this, the powder of pure or modified TEG was previously tightened to a higher density ~(0.22–0.80) g/cm^3. The powder was poured into a glass tube with diameter 14 mm and height from 0.5 to 2 mm, and then the specimen prepared in this way was compressed to the density ~2 g/cm^3 using hydraulic press in the press mould with diameter 14 mm. Using this method, the specimens of modified TEG with nickel, cobalt, iron and nickel–iron have been obtained.

The density of bulk specimens prepared from modified TEG with high modifier concentration (up to 50 mass %) is (2.7–2.9) g/cm^3. So, to produce compacted specimens with high density, no supplementary compacting of TEG powders modified by this method is required. These density values exceed the density of bulk TEG

specimens and density of natural graphite, because the density of metal is considerably larger than the density of graphite. Wherefore for comparison properties of compacting modified TEG parameter of porosity, $\frac{\Delta V}{V} \frac{\Delta V}{V}$ has been used:

$$\frac{\Delta V}{V} = \frac{C_{GR} A_{GR} + C_M A_M}{d \cdot V} - 1, \tag{1}$$

where ΔV is the total volume of pores, V is the molar volume of source TEG or modified TEG, C_{GR} and C_M are, respectively, atomic concentrations of graphite and modifier, A_{GR} and A_M are, respectively, atomic masses of graphite and modifier, and d is the density of bulk specimen.

2.2 Measurement of Electrical Resistance

The measurements of electrical resistance of bulk specimens of modified TEG have been carried out at room temperature along compression axis (ρ_c) and perpendicular to compression axis (ρ_a), and then anisotropy parameter ρ_c/ρ_a was determined for modified TEG specimens with different densities and different modifiers. Also, the temperature dependence of electrical resistance was measured perpendicular to compression axis for bulk specimens of modified TEG with different densities and modifiers in dependence on type and concentration of modifier.

For room temperature measurements of electrical resistance along compression axis and perpendicular to compression axis, specimens of cylindrical form with height $h = (3–10) \times 10^{-3}$ m and diameter $D = 1.4 \times 10^{-2}$ m have been used. Electrical resistance was measured by four-point method described in [7]. The principle schemes of arrangements specially designed for measuring resistance ρ_a perpendicular to compression axis and resistance ρ_c along compression axis are shown in Fig. 1.

The electrical resistance ρ_a was calculated according to the following formula:
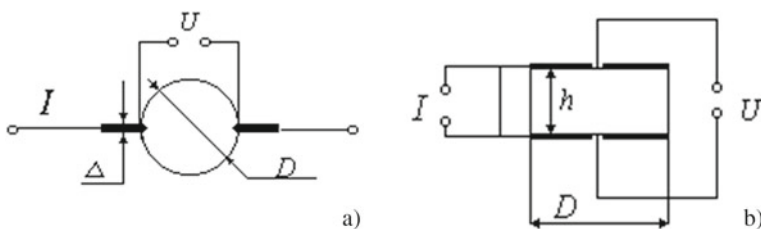

Fig. 1 Principle schemes of arrangements for measuring resistance ρ_a perpendicular to compression axis **a** and resistance ρ_c along compression axis **b**, (notation in the text)

$$\rho_a = \frac{\pi R h}{2 \ln(D/\Delta)}, \quad R = R_{et}\frac{U_x}{U_{et}}, \tag{2}$$

where Δ is the thickness of electrodes supplying the specimen with power, R_{et} is the standard resistance equal to 0.01 Ω, U_x is the potential drop on the specimen, and U_{et} is the potential drop on the standard resistance.

The electrical resistance ρ_c was calculated by the following formula:

$$\rho_c = \frac{\pi D^2 R}{4h}, \tag{3}$$

where R is determined in the same way as in formula (2).

The measurements of temperature dependences of electrical resistance for bulk specimens of modified TEG have been carried out in temperature interval from 77 up to 293 K. The resistance was measured by standard four-probe method in direction perpendicular to pressure axis. The resistivity measurement error did not exceed 0.5%.

3   Results and Discussion

3.1   Resistivity of Modified TEG with Small Concentration of Modifier

The electrical resistances perpendicular to compression axis (ρ_a) and along compression axis (ρ_c) were investigated in source TEG and TEG modified with nickel, cobalt, iron as well as a mixture of nickel and iron by the method of simultaneous thermoexfoliation and modification. The concentration of modifier is ~1% at., and the range of densities was from 0.2 to 2 g/cm³.

