



Peculiarities of the structure of copper- and nickel-fluoropolymer composites fabricated by explosive pressing



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ABSTRACT

The present study demonstrates investigation two composites based on PTFE. The composites were obtained by explosive pressing of powder mixtures: 30%PTFE and 70%Cu and 30%PTFE and 70%Ni. The morphology, composition, molecular structure, thermal and magnetic properties of these materials were researched. Differences in the characteristics of composites depending on using a metal were detected.

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1. Introduction

Polytetrafluoroethylene (PTFE) is one of the well-known basic fluoropolymers. The PTFE structural unit comprises a linear macromolecule—a polymer chain composed of monomer units – CF₂– [1,2]. High strength of the bond between fluorine and carbon atoms yields a unique combination of useful properties of this material that distinguishes it from other polymers [1]. Although PTFE and products based on it are extensively applied in different fields of industry, in household, and even in medicine, the requirements of advanced technologies stipulate for the necessity of improvement of the materials functioning parameters, which is implemented through modification of available materials or fabrication of novel ones. Modified forms of the material having some promising properties were fabricated using different methods of PTFE treatment. For instance, the thermal gas-dynamic treatment was used to obtain ultradispersed PTFE (UPTFE-FORUMTM) [1,3]; the PTFE laser ablation yielded a fiber-porous material “Griftext” [4]; whereas the PTFE radiation treatment at a temperature above that of the crystalline phase melting increased its wear resistance by four orders of magnitude and its radiation stability by two orders of magnitude [5].

One of the promising methods of polymer modification is the explosion treatment (explosive pressing, EP) [6]. Application of explosives in materials treatment enables one to create shock waves in a solid body, which propagate at high velocity from the

loading surface to the solid depth. Explosive detonation can provide pressures from dozens of MPa to a few hundreds of GPa and a broad range of temperatures from dozens to a thousand of centigrades in the material to be treated. The latter affects not only the material density, but also its other characteristics, which provides the emergence of new properties not inherent to any individual component [7]. In [8,9], this method was applied to PTFE and UPTFE-FORUMTM materials. The studies demonstrated that, aside from changes in a number of characteristics of these materials during treatment, metal atoms had been found in the surface layer of polymer samples. The latter fact was explained by penetration of metal into polymer in the process of explosion as a result of a partial interaction of polymer with the steel ampoule containing the sample. The possibility of fabrication of powder mixtures of composite metal-polymer materials (for instance, PTFE + metal) using the explosive pressing was shown in a series of works performed at the Volgograd State Technical University [10,7,11–15]. The present work was devoted to studying of the composites 30%PTFE + 70%Cu and 30%PTFE + 70%Ni. The main attention was paid to revealing of the composite morphology, molecular structure, phase composition, and thermal properties.

2. Results and discussion

2.1. Sample 30%PTFE + 70%Cu

2.1.1. Morphology

In the course of the SEM study of the morphology and element composition of the sample 30%PTFE + 70%Cu, two types of areas marked as 1 and 2 in Fig. 1 were revealed. The data of the

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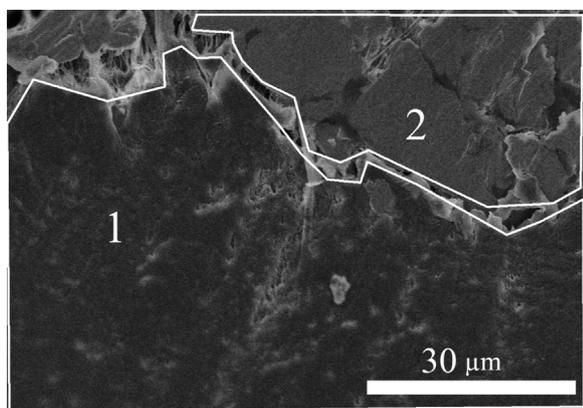


Fig. 1. SEM images of the sample 30%PTFE +70%Cu.

