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Transport and magnetic resonance characteristics of SiO₂- γ -Fe₂O₃ composites obtained by sol gel method

Abstract. Some electron-transportation and magnetic-resonance characteristics of SiO₂- γ -Fe₂O₃ nanocomposites obtained by sol gel method on dielectric substrates were studied. It is shown that the current-voltage characteristics of the film composite materials are non-linear which is due to the presence of ionic component in the charge transfer mechanism. An increase of the measurement temperature causes their linearization. There were superwide magnetic resonance lines found, the presence of which in the ESR-spectra of the samples is due to the ferromagnetic phase of the composite material.

Streszczenie. Zbadano właściwości przewodnictwa elektrycznego i rezonansu magnetycznego nanokompozytów SiO₂- γ -Fe₂O₃ otrzymywanych metodą żelowania koloidalnego na podłożu dielektrycznym. Wykazano że charakterystyka prądowo-napięciowa jest nieliniowa na skutek obecności składowych jonowych. Przy wzroście temperatury następował efekt linearyzacji. Otrzymano szerokie linie rezonansu magnetycznego, których obecność wskazuje na istnienie składowej ferromagnetycznej w materiale kompozytowym. (Właściwości przewodnictwa elektrycznego i rezonansu magnetycznego nanokompozytów SiO₂- γ -Fe₂O₃ otrzymywanych metodą żelowania koloidalnego).

Słowa kluczowe: nanokompozyty, cienkie warstwy, żele.

Keywords: nanocomposite, ferromagnetism, sol gel method, charge transfer, film.

Introduction

A great interest towards to nanostructured composition materials, being either isolating or conducting matrixes with magnetic active nanoclusters, is mostly due to their possible applications in spintronics [1]. However, a stable concept of physics of the processes and the electronic states has not been formed yet. This demands both theoretical and experimental research of the properties of like-systems, obtained by different methods.

Experimental

The films of nanocomposites obtained by SiO₂ and Fe₃O₄ (8 mass %) sols mixing and depositing on dielectric substrates with their further thermal treatment in the temperature range of 373 – 723 K. The width of the obtained samples was in the range of 100 – 200 nm. These nanocomposites are characterized by high heat stability of γ -Fe₂O₃ phase, the size of the particle in which does not exceed 5-7 nm after heating at 1173 K.

The transport characteristics of the SiO₂- γ -Fe₂O₃ films on dielectric substrates with platinum contacts of interdigital geometry spaced by 0.2 mm were studied in the temperature range of 300 – 400 K. Voltage-current characteristics (VCC) and the resistance temperature dependencies were studied in the DC-regime in the electric fields magnitude range up to 250 V/cm. The studies of the magnetic resonance properties were carried out using the immovable «Varian E-112» ESR-spectrometer in the X-range (9.3 GHz). The annealing of the samples was realized directly within the microwave resonator of the spectrometer in the temperature range 293 – 403 K.

Results and discussion

The voltage-current characteristics of two SiO₂ films of different thickness measured at room temperature are shown in Figure 1. In spite of low thickness, their VCC is linear within the studied electric field range. The resistance values grew up to several GOhm depending on the number of the layers forming the film itself. Introduction of γ -Fe₂O₃ to the interstice of SiO₂ causes a significant (by several orders of magnitude) decrease of the resistance. At that the VCC of the film nanocomposites become ultralinear even in weak electric fields and the voltage of 1 V. Figure 2 presents some typical for the majority of the samples VCC measured at room temperature.

The current instability is another characteristic feature of the VCC of the samples in question. So, an application of a voltage “stage” up to 3 V causes current increase and further stabilization. Higher voltage application causes the current increase and its further stabilization. The arrows point this out in figure 1. This current instability with voltage increase may be due to the Joule heat in the samples.

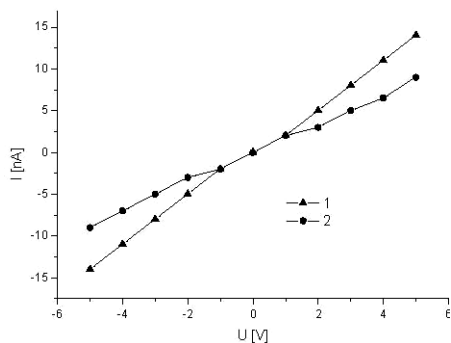


Fig. 1. Voltage-current characteristics of two films with different thickness (T=293 K)

To confirm this assumption there were some further VCC measurements at different temperatures conducted. It was found that scaling the temperature causes fewer changes in the current values when voltage “stage” is applied. The temperature range of 323 – 343 K is a transition one and at 353 K the VCC becomes linear and stable. The VCC of the same sample at 353 K is shown in figure 3. It should be noted that the resistance of the samples may increase by several orders of magnitude at that.

There was the resistance dependence on temperature studied. It was found that a slight increase of the resistance and its' further decrease occurs as the temperature increases up to 373 K. There was a hysteresis found of the resistance temperature dependence. In the process of the samples cooling the value of the resistance could increase 6 – 10 times compared to the initial one, which is in a good agreement to the current increase as the voltage “stage” of the magnitude higher than some critical is applied.

The distinguished features of the transport properties of nanocomposites in study are characteristics of the systems being electrochemical capacitors [2]. The OH-groups presence may be a reason for the ionic component of the conductivity as well as for the long-term charge accumulation at the interface. The measurements of the potential of the platinum contacts after the voltage release made it possible to register the values of the voltage of about tens of mV. Some additional measurements of VCC in the dried by silica gel atmosphere and the VCC of the films covered by a chemical-resistant varnish showed the expected stabilization and linearization of the voltage current character.