Figure 2 presents the dependence of resistance ρ_a (a) and ρ_c (b) on material density for TEG with different modifiers.

As it is clear from Fig. 2a, with increase of the material density, the value of electrical resistance perpendicular to compression axis decreases. As the density increases from nearly 0.2 to 0.4 g/cm³, an abrupt decrease from $(1.0–1.2) \times 10^{-4}$ Ωm to 2×10^{-5} Ωm is observed, i.e. by five or six times. With further increase of density material to 2 g/cm³, ρ_a slowly decreases almost two times. Thus, the increase of material density from 0.2 to 2 g/cm³ leads to decrease of electrical resistance perpendicular to compression axis in almost ten times, and a small addition of metal does not influence the magnitude of resistance and behaviour of the dependence of ρ_a on density.
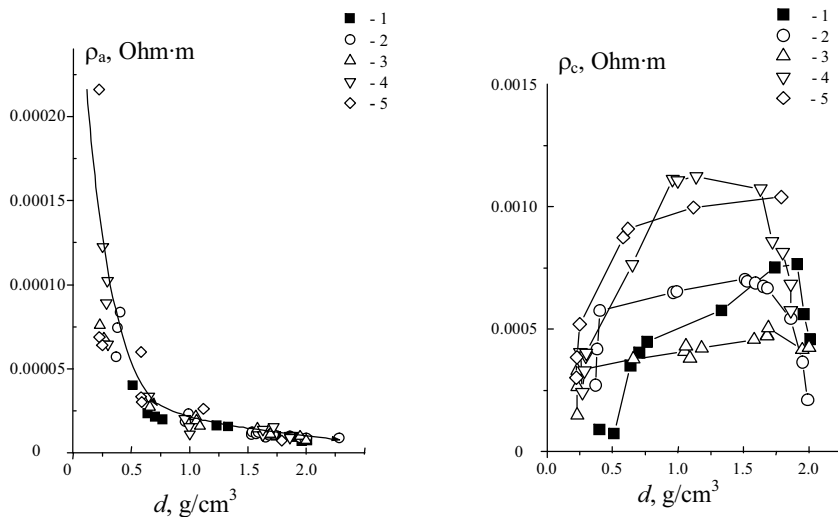
Fig. 2 Dependence of electrical resistance ρ_a **a** and ρ_c **b** on material density for bulk specimens of TEG with different modifiers: 1—TEG; 2—TEG-Ni; 3—TEG-Co; 4—TEG-NiFe; 5—TEG-Fe

As can be seen from the figure, the character of dependence ρ_c on TEG density d is rather complicated for source TEG. In the specimens with small densities $d \sim (0.2–0.5)$ g/cm^3, ρ_c is approximately 1×10^{-4} Ωm, i.e. has the same value as ρ_a. As TEG density increases, resistivity ρ_c gradually grows and comprises 7.5×10^{-4} Ωm for the specimens with the density $d \sim 1.9$ g/cm^3. Further insignificant increase of density to 2 g/cm^3 results in abrupt almost twofold decrease in ρ_c. The dependence $\rho_c(d)$ for modified TEG is very similar to that for pure TEG. But in contrast to ρ_a, which does not change its behaviour for TEG with small additions of metal, the value of ρ_c is different for the specimens with the same density but with different modifiers. For the specimens of modified TEG with nickel and cobalt, the value of ρ_c is close to those obtained for source TEG.

For modified nickel TEG, ρ_c is a little larger, and for modified cobalt TEG, ρ_c is a little smaller than for source TEG. For the specimens of TEG modified with iron and nickel, the value of ρ_c is almost two times larger than in source TEG and comprises $\sim (1.1–1.2) \times 10^{-3} \Omega$m.

