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Peculiarities of electromagnetic waves absorption in polymer magnetic nanocomposites (La,Sr)MnO_3

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The results of research of the nonresonant and magnetic resonance absorption of electromagnetic waves in polymer magnetic nanocomposites ($La_{0.7}Sr_{0.3}MnO_3$) in 10–40 GHz and 60–78 GHz frequency bands are presented. The absorption magnitude up to –7.5 dB at 32–35 GHz has been detected for the composite with 60 % filling and a layer thickness of 0.53 mm. The analysis of results obtained proves a high level homogeneity of magnetic nanoparticles distribution in the polymer matrix. The most probable reasons of unusually large value of the spectroscopic splitting factor, obtained by Electron Spin Resonance technique, are under discussion. The results of design of the technology and the manufacturing technique of ferromagnetic composites together with epoxy-polysiloxane polymer matrix are presented.

Приведены результаты исследований особенностей нерезонансного и магниторезонансного поглощения электромагнитных волн в полимерных магнитных нанокомпозитах ($La_{0.7}Sr_{0.3}MnO_3$) в диапазоне частот 10–40 ГГц и 60–78 ГГц. Обнаружена величина поглощения –7.5 дБ при 32–35 ГГц для композита с заполнением 60 % и толщиной слоя 0.53 мм. Анализ полученных результатов доказывает высокую однородность уровня распределения в матрице полимера магнитных наночастиц. Обсуждаются наиболее вероятные причины необычно больших значений фактора спектроскопического расщепления, полученного при помощи Электронного Спинового Резонанса. Представлены результаты исследований по отработке технологии и методики изготовления ферромагнитных композитов, а также эпоксидно-полисилокановых полимерных матриц.

1. Introduction

Magnetic composites with polymer host matrix are considered now as the most promising materials [1–4] among variety of microwave absorbers. These composites are advantageous over bulk magnetic materials because of high resistivity, low density, high mechanical properties, chemical stability and machineability. Magnetic properties of composites may be easily varied by changing the concentration of magnetic inclusions, by mechanical treatment of the inclusions, etc. For good microwave absorbing performance, one needs a material with high magnitude of permeability, high magnetic loss, a remarkable type of frequency dependence of permeability, and a proper ratio between the permeability and permittivity. A number of publications report on

studies of microwave properties of composites comprising various magnetic powders imbedded into a polymer-host matrix [1–4]. Nanosized magnetic particles are believed to open new opportunities for developing composites with high microwave permeability and good absorbing performance, see e.g., [5]. Particularly, an evident advantage of nanosized inclusions is the negligible small effect of eddy currents. Today one of the promising candidates for magnetic inclusions is nanosized particles of doped manganites [6–10].

Magnetic composites based on polymers [11–14] are being considered as prospective absorbing coatings. The possibilities of wide selection of matrix material and magnetic/nonmagnetic impurities as well belong to their main advantages. Thus it is expected that such materials should combine advantages of polymers with the advantages of the functional properties of the composites.

Doped manganites are among the most promising components of the magnetic nanocomposites. Moreover they can be used as "left-handed" metamaterials electromagnetic properties which are relatively easy to vary by the external magnetic field [15–17].

This article presents the results of the study of manufacturing technology of magnetic composites based on nanosized powders of manganite-perovskite ($La_{0.7}Sr_{0.3}MnO_3$) and epoxide polymer polymers. The results of research of electron spin resonance (ESR) and nonresonant absorption of electromagnetic waves in the nanocomposite layered specimens ($La_{0.7}Sr_{0.3}MnO_3$) in the frequency band 10–40 GHz and 60–78 GHz are analyzed as well.

2. Samples

2.1. Manganite (La,Sr)MnO_3 nanopowder synthesis

Powders of lanthanum manganites doped by strontium — $La_{1-x}Sr_xMnO_3$, (LSMO) — were synthesized by co-precipitation method supplemented by microwave heating and ultrasound treatment. All chemicals that were used in the synthesis procedure were reagent grade. A nanometer scaled $La_{1-x}Sr_xMnO_3$ powder was obtained directly by annealing the solution at 900°C for 60 min. The precipitate was then washed several times in order to remove the by-products of the reaction. The hydrogel was dried in thermo-box. The powders were dried in the pulsed magnetic field at 120°C in a special setup (for more details see [10, 11].) The Curie temperature measurements shown that the sample with $x = 0.25$ has too small T_C. However the sample with $x = 0.35$ gives large enough Curie temperature of powder $La_{0.7}Sr_{0.3}MnO_3$ with particles size of about 50 nm was about 320 K.