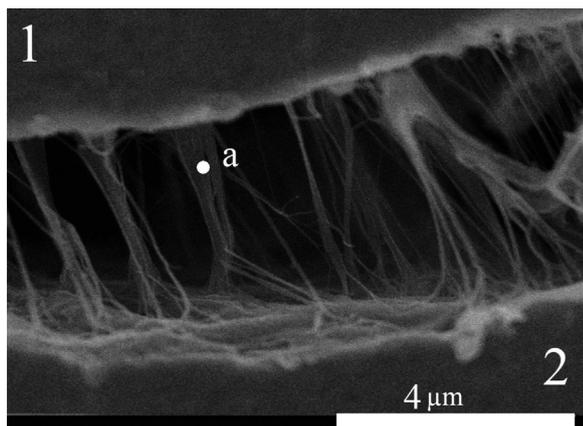


Fig. 2. SEM image of the sample 30%PTFE +70%Cu.

performed element analysis of these areas, as compared to the initial PTFE, are shown in Table 1. According to the obtained data, the area 1 comprises the metal part containing carbon, whereas the area 2 consists of fluoropolymer formations.

Since PTFE was the initial fluorocarbon material, it was expected that the area 2 would consist of PTFE particles. In addition, as was found in [8], the explosion treatment of PTFE did not significantly change the material composition. As was already mentioned, a linear polymer chain $[C_2F_4]_n$ was the PTFE structural unit, so that the carbon/fluorine ratio was 1/2. However, in the area 2 the C/F ratio is almost 1/3. Such an increase of the number of fluorine atoms could occur at the expense of destruction (at least, partial) of PTFE under effect of explosion treatment. A part of the released carbon remains in the form of carbon black, thus transferring into the area 1. The released fluorine forms CF_3 groups (end or side ones). The above features are characteristic for PTFE destruction [16]. In the area of transition of these monolith areas (1&2), one observes from the SEM data the area composed of fibril structures (length 1–5 μm , thickness 40–150 nm).

Table 1
Element analysis of the sample 30%PTFE + 70%Cu and PTFE (F4) performed using the SEM method.

Sample	C at.%	F at.%	O at.%	Cu at.%	F/C
PTFE initial	33.2	66.8			2
PTFE + Cu (1)	24.78			75.22	
PTFE + Cu (2)	25.57	74.43			3
Fibril in the point (a) in Fig. 2	27.31	43.53	8.25	20.91	1.6

Fibril structures were also observed in the initial PTFE, but they were scattered over the whole volume [17]. Accumulations of nanofibrils of diameters of 50–200 nm and of lengths of 5–10 μm were observed in the UPTFE surface layer upon explosion treatment even at small magnifications [9]. Loose fibril structures of a size of about 1 μm were observed in the EP-treated PTFE [8], in which case the surface layer fibrils were of a larger length of 10–20 μm . One should mention that the surface layer contained metal particles upon EP treatment in both cases (PTFE and UPTFE). The energy-dispersive analysis of the sample 30%PTFE + 70%Cu demonstrated that the transition area contained copper, carbon, oxygen, and fluorine (Table 1). One can assume that in all three cases (PTFE, UPTFE, Cu + PTFE), under effect of pressure and interaction with metal particles, fibril structures contained in the initial PTFE were separated from each other and removed from PTFE. One should also mention that in all cases the latter comprises the carbon-enriched material.

As was found at studying the sample 30%PTFE + 70%Cu using a Raman microscope, it had a heterogeneous structure. As seen from Fig. 3, it is composed of a monolith mass with embedded particles of sizes from 50 up to 200 nm. The Raman spectra measured in the monolith mass center and in embedded particles are different from each other (Fig. 4). The spectrum of the analyzed particle substance is virtually identical to that of the initial PTFE.

2.1.2. Molecular structures of particles

The above indicates to the fact that, first, these particles are composed of PTFE and, second, the PTFE molecular structure, in general, does not change in spite of the treatment. The spectrum

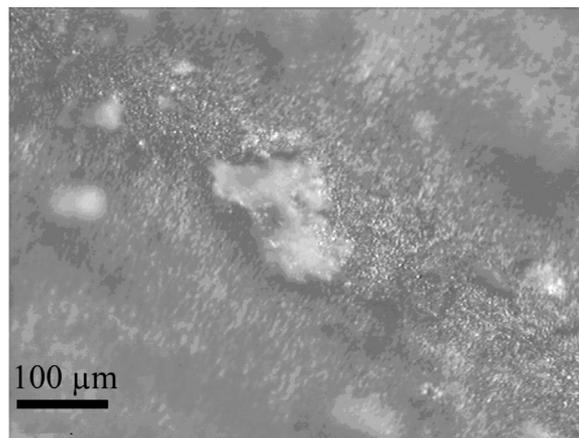


Fig. 3. Microphotograph of the sample 30%PTFE + 70%Cu obtained using a WiTec alpha500 confocal Raman microscope.