As it is known from investigations [12], the modified TEG consists of microcrystallites which have the structure similar to the structure of natural graphite. Every microcrystallite consists of several graphite layers, the electrical resistance along the layers is small, below 10^{-6} Ωm, and electrical resistance perpendicular to the layers is large. The ratio ρ_c/ρ_a for natural monocrystalline graphite is $\sim 10^5$ and for high-oriented pyrolytic graphite is $\sim 10^4$. The source TEG and modified TEG with the density of $(0.2–0.3)$ g/cm^3 are completely isotropic materials in which TEG particles are distributed chaotically. These have the same value of electrical resistance $\sim (1.2–1.5) \times 10^{-3}$ Ωm both perpendicular and parallel to compression axis. In the

specimens of larger densities, i.e. in the specimens prepared under compression at larger pressures, the values of electrical resistance measured perpendicularly and in parallel to compression axis begin to differ. In such specimens, TEG particles are oriented in the same direction at high pressure of compression. As a result, a layered structure is formed perpendicular to compression axis which leads to considerable decrease of electrical resistance in the direction perpendicular to compression axis and its increase in the direction parallel to compression axis. This effect is observed for the specimens with the densities above (0.7–0.8) g/cm^3. With further increase of specimen's densities up to 2 g/cm^3, resistance ρ_a becomes still smaller but unessentially which is associated with the formation of more ordered layered structure. For TEG with the density ~2 g/cm^3, ρ_a is (9–10) $\times$ 10^{-5} Ωm, i.e. it is close to ρ_a value for pyrolytic anisotropic graphite. Small additions of metal (1% at.) practically have no effect on the magnitude of electrical resistance perpendicular to compression axis.

The electrical resistance of compacted materials is known to include electrical resistance of particles themselves that make up compacted material and electrical resistance of contact between these particles. The electrical resistance of metal modifier particles whose concentration is very small in given materials is smaller than that of graphite particles. But the decreased, in comparison with source TEG, electrical resistance in TEG metal system due to metal particles is compensated by electrical resistance of contacts between TEG particles and metal in these systems.

When the density of compacted materials is increased from 0.8 to 1.9 g/cm^3, the electrical resistance ρ_c along compression axis increases unessentially for the TEG specimens and practically does not change for the specimens of modified TEG. The value of ρ_c in the specimens of TEG modified with cobalt and nickel is close to that for source TEG, while in the specimens of TEG modified with iron as well as in TEG modified with iron and nickel, the value of ρ_c is considerably larger. The electrical resistance perpendicular to graphite layers has a jump-like character. Charge transfer takes place on the boundaries of crystallite, grains and defects. And it is in these places where the modifier is deposited in TEG. Therefore, the conductivity in the direction perpendicular to graphite layers takes place through metal impregnations located on the boundaries of crystallite, grains and defects. The decrease in electrical resistance along compression axis due to metal depositions is compensated by contact resistance, and in TEG nickel system as well as in TEG cobalt system, the value of ρ_c is close to that for source TEG. From the data on X-ray analysis, electron microscopy and Auger spectroscopy [12], it was found that iron is present as an oxide Fe_3O_4 in TEG iron and TEG iron–nickel systems, i.e. the boundaries of grains and graphite particles are covered with oxide films, and this results in the increase of contact resistance and growth of total electrical resistance of the system. The abrupt decrease of ρ_c both in source and modified TEG at the densities increasing insignificantly from 1.9 to 2 g/cm^3 is accounted for by the fact that at the pressure applied for producing TEG specimens with the density up to 2 g/cm^3, graphite grains are reduced, graphite layers are partially destroyed, and several graphite layers may be cut through by modifier particles. As a result, the electrical resistance ρ_c decreases.

Figure 3 presents the dependence of anisotropy parameter ρ_c/ρ_a on the density of source and modified TEG specimens.