2.2. Polymer host-matrix selection

Epoxy resins and composites based on them have a number of properties which provide possibility of their usage as a polymer matrix. Different classes of compounds can be hardeners of such systems: anhydrides of carboxylic acids, different types amines, Lewis's acid, etc. Considering necessity of reception of high degree filling as a basis of polymer component of composite have been choosen low-viscosity epoxy oligomer, which does not contain aromatic cycles in the structure, EPONEX 1510 (the hydrobathing analogue epoxy resin ED-22). Epoxy matrix formation was carried out with using of amine curing agent Jeffamine T403.

According to thermal mechanical analysis the received polymer is characterized by high crosslinking density n_ν, that, in turn, provides high magnitude of its elasticity module in highly elastic state, and glass state at enough high temperature: glass transition temperature T_g and elasticity temperatures T_e are 63°C and 77°C, accordingly. The data of gasovolumometric researches confirms high resisitance of a composite polymer matrix concerning thermooxidizing degradation. Speed of absorption of molecular oxygen at temperature 180°C is 3.3 mole·kg^{-1}·sec^{-1}.

Filling from 20 to 60 volume percents of manganite in such polymer practically does not influence on thermophysical properties of the polymer matrix: glass transition temperature irregularly changes within 53÷65°C with tendency of insignificant fall. It gives the grounds to assume that nanofiller does not render essential influence on course of polycondensation process and on crosslinking density of the polymer matrix.

Thus, the chosen system based on epoxy oligomer EPONEX 1510 and amine hardener Jeffamine T403 has a complex of rheological, technological and thermal properties, which provides obtaining of composites with the high maintenance of magnetic nanoparticles.

2.3. Structure and chemical elements composition of the layered samples

To make quantitative analysis of the structure and chemical composition analysis of the sample X-ray energy dispersive spec-

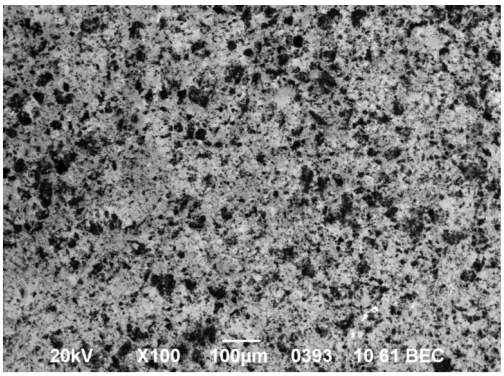

Fig. 1. Scanning electron microscopy image for the film with 60 % $La_{0.7}Sr_{0.3}MnO_3$ nanoparticles filling; the magnification equals 100.

troscopy (EDS) using INCAPenta-FETx3 spectrometer and JSM-6490LV scanning electron microscopy (SEM) have been applied. The SEM image of the layer having 60 % $La_{0.7}Sr_{0.3}MnO_3$ nanoparticles filling is shown in Fig. 1. A quite high level homogeneity of magnetic nanoparticles distribution in polymer matrix is directly visible. The chemical composition analysis reveals a presence of small amount (~0.01 %) of Ni ions in the 60 % $La_{0.7}Sr_{0.3}MnO_3$ layered sample.

3. Experimental technique

Experimental detecting non-resonant absorption in the sample was carried out using Vector Newtork Analyzer Agilent PNA-L N5230A. Transmission spectra of the studied sample in 22–40 GHz band were under analysis. Also the spatial distribution of the spectra structure were obtained at temperature T = 300 K.

The specially designed experimental module for ESR measurements (Fig. 2) has been applied for research at T = 300 K. The module possess several principal features which distinct it from typical spectrometers significantly. Namely, the module has a kit of volumetric resonator experimental cells (Fig. 2), which provide a) — transmitting regime and b) — angular regime of the spectra detecting. In order to realize regimes mentioned, the module is adjusted with Agilent PNA-L N5230A. Thus the realization of two principle operating modes for this magnetic resonance spectrometer was ensured. These modes are: 1) scanning the frequency at the fixed magnetic field, and 2) scanning the magnetic field at the fixed frequency (the traditional one). The resonator is placed in the gap of the electromagnet providing with that mutually perpendicular orientation of vector of a permanent magnetic field and the vector of an alternative magnetic field. The resonators were provided with a movable piston for the resonator frequency setup and the goniometer. As a result a possibility to move the sample along the longitudinal axis of the resonator and the sample rotation are realized.