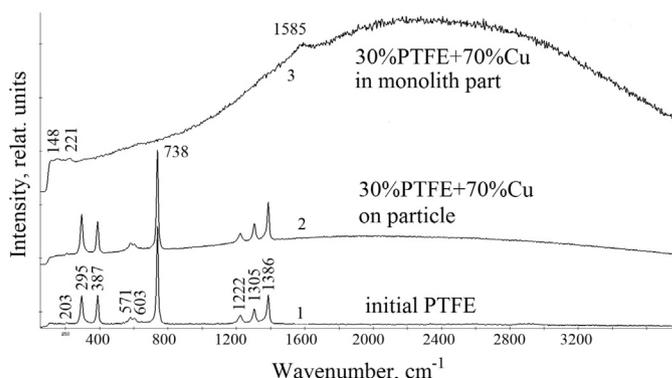


Fig. 4. Raman spectra of the sample 30%PTFE + 70%Cu and the initial PTFE.

contains all the bands characteristic for the initial PTFE: 1386 cm^{-1} —stretching vibrations (C–F); 1305 cm^{-1} —asymmetric stretching CF_2 vibrations; 1222 cm^{-1} —stretching C–C vibrations; 738 cm^{-1} —symmetric CF_2 vibrations; 603 cm^{-1} —symmetric deformation CF_3 vibrations; $571, 387\text{ cm}^{-1}, 295\text{ cm}^{-1}, 203\text{ cm}^{-1}$ —out-of-plane CF_2 vibrations [18]. A small difference in band intensity ratios at 387 and 295 cm^{-1} could be related to changes in packing of chains constituting the PTFE structure under pressure.

The Raman spectrum of the sample monolith part is radically different from that of the particle. The former does not contain bands characteristic for the PTFE spectrum, whereas, along with the general increase of the spectrum background, one observes very weak bands at 148 and 221 cm^{-1} and stronger band at 1585 cm^{-1} . The latter band was discussed in [18] and related to the formation of graphite-like structures as a result of a partial decomposition of the initial PTFE. The reasons of the background increase are not clear. Possibly, the effect is concerned with a strong scattering on copper particles and heating of this sample part under effect of laser beam. This situation was often present in studying copper-containing samples by the Raman spectroscopy. Since weak bands at 148 and 221 cm^{-1} are present in Cu_2O spectra [13], this can be explained by partial (very small) copper oxidation.

The IR reflection spectrum of the sample under examination is very similar to that of the initial PTFE. The differences are concerned with the emergence of weak unstructured bands around $1400\text{--}1500\text{ cm}^{-1}$, which can be attributed, just like in Raman spectra, to the emergence of graphite-like structures or oxidized carbon black in the sample.

The sample surface was investigated by means of the XPS method. As follows from the pool of data for the sample 30%PTFE + 70%Cu shown in Table 2, carbon present in the surface layer is predominantly of the aliphatic nature (C–C, C–H), 49.2 at.% and contained, in smaller parts, in ‘weakly fluorinated’ –C–F– and ‘weakly oxidized’ –C–O– groups (approximately 5.5 at.% each). A part of carbon is contained in the composition of CF_2 -groups (5.8 at.%). To sum up, the above fluorine-containing carbon compounds must contain about 17.1% of fluorine, which is in agreement with the fluorine quantity and state observed in the spectra. In the upper layer, copper is in the state of Cu^{2+} or, possibly, in the composition of $\text{Cu}(\text{OH})_2$ and/or CuO . According to the spectra, oxygen is of the hydroxyl (organic) nature.