**Fig. 3** ρ_c/ρ_a ratio for bulk specimens of TEG with different modifiers in dependence on density and type of modifier: 1—TEG; 2—TEG-Ni; 3—TEG-Co; 4—TEG-NiFe; 5—TEG-Fe
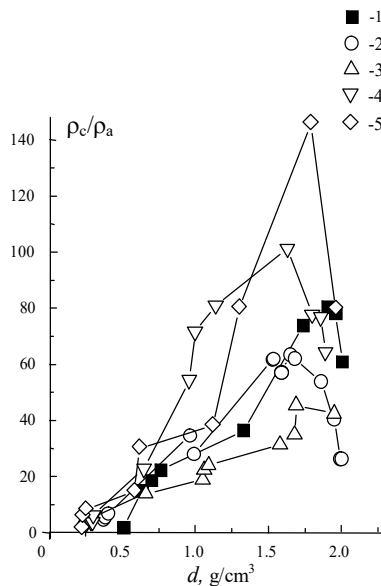
As Fig. 3 shows, the anisotropy parameter ρ_c/ρ_a slowly grows for all compact materials up to the density ~1.9 g/cm^3. After this point, anisotropy parameter decreases. For the specimens with small densities, ρ_c/ρ_a is almost the same for all materials, ρ_c/ρ_a ~1–5, which is typical for porous isotropic materials. The value of ρ_c/ρ_a becomes maximal in TEG with the density 1.91 g/cm^3 and equals to 80. In TEG modified with nickel or cobalt, the value of anisotropy parameter ρ_c/ρ_a is maximal at slightly smaller density, $\rho_c/\rho_a = 63$ in TEG with nickel and $\rho_c/\rho_a = 45$ for TEG with cobalt. The values ρ_c/ρ_a for TEG with cobalt are being smaller than those for source TEG at all densities of both materials. This is associated with the decrease of contact resistance between graphite and metal particles at higher pressures which results in smaller values in comparison with source TEG values of ρ_c. The maximal value of ρ_c/ρ_a ~100–145 in TEG modified with iron and nickel as well as for TEG modified with iron was observed at the density ~(1.6–1.79) g/cm^3. As it was mentioned above, this is accounted for by larger electrical resistance ρ_c for these compacted materials.

Thus, the maximal value of anisotropic parameter ρ_c/ρ_a for compacted source and modified TEG is ~100–140 which is 7–10 times smaller that in pyrolytic anisotropic graphite. This is explained, first, by partial destruction of graphite layers and reduction of graphite grains due to compression and, second, introduction of isotropic metal particles into graphite matrix which "shunt" the graphite layers. All this significantly reduces electrical resistance in the direction perpendicular to graphite layers (i.e. along compression axis of bulk specimen) in comparison with pyrolytic graphite. But at the same time, electrical resistance along graphite layers remains practically the same as in pyrolytic anisotropic graphite.

Fig. 4 Dependence $\rho_a(T)$ for compacted source (2) and modified TEG with 50% mass (1), 10% mass (3) and 30% mass (4) of nickel
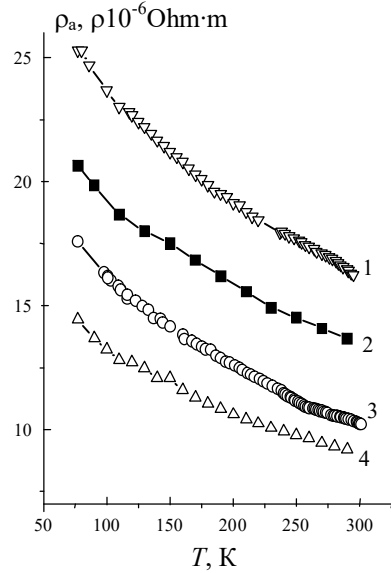
3.2 The Temperature Dependence of Resistivity for Compacted Modified Thermo-Exfoliated Graphite

With the aim to study the influence of structure, type and concentration of modifier on temperature dependence of electrical properties of modified TEG, the electrical resistance in direction perpendicular to compression axis (ρ_a) in TEG modified with different concentrations of nickel, copper, iron and cobalt in the temperature range (77–300) K has been measured.

Figure 4 presents the results of measurements $\rho_a(T)$ dependence for compacted TEG modified with nickel. The nickel concentration in the samples is 10%, 30% and 50% mass. For comparison dependence, $\rho_a(T)$ for source TEG is also presented.

As it is seen from the figure, in general, the character of resistivity temperature dependence $\rho_a(T)$ is similar for all modified with different concentration of nickel TEG and source TEG. The ratio of resistivities at $T = 77$ K and room temperature ρ_{77}/ρ_r is near to that for source TEG and is about 1.5. For modified TEG with 5% mass of nickel, this ratio is nearly equal to that for TEG. For modified TEG with 30% mass and 50% mass of nickel ρ_{77}/ρ_r ~1.6 that is slightly larger than for modified TEG with 10% mass of nickel. The analogous results have been obtained for TEG modified with copper, cobalt and iron in different concentrations. For all these materials, the temperature dependence of electrical resistance is similar in it than for source compacted TEG. For all these compacted modified TEG, the values ρ_{77}/ρ_r are close to the values ρ_{77}/ρ_k for source compacted TEG. The exception is the specimens of TEG modified with 10 mass % of cobalt for which ρ_{77}/ρ_k is ~2.25. All specimens of compacted modified TEG including the specimens with the maximal concentration of