The cryomagnetic radiospectrometer BURAN [20] was applied for carrying out low-temperature experiments.

4. Results and discussion

The results of analysis of the magnetic resonance absorption in 60–78 GHz (at T = 4.2 K) and 10 GHz frequency bands (at T = 300 K) in $La_{0.7}Sr_{0.3}MnO_3$ sample and for the concentration of manganite equals to 60 % are given below.

4.1. Non-resonant absorption

The typical transmission spectra are presented in Fig. 3. The sample with the size of 50×30×0.53 mm^3 was located in the rectangular waveguide 7.2×3.4 mm^2 (between the waveguide flanges). For the purpose to investigate the spatial uniformity such flat sample, the latest was moved perpendicularly to the waveguide axis with a certain step of offset: d(mm) = 0; 5; 10; 20. It was found that such offset does not change practically the frequency spectra of trans-

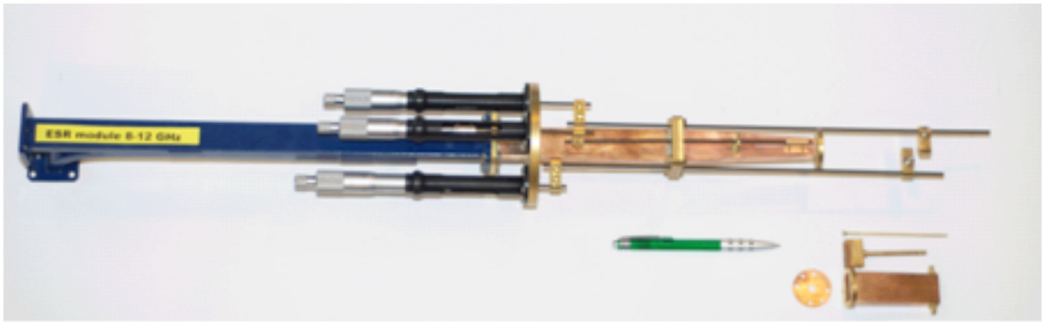

Fig. 2. ESR module with a tunable resonator for research in 7–12 GHz band.

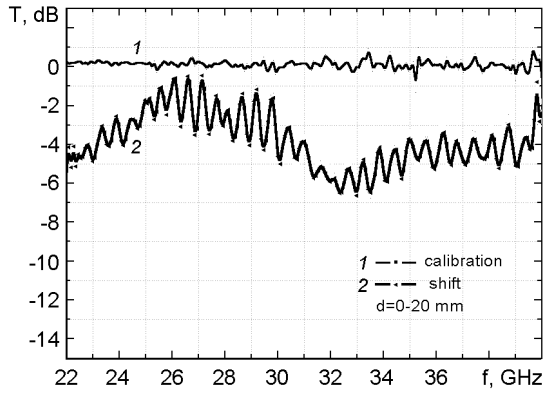

Fig. 3. Microwave transmission through a sample. The averaging of 4 kit of data for different values of waveguide channel offsets d = 0; 5; 10; 20 mm.

mission coefficient. A little difference between dependences lies within an error of experiment of ±0.5 dB. Note that such weak frequency dependence of the material transmittance proves a very homogeneous distribution of clusters in a manganite sample volume. The absence of resonances in the spectrum suggests the idea, that gaps between the clusters in the sample and/or cluster sizes are much smaller than $\lambda/2\cdot(\varepsilon)^{1/2}$ magnitude where λ is the wavelength, and ε is the permittivity of a sample.

A certain small non-homogeneity of spectrum in Fig. 3 (which manifests itself as the presence of oscillations with frequency of ~0.5 GHz) is a consequence of non-ideal matching of the sample with the waveguide line as well as by the presence of gaps between the sample surface and the plane of the waveguide open end.