Upon etching the upper layer (about 5 nm), copper is observed in larger quantities and less oxidized states (Cu^+ and/or Cu^0). The ratio of CF_2 groups CF, possibly, changes into prevalence of the latter, as follows from changes in elements contents and respective manifestations in the spectra (one observes broadening and emergence of a shoulder for the band F1s and the decrease of the CF_2 band intensity). All these facts must be related to exposure of the lower layer, although in this case one should also take into consideration the destruction of initial compounds during ion bombardment (in the process of study).

Table 2
Samples element analysis performed by the XPS method.

	Cu/Ni	F	O	CF_2	CF, COR	CC
F4Cu	934.9	689.9	532.2	292.6	288.7–286.9	285.0
	2.3	17.2	12.5	5.8	11.1	49.2
Etched	933.1	690.1	532.4	293.1	288.6–286.7	285.0
	4.4	15.3	5.4	3.2	15.4	56.3
Scraped	932.9	690.1	532.0	292.8	288.7–286.7	285.0
	4.7	50.9	7.2	19.0	1.5	16.6
F4Cu–S	933.8	689.9	531.4	292.4	–	285.0
	1.6	54.7	4.2	24.1	–	15.5

Upon mechanical scraping of the surface layer (~dozens of microns), the composition is dominated by metal (or weakly oxidized, Cu^+) copper and $(\text{CF}_2)_n$. The copper/fluorine weight ratio is about 3/10. Noticeable quantities of aliphatic carbon and oxygen are present here as well.

The upper layer of the side face (on cleavage) consists predominantly of $(\text{CF}_2)_n$. The contributions of aliphatic carbon are significant. The contribution of copper ($\text{Cu}^{0/+}$) is negligible.

2.1.3. Phase composition

The conclusions made on the basis of the data obtained by the methods of IR and Raman spectroscopy are in full agreement with XRD results for the sample Cu + PTFE (Fig. 5). The XRD pictures of this sample contain a strong reflection assigned to copper and weak ones characteristic of PTFE and Cu_2O . The reflections intensity ratio does not allow judging on the quantitative composition of the mentioned formations, since copper forms solid monolith areas, whereas PTFE and, possibly, oxidized copper are present in the composition of nanosized particles.

2.1.4. Thermal properties

The data of DSC studies of this sample (see Fig. 6) indicate to the presence of two temperature features of the DSC curve. One (high-intensity) peak is almost identical to that observed on the DSC curve of the initial (crystalline) PTFE and corresponds, according to [2], to PTFE melting. A very small increase of the melting point upon EP (3°) indicates to the increase, albeit insignificant, of the polymer crystallinity. The second feature manifested as a low-intensity peak at 317°C can be also related to melting, but in this case to that of the part of the fluorocarbon material formed during partial destruction of PTFE in the process of EP treatment. PTFE short chains and defect structures are characterized by lower melting points [16]. The possible emergence of such formations was shown above by the energy-dispersive analysis data.

2.2. Sample 30%PTFE + 70%Ni

2.2.1. Morphology

The morphology of the sample 30%PTFE + 70%Ni is different from that of the above described copper composite. The sample has a developed relief, individual particles of the initial nickel powder and PTFE cannot be discerned, while areas of different morphology are clearly seen (Fig. 7). Probably, in this case, the explosion treatment yields the particle deformation, sintering, or compacting. Similar structure was found in [13] at AFM studies of a

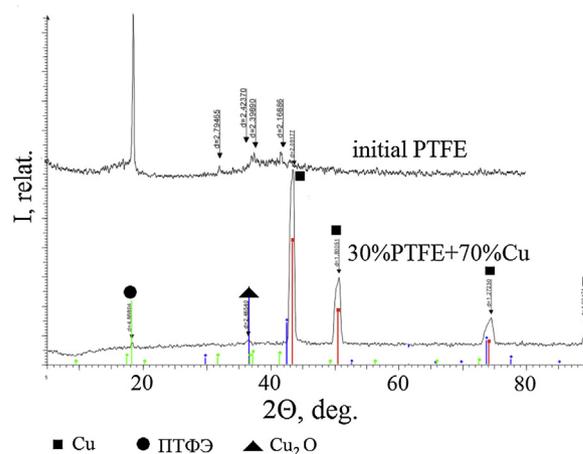


Fig. 5. X-ray images of the sample 30%PTFE + 70%Cu.