modifier 50% mass or 16–17% at. do not show considerable decrease of resistivity ρ_r in the temperature range from 77 to 300 K. This can be explained, first, by a large value of contact resistance between the particles of TEG and modifier which compensates the decrease in the specimen's resistance due to metal impregnation. Second, in terms of percolation theory, TEG-metal system can be viewed as a three-dimensional net with metal particles as conductivity sites. However, the metal concentration in TEG under study (beyond 50% mass) is small to reach a current threshold, i.e. at this concentration of metal in TEG metal system, the particles of metal have not been grouped into continuous cluster which results in abrupt drop of electrical resistance of the material.

The analysis of the properties of the modified TEG should take into account the whole complex of parameters including the type of modifier, its concentration and the parameter characterizing material structure, namely specimens porosity $\Delta V/V_0$, i.e. the volume of pores (ΔV) in specimen in comparison with the volume specimen.

Figure 5 presents the temperature dependences of electrical resistance $\rho_a(T)$ for compacted modifier TEG with the same concentration of modifier (nickel, cobalt, iron and copper) of 10% mass. For comparison, the figure also presents the temperature dependence $\rho_a(T)$ for source TEG and gives data on porosity for each material.

As can be seen from the figure, ρ_a for all specimens of modified TEG is smaller than for source TEG irrespective of modifier type and specimen's porosity. The comparison of specimens with the same porosity shows that ρ_a is the smallest in TEG with copper, it is almost two times smaller than in pure TEG and is slightly
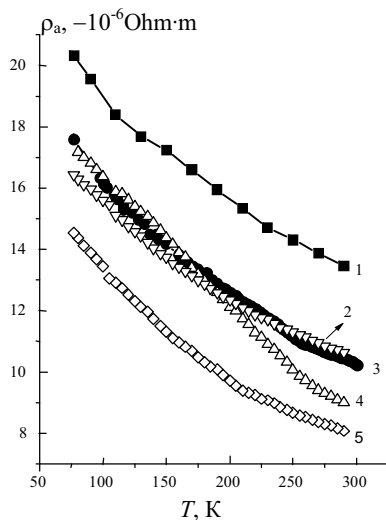

Fig. 5 Dependences $\rho_a(T)$ for bulk specimens of modified TEG with the same concentration of modifier of 10% mass: 1—pure TEG, $d = 1.8$ g/cm^3, $\Delta V/V_0 = 0.2$; 2—iron, $d = 1.94$ g/cm^3, $\Delta V/V_0 = 0.25$; 3—nickel, $d = 1.92$ g/cm^3, $\Delta V/V_0 = 0.27$; 4—cobalt, $d = 2.06$ g/cm^3, $\Delta V/V_0 = 0.18$; 5—copper, $d = 2$ g/cm^3, $\Delta V/V_0 = 0.22$

larger than in TEG with cobalt. The value of ρ_a in TEG with nickel and iron even with larger porosity is smaller than that in source TEG with $\Delta V/V_0 = 0.2$. That is, the modification of TEG with metal in concentration of 10% mass even in more porous material results in slight decrease of electrical resistance in the direction perpendicular to compression axis.

Figure 6 presents the temperature dependence ρ_a for TEG with different modifiers, different modifier concentrations and the same porosity.

As seen from the figure, bulk specimens of TEG with different modifiers, but the same porosity values, have identical temperature dependences of ρ_a, the value of ρ_a being smaller than that for less porous source TEG. The exception is bulk specimen of TEG with cobalt. For this specimen, the value of ρ_a is larger than in the other modified TEG, but this bulk specimen of modified TEG has the largest concentration of modifier (50% mass) among the other specimens shown in Fig. 6.

Thus, the above results show that the modification of TEG by metal results in a slight (less than twofold) decrease of electrical resistance in the direction perpendicular to compression axis. The type of modifier does not influence essentially the value of ρ_a in the materials with the same porosity and modifier concentration of 30% mass in the temperature range from 77 to 300 K.