Besides the tendency of growth of the averaged (over each scan) transmission coefficient with the frequency increasing is observed. This indicates the dielectric character of the sample properties, i.e. the absence of metallic inclusions. This assumption is confirmed as well by the study of magnetoresonance absorption in the sample.

Fig. 3 shows that in a composite of 60 % concentration of manganite the absorption reaches the value of −7.5 dB in the 32–35 GHz band (for the sample thickness of a 0.53 mm). We expect that according to existing literature data [21–25], increasing of the manganite content up to 80 % allow to receive the absorption more than 90 %.

4.2. Magnetoresonance absorption in 10–40 GHz band (T = 300 K)

In order to research the magnetic phase states the study of magnetic resonance absorption of waves 10–40 GHz band was carried out.

The results of ESR absorption for the "parallel" geometry of the experiment (the vectors external magnetic field and the magnetic component of microwave field lie in the plane of the thin-plate sample and directed mutually perpendicular) are shown in Fig. 4(a). The results for the "perpendicular" geometry (the vector of magnetic component of microwave field lies in the sample plane and the vector magnetic field directed perpendicularly to the sample plane) are presented in Fig. 4(b).

The Electron Spin Resonance line width ($\Delta H_{1/2}$) does not depend on frequency and on orientation of the sample relatively the external magnetic field and it equals $\Delta H_{1/2} \approx 650$ Oe. The inserts in (Fig.4(a),

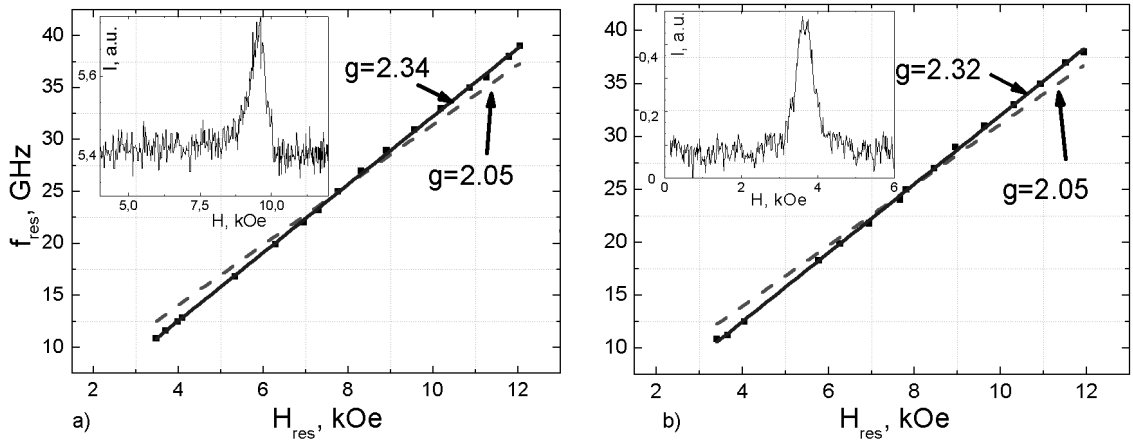

Fig. 4. Resonant frequency/field dependence for the sample with concentration of manganite of 60 %, $T = 300$ K: (a) "*parallel*" *orientation (insert: the ESR-lineshape at* f = 31.00 GHz); (b) "perpendicular" orientation (insert: the lines hape at f = 11.21 GHz).

Table. The results of approximation of the experimental data for g-factor at T = 300 K

"Parallel" orientation		"Perpendicular" orientation	
g = 2.05	M_s = 157 Gs (±10 Gs)	g = 2.05	M_s = −101 Gs (±10 Gs)
g = 2.32±0.01	M_s = −27 Gs (±25 Gs)	g = 2.32±0.01	M_s = 19 Gs (±25 Gs)

(b)) show the typical ESR line in the composite for corresponding geometries of experiment at T = 300 K. Note that the shape of the line is intermediate between Lorentzian and Gaussian ones, but much closely to Lorentzian. This fact indicates that there is no any significant anisotropy in the nanocrystalline, however more than one type of magnetic interactions [17] are presents.