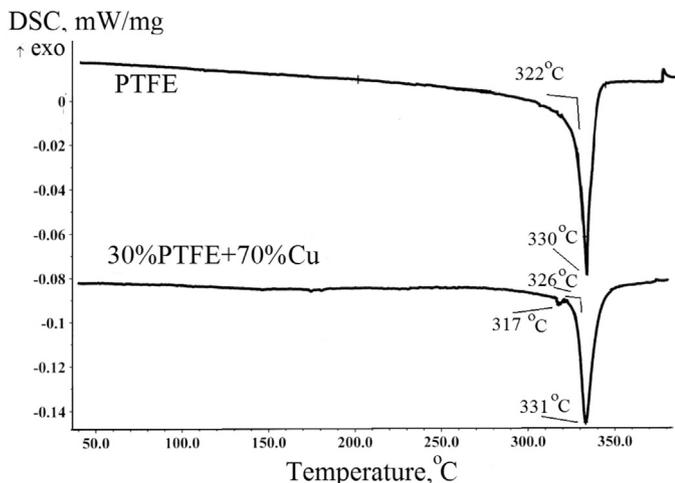


Fig. 6. DSC curves of the initial PTFE and the sample 30%PTFE + 70%Cu.

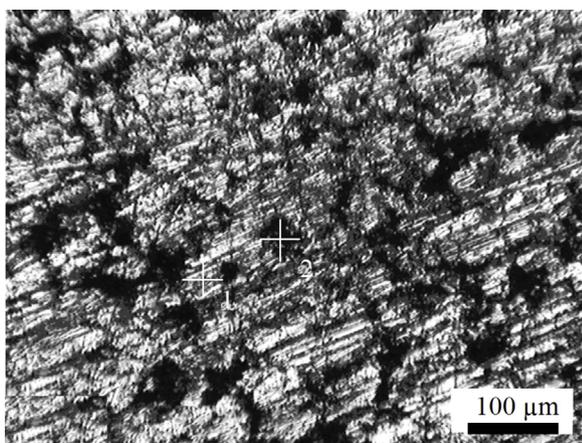


Fig. 7. Microphotograph of the sample 30%PTFE + 70%Ni obtained by a WiTec alpha500 confocal Raman microscope.

composite fabricated through explosive pressing of nickel and PTFE.

2.2.2. Molecular structures of particles

The Raman spectra (Fig. 8) registered in different parts of the sample (1&2) contain the bands at 1360 and 1579 cm^{-1} in both cases. These bands, just like in the case of the copper sample, can be explained by the emergence of carbon formations in the sample after EP treatment [19]. Most probably, the EP treatment (as for the copper sample) results in a partial destruction of the initial PTFE and the formation of new graphite-like structures and carbon black. One should mention that the process occurs during EP of polytetrafluoroethylene (F-4) in the absence of metals as well. Such bands emerge in Raman spectra of PTFE (F-4) that underwent EP [8]. Aside from the above discussed bands, Fig. 8 (spectrum 2) contains bands at 295, 387, 738, and 1305 cm^{-1} characteristic for PTFE Raman spectra (see above). Therefore, dark areas consist of PTFE particles, whose structure, in general, does not change. A more detailed conclusion can be made on the basis of analysis of the IR reflection spectrum of the sample 30%PTFE + 70%Ni: the bands (1208, 1152, 748, 638, 554, 504 cm^{-1}) characteristic for the IR spectrum of PTFE are observed in that of the sample, which means that PTFE remains in the sample composition upon EP treatment (Fig. 9). However, the band at 981 cm^{-1} appears in the spectrum. It corresponds to vibrations of the side trifluoromethyl –CF–CF₃ groups [20,21].