Figure 7 presents the values of ρ_a at room temperature for all investigated bulk specimens of modified TEG with different modifiers and different concentrations in dependence of material porosity.
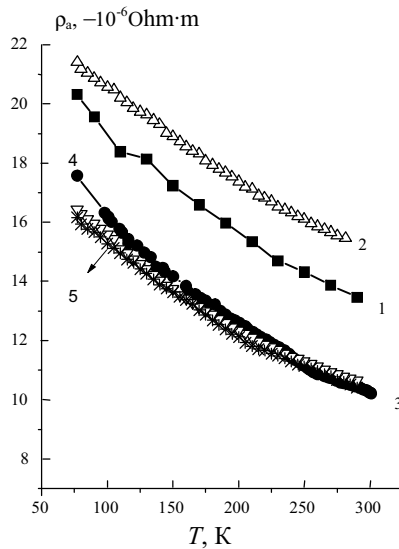

Fig. 6 Dependences $\rho_a(T)$ for bulk specimens of modified TEG with close values of porosity: 1— source TEG, $\Delta V/V_0 = 0.2$, $d = 1.8$ g/cm^3; 2—50% mass Co, $\Delta V/V_0 = 0.26$, $d = 2.85$ g/cm^3; 3—10% mass Ni, $\Delta V/V_0 = 0.27$, $d = 1.92$ g/cm^3; 4—10% mass Fe, $\Delta V/V_0 = 0.25$, $d = 1.94$ g/cm^3; 5—30% mass Cu, $\Delta V/V_0 = 0.26$, $d = 2.3$ g/cm^3
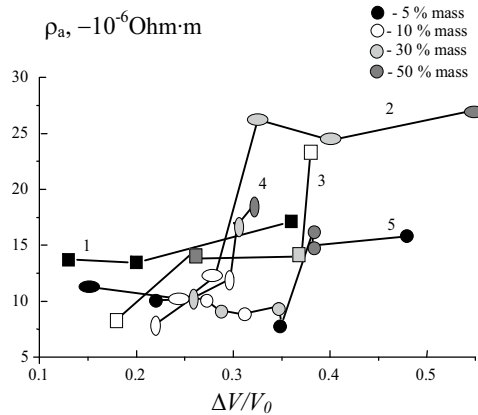
Fig. 7 Dependence $\rho_a(\Delta V/V_0)$ at room temperature for all bulk specimens of modified TEG with different modifiers and different concentrations: 1—source TEG; TEG modified by Fe (2); by Co (3); by Cu (4); by Ni (5). Each metal concentration has its mark on the figure

As it follows from figure the character of ρ_a dependence on porosity is the same for all bulk specimens of modified TEG regardless of the modifier's type. For each TEG modifier system, there is a limiting porosity beyond which the values of ρ_a are very similar and do not depend on modifier concentration in the interval up to 30% mass. At larger modifier concentrations, ρ_a increases abruptly 1.5–2 times. Further on as the porosity of bulk specimens grows to 0.6, ρ_a again changes insignificantly.

For bulk specimens of modified TEG with iron and copper, the limiting porosity is ~0.3, and for bulk specimens of modified TEG with cobalt and nickel, it is ~0.35.

4 Conclusion

Thus, the performed investigations of electrical resistance in bulk specimens of modified by metals TEG allow to make the following conclusions:

1. Impregnation of metal into TEG in small concentrations does not significantly influence the magnitude of electrical resistance of bulk specimens in the direction perpendicular to compression axis.
2. Impregnation of metal into TEG results in a significant decrease of anisotropy parameter ρ_c/ρ_a in modified TEG; the more uniform and even the distribution of metal particles in TEG, the greater is the increase of anisotropy parameter.
3. Impregnation of modifier from 10% mass to 50% mass into TEG results in insignificant (less than two times) decrease of electrical resistance perpendicular to compression axis of the specimens. The magnitude of resistance does not depend essentially on modifier type or modifier concentration (at the concentration below 30 mass %) but depends on material porosity.

4. There exists limit porosity for each TEG modifier system at which electrical resistance perpendicular to compression axis increases sharply. Electrical resistance in the specimens below or above this limit practically does not depend on modifier concentration. The more uniform and even the distribution of metal particles in TEG, the greater is the value of limit porosity.

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