As a result of analysis of the experimental data the resonance frequency-field dependencies for both orientations of applied magnetic field relative to the plane of sample were constructed (Fig.4(a) and (b)). In order to determine the effective magnetization of the sample the experimental curves were approximated by the known Kittel's formula for "parallel" (1) and "perpendicular" (2) geometry of experimental [26]:

$$\nu_{res} = \frac{g\mu_\beta}{h}\sqrt{H_{res}(H_{res} + 4\pi M_{eff}}, \quad (1)$$

$$\nu_{res} = \frac{g\mu_\beta}{h}(H_{res} - 4\pi M_{eff}\}, \quad (2)$$

where ν_{res} is resonance frequency, g is spectroscopic splitting factor, μ_β is Bohr magneton, h is Plank constant, H_{res} is resonant magnetic field. The effective magnetization M_{eff} [28] produces the so-called "effective demagnetizing field" $4\pi M_{eff} = H_a + 4\pi M_s$, where H_a is some anisotropy field. M_s is saturation magnetization, which equals M_{eff} in the absence of anisotropy fields. Note that this approach allows count the non-flat shape of the manganite granules; as well as possible magnetostatic interaction and anisotropy fields.

If to perform the approximation of experimental data by the standard way g-factor (the spectroscopic splitting factor) should be chosen by g = 2.05, i.e. close to value of g-factor for typical ferromagnet. However, after calculations using (1), (2) — (see Table) at such assumption one obtains that M_s for "perpendicular" orientation appears a negative, that contradicts with the physical concepts of magnetization [27]. Let remain that as it mentioned above there is no reasons to introduce any noticable anisotropy field, so H_a.

To solve this contradiction let's note that g-factor obtained experimentally (an inclination of the approximating line on resonant frequency/field dependence, Fig. 4) equals g = 2.34. This value differs essentially from the value of g-factor (an inclination of the approximating line of resonant frequency/field dependence) for typical ferromagnet is g = 2.05. Let's note that an accuracy of the experiment is high enough to claim that experimental errors do not change out comes. Namely the error for g-factor definition is $\Delta g < 1-2$ % and for magnetizations of saturation is $\delta M_s < \pm 10-25$ Gs.

Experimentally obtained magnitude g = 2.32 testifies to an existence of certain additional sources [29] of magnetism in this system. Such source can be particles of Ni, which remains in the nanomagnet composite due to the technology process.

This assumption is closely to reality. In fact, the value of M_s, obtained in this suggestion, is close to zero within the error of measurement (Table). The anisotropy field is also equals to zero. It seems to be quite natural, since the only source of the anisotropy field at T = 300 K may be internal stresses, which is excluded under the described above technology manufacturing sample. In such a case the most probable explanation of the anomalously large g-factor value can be given in frames of classical concepts of magnetism [30]. The given sample can be considered as magnetic with quite strong spin-orbit interaction. The value of g-factor is very sensitive to the ligand environment of the ion, which forms a crystalline field. For such ions g-factor is defined [30] as $g = 2*(1 - \xi* \cdot \Delta)$, where ξ is the constant of spin-orbital interaction and Δ is the magnitude of so-called "ligand field splitting". It is well known that for free radicals the magnitude of Δ can be very large, and the magnitude of ξ becomes small and negative. Therefore for such systems g-factor is close to that for a free electron and changes within the third sign after a comma. However in our case for manganite-perovskite

nanocomposite the magnitude of ξ can be substantially higher, because the magnitude of spin-orbital interaction for manganites-perovskites is much more than for free radicals. Therefore the value of *g*-factor should exceed standard *g*-factor for electron.

Really, the most probable is that presence of Ni in this composite can exceed the value of spin-orbital interaction ξ. It also leads to that the detected value *g*-factor appears almost 12 % greater than *g*-factor for a free electron.

4.3. Magnetoresonance absorption in 60–78 GHz band (T = 4.2 K).

To learn the temperature influence on the magnetic phase state of the sample the magnetic resonance absorption in 4 mm waveband has been analyzed at $T = 4.2$ K. The resonant frequency/field dependences are given in Fig. 5. A typical ESR absorption lineshape is given in (Fig. 5 insert). Let's note that the symmetric lineshape indicates on the uniformity of magnetic system.