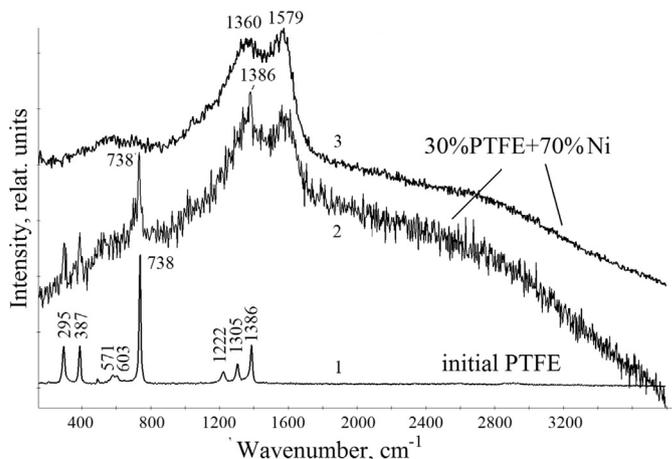


Fig. 8. Raman spectra of: PTFE and 30%PTFE + 70%Ni in points 1 and 2 (2, 3).

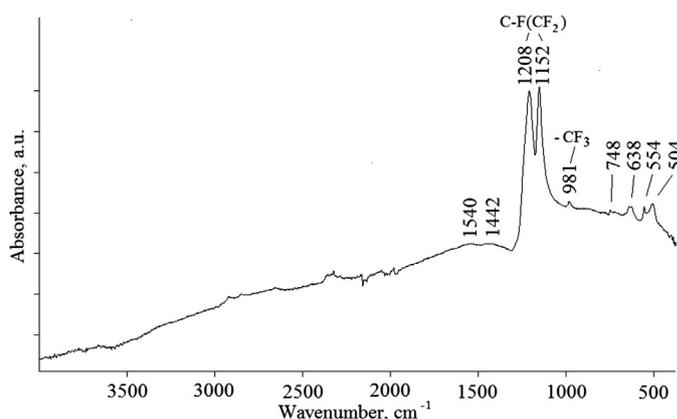


Fig. 9. IR reflection spectrum of the sample 30%PTFE + 70%Ni.

As regards surface properties, the composition of the upper layer of the sample 30%PTFE + 70%Ni is similar to that of 30%PTFE + 70%Cu described above. One can also observe the same components (CF₂, CF, OH, CC) with contents close to those in the ‘copper’ sample. In this case, nickel is in the strongly oxidized state, like Ni₂O₃ (566 eV) (Table 3).

On the cleaved face, the upper layer of the sample, just like in PTFE-Cu, consists predominantly of (CF₂)_n. The contribution of aliphatic and weakly fluorinated carbon is here somewhat larger (possibly, because of capturing edges of other faces), while the presence of nickel is not detected. The absence of nickel can be explained by the fact that the cleavage takes place over cracks filled with PTFE that is detected using the XPS method.

2.2.3. Phase composition

PTFE particles are concentrated around dark formations (area 2, Fig. 7). We could not reveal whether nickel was present in this area,

Table 3
Samples element analysis performed by the XPS method.

	Ni	F	O	CF ₂	CF, COR	CC
F4Ni	856.8	689.7	532.2	292.4	288.3–286.2	285.0
	3.4	16.5	13.7	4.4	16.3	45.7
F4Ni-S	–	690.3	532.6	292.9	288.8–286.7	285.0
	–	40.1	6.5	16.8	7.3	29.3

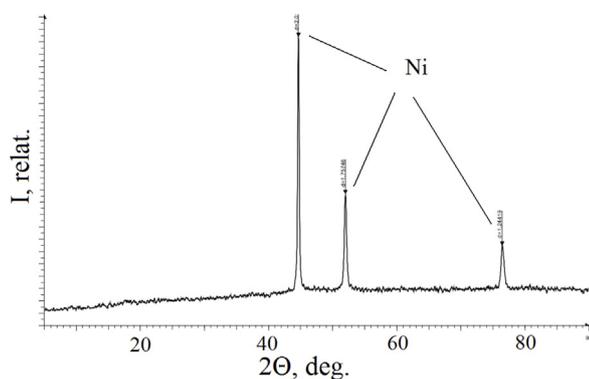


Fig. 10. Diffractogram of the sample 30%PTFE + 70%Ni.

since the energy-dispersive analysis method does not provide satisfactory results in case of samples with magnetic nickel. The whole monolith area must consist of nickel particles, which is corroborated by XRD data (Fig. 10). PTFE reflections are surprisingly absent on the X-ray image. Since the presence of PTFE was corroborated by IR and Raman spectroscopy methods, the PTFE absence on the X-ray image can be related to essentially amorphous polymer state in the sample. Although it is in agreement with [13] stating significant changes in the PTFE crystallinity degree at such composite explosion treatment, the absence of halo on the diffractogram is surprising. In view of this, we assume that the effect can be related to another reason: nanodispersity of PTFE particles.