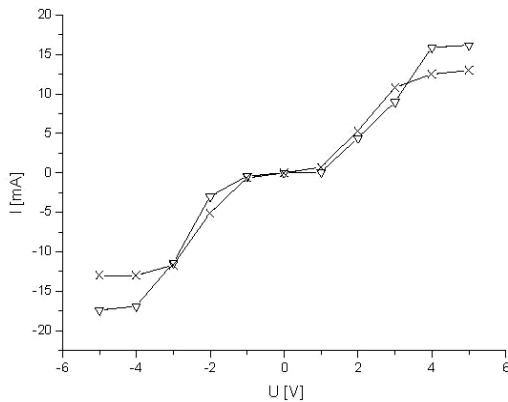


Fig. 2. Voltage-current characteristics of the $\text{SiO}_2\text{-}\gamma\text{-Fe}_2\text{O}_3$ composite at room temperature

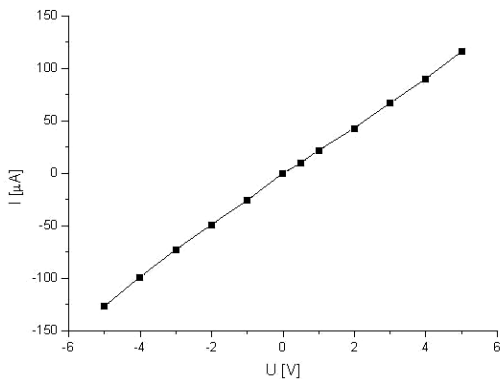


Fig. 3. Voltage-current characteristics of the $\text{SiO}_2\text{-}\gamma\text{-Fe}_2\text{O}_3$ composite at 353 K

The intensive ultrawide ESR signal ($\Delta H = 970$ Oe) with g-value of 2.00 is a characteristic of the studied samples both at room temperature and the temperature of liquid nitrogen. Fig. 4 shows some typical spectra of the samples in study. One can see that both the position and the width of the resonance line depend on the angle between the applied magnetic-field vector and the samples' surface normal. The angular dependence of the signal and its width testify to the ferromagnetic resonance observation [3].

It was found that the intensity of the resonance lines of the samples with different $\gamma\text{-Fe}_2\text{O}_3$ concentration increases without saturation as the applied microwave power increases, which is specific for the homogeneous character of the line broadening and magnetic particles distribution within the dielectric matrixes. At that the magnetic resonance line width depends on the angular position of the sample in the resonator of the spectrometer.

The area under curve increased proportionally to the increase of the magnetic nanoparticles concentration in the sample. One can see from figure 5 that the rotation of the sample by 90. about its axis in the resonator results in the shift of the signal position in the magnetic field by 300 Oe, which denotes the magnitude of the magnetic field that should be applied to change the direction of magnetization of the nanoparticles.

The temperature dependencies of the ferromagnetic resonance parameters of the films in study are of a particular interest in the range of 293 – 403 K. They show the ESR-line width non-monotone behavior both while heating and cooling. At the same time the position of the resonance line in the spectrum does not change and its intensity undergoes a linear increase up to 403 K. This results are in agreement with the results of the electric studies and are probably due to the presence of the water molecules dissociated in the substrate which is a subject of further studies.

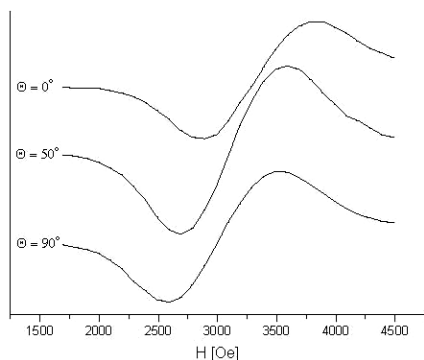


Fig. 4. The magnetic resonance spectra of the sample with 8 mass % concentration of $\gamma\text{-Fe}_2\text{O}_3$. θ – is an angle between the applied magnetic-field vector and the samples' surface normal. $T = 293\text{ K}$

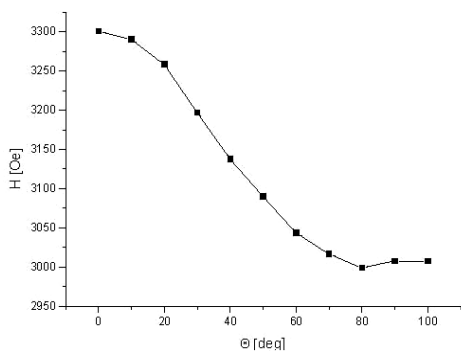


Fig. 5. The ferromagnetic resonance line position in the magnetic field versus the angle between the applied magnetic-field vector and the samples' surface normal. $T = 293\text{ K}$

Conclusion

It was estimated that the electron-transport characteristics of the $\text{SiO}_2\text{-}\gamma\text{-Fe}_2\text{O}_3$ film nanocomposites are nonlinear at room temperature, which is due to the presence of OH-groups in the interstice of the samples. The results of the magnetic resonance studies testify to the presence of ferromagnetic phase in the samples in question. The character of the angular dependence of the ESR-resonance signal suggests the form of the magnetic nanoparticles being close to spherical one.