As it is known [27] manganite-perovskite under study is a very complicated magnetic system with a lot of various combinations of intrinsic interactions (including dipole-dipole, double exchange ones, etc.). This composite transforms into a collinear magnetic state with temperature decreasing and appears in a ferromagnetic phase at $T = 4.2$ K. This proved indirectly by the fact that measured value of *g*-factor $g = 2.05$ is close to *g*-factor for typical ferromagnet. It is possible to estimate (with the help of (1)–(2)) the magnitude of magnetization of saturation of $M_s = 119$ Gs (±20 Gs), taking into account that the anisotropy field caused by thermal shrinkage of polymer matrix lies in the sample plane.

Note that for low-temperature experiments, the resulting line magnetic resonance absorption becomes wider ($\Delta H_{1/2} \approx 3.5$ kOe) in comparison with linewidth at $T = 300$ K ($\Delta H_{1/2} \approx 650$ Oe) — (see Fig. 5 insert). The broadening of the line at $T = 4.2$ K most probable [31] associated with the transition into some sort of weak collinear magnetic phase. It is evident that as the temperature is lowered, so the local fields play more important role. This causes the disordering of magnetic system of the sample. More detailed this process is described in [31]. However, the fact that magnetization M_{eff} has final (nonzero) value, testifies to the asperomagnetic character of this magnetic phase.

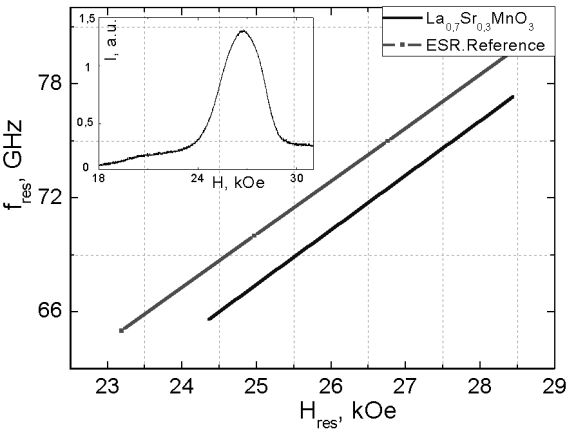

Fig. 5. Resonance frequency/field dependence for "perpendicular" orientation at $T = 4.2$ K (insert: the ESR-line shape at $f = 74.00$ GHz).

5. Conclusions

The method of manufacturing of microwave absorbing materials has been designed and composites with different content of manganite have been prepared by mixing $La_{0.7}Sr_{0.3}MnO_3$ nanoparticles and polymer compound. The technology of production prevents magnetic nanoparticles conglomeration and yields homogeneous distribution of magnetic nanoparticles in polymer matrix.

The analysis of the non-resonant electromagnetic wave absorption in frequency range 22–40 GHz proves the high homogeneity of magnetic particles distribution. The established trend of improving transmission coefficient with frequency increasing indicates the absence of regions with metallic conductivity in the sample under study.

Research of electron spin resonance absorption of electromagnetic waves of 10–40 GHz wavelength (at $T = 300$ K) and 60–78 GHz wavelength (at $T = 4.2$ K) in the magnetic nanocomposite ($La_{0.7}Sr_{0.3}MnO_3$) was performed. It was determined that *g*-factor became anomalously large, that took place most likely due to the presence of Ni in the nanocomposite. While temperature decreases up to helium ones the broadening of the magnetic resonance line is observed. This occurs due to disordering the electron spin system of the sample and corresponds most likely to the transition of the magnetic phase of the sample from strongly collinear into weakly collinear magnetic phase.

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Особливості поглинання електромагнітних хвиль у полімерних магнітних нанокомпозитах (La,Sr)MnO_3

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Наведено результати досліджень особливостей нерезонансного і магніторезонансного поглинання електромагнітних хвиль у полімерних магнітних нанокомпозитах ($La_{0.7}Sr_{0.3}MnO_3$) в діапазоні частот 10–40 ГГц і 60–78 ГГц. Виявлено величину поглинання –7.5 дБ при 32–35 ГГц для композиту із заповненням 60 % і товщиною плівки 0.53 мм. Аналіз отриманих результатів доводить високу однорідність рівня розподілу у матриці полімеру магнітних наночасток. Обговорюється найбільш вірогідні причини незвично великих значень фактора спектроскопічного розщеплювання, отриманого за допомогою Електронного Спінового Резонансу. Представлено результати досліджень з оброблення технології і методики виготовлення феромагнітних композитів, а також епоксидно-полісилоканових полімерних матриць.