2.2.4. Thermal and magnetic properties

The DSC curve of the sample 30%PTFE + 70%Ni is also rather unusual. Unlike that of the sample 30%PTFE + 70%Cu, in which one can easily discern the peak of crystalline PTFE melting, in Fig. 11 one observes two fuzzy temperature features. The feature with the maximum at 279.9° could correspond to more defect (or finer) crystalline structure of PTFE. The second feature must be related to the formation of an interfacial area due to the adhesion interaction of PTFE with nickel, since it coincides almost completely with the DSC curve of the nickel sample (dashed line in Fig. 11). The latter effect can be related to the metal demagnetizing ability in the revealed temperature range. To corroborate this assumption, magnetic properties of the sample 30%PTFE + 70%Ni were

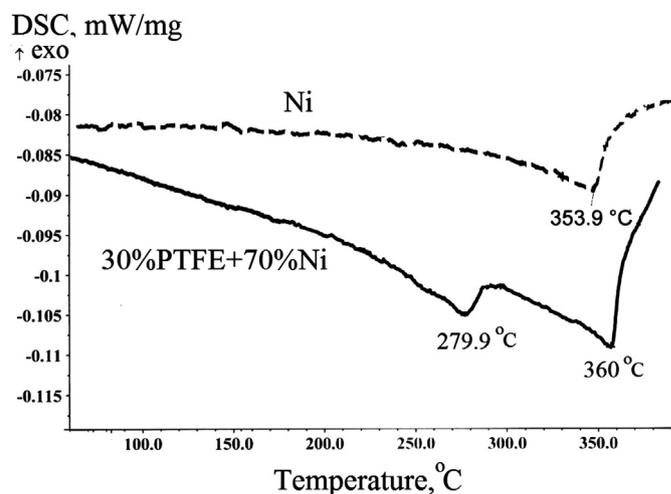


Fig. 11. DSC curve of the sample 30%PTFE + 70%Ni.

investigated. The results of investigations of magnetic characteristics of the composite material 70%Ni + 30%PTFE are shown in Figs. 12a and b. According to the dependencies of magnetization on the field intensity obtained at 27 and -271 °C (Fig. 12b), this material can be considered as high-coercive with the coercive force higher than 26 Oe, saturation magnetization about 42 emu, and the saturation field intensity about 3000 Oe. The temperature decrease results in just small increase of the saturation magnetization and does not change the type of the saturation magnetization on the field intensity.

As follows from the magnetization temperature dependence obtained in the range -271 to 727 °C (Fig. 12a), the composite manifests magnetic properties similar to those of metallic nickel. The Curie temperature of the composite under study is close to that for metallic nickel (358 °C) [22] and equal to 362 °C (Fig. 12a). Note that it almost coincides with the temperature feature on the DSC curve.

3. Conclusions

Studies of composite materials fabricated using the method of explosive pressing of mixtures of copper or nickel powders with polytetrafluoroethylene demonstrated that in the process of

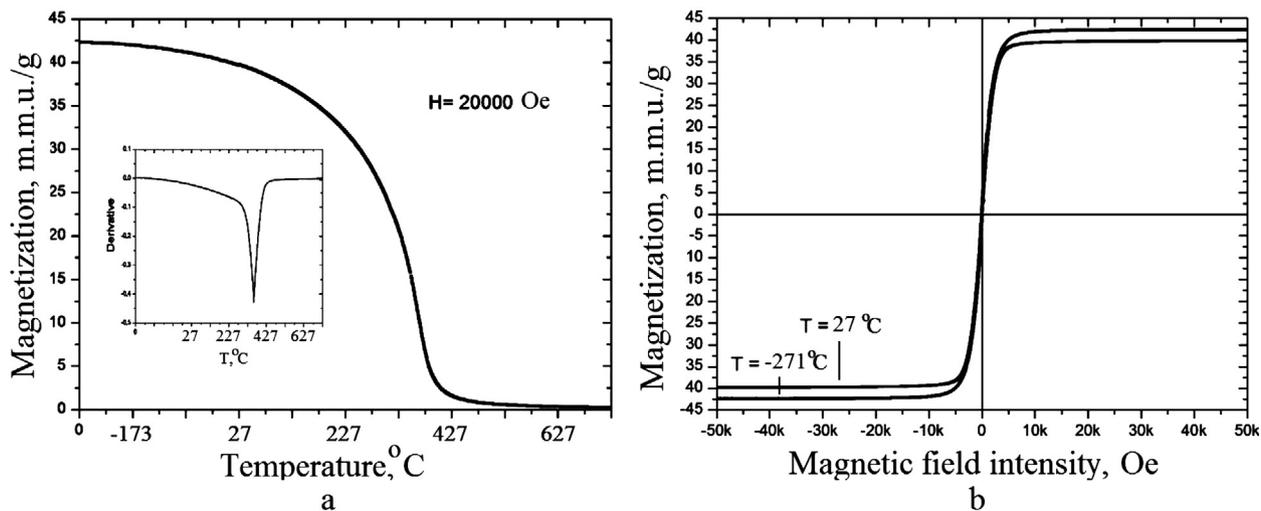


Fig. 12. Dependence of magnetization on temperature (a) and curves of remagnetization (b) of the sample 30%PTFE + 70%Ni.

treatment the material 30%PTFE + 70%Cu is formed as a copper monolith with embedded polytetrafluoroethylene crystals. The areas of particle-monolith transition consist of fibril structures composed of fluorocarbon material and copper (including the oxidized one), which indicates to the adhesion interaction with PTFE followed by formation of chemical bonds.

In the course of fabrication of a composite with nickel, a loose nickel monolith is formed: its pores and interfacial layer are filled with formations from melted polytetrafluoroethylene particles, whose molecular structures contain branching. The nickel magnetic properties are preserved in the final material.

4. Experimental

The present work describes studies of two samples fabricated using the method of explosive pressing by a ring shock wave of a pressure of 1–1.5 GPa. The samples were fabricated by explosion treatment of powder mixtures: 30%PTFE (grade F-4) and 70%Cu and 30%PTFE (grade F-4) and 70%Ni. The samples appear as monolith bars of dark-orange and grey colors, respectively.

To study the samples morphology, a Hitachi S5500 high-resolution scanning electron microscope (Japan) was used. The samples local element composition was investigated on a Thermo Scientific energy-dispersive spectrometer (EDS) installed as an accessory on the S5500 microscope. The samples were prepared for SEM through sputtering of a conducting material (gold) on a sample followed by a placement into the electron microscope using a special holder.

The samples IR reflection spectra were recorded at room temperature on a VERTEX 70v spectrometer (Bruker, Germany) in the range 4000–400 cm^{-1} with the recording error not higher than 1 cm^{-1} , using a platinum ATR accessory with the diamond crystal.

The samples microphotographs and Raman spectra were obtained on a WiTec alpha500 confocal Raman microscope. The laser wavelength—531.8 nm; the signal accumulation time—1 s, spectra were averaged on 100 measurements, the measurement error—4 cm^{-1} .

DSC curves were obtained using a DSC 200 F3 Maia calorimeter (NETZSCH, Germany). The sample under study was placed into an aluminum crucible and heated from room temperature up to 580 °C at a rate of 5 °C/min.

The composition of crystalline phases was established from X-ray pictures of samples registered using a D8 ADVANCE diffractometer (Bruker, Germany) in $\text{CuK}\alpha$ radiation.

XPS spectra were recorded on a SPECS complex for surface studies (Germany) using a 150-mm hemispheric electrostatic

analyzer. $\text{MgK}\alpha$ radiation was used for spectra excitation. The bond energy calibration was performed on the C1s hydrocarbon line with the accepted $E_{\text{bond}} = 285.0$ eV.

Studies of magnetic properties were carried out on a PPMS9TECII vibromagnetometer (Quantum Design, USA) in the temperature range 2–1000 K and the field intensity range ± 5 T.

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