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MAGNETIC AND DIELECTRIC PROPERTIES OF $Ni_{0.5}Zn_{0.4}Cu_{0.1}Fe_2O_4$ AND $CoFe_2O_4$ NANOCOMPOSITES INCORPORATED WITH PMMA POLYMERIC MATRIX

Abstract. Magnetic and Dielectric properties of $Ni_{0.5}Zn_{0.4}Cu_{0.1}Fe_2O_4$ and $CoFe_2O_4$ doped PMMA polymeric matrixes were investigated by VSM and dielectric spectroscopy analysis. They exhibit hysteretic behavior at room temperature with a rounded shape characteristic for superparamagnetic particles. The coercive fields are decreased significantly with increase of complex ferrite concentration. At low temperatures, *M–H* loops dramatically changes, especially for intermediate ferrite concentrations and attributed to the formation for two types of cluster from different materials due to concentrations of complex and cobalt ferrites. Dielectric strength and activation energies of cobalt ferrite and complex ferrite doped insulator PMMA were acquired throughout the performed analysis and the best contributing composition, in terms of the electrical peculiarity, was also optimized in the scope of the work. Equivalently doped cobalt and complex ferrite demonstrated compatible behaviors to PMMA in all measured temperature ranges according to obtained results. It is also observed that the contribution of cobalt ferrite to the conductivity is more effective whereas complex ferrite formed cohesively stronger structure in the matrixes in all temperatures.

The increase in the electrical conductivity as temperature increases is related to the increase in drift mobility of the thermally activated charge carriers in accordance with the hopping conduction mechanism. The variation of AC conductivity as a function of temperature represents almost a straight line by changing its slope at the curie temperature T_c and thereafter a break occurs indicating a change of magnetic ordering from ferromagnetism to paramagnetism. The values of activation energy in paramagnetic region are found to be greater than those in ferrimagnetic region, which suggests that the process of conduction is affected by the change in magnetic ordering.

Keywords: ferritic semiconductor, complex ferrite, dielectric spectroscopy, relaxation time, nanocomposite

Тірек сөздер: спектроскоп, ферриттер, температура, заряд, талдау, күш, энергия.

Ключевые слова: спектроскоп, ферриты, температура, заряд, анализ, сила, энергия.

1. Introduction. Complex ferrite systems are quite useful since they promise many technological applications ranging from microwave to radio frequencies [1]. Among these applications radar absorbent materials for stealth technology can be mentioned [2]. Nanocrystalline spinel ferrites can be used in many areas, such as magnetic devices, switching devices, recording tapes, permanent magnets, hard disc recording media, flexible recording media, read-write heads, active components of ferrofluids, color imaging, magnetic refrigeration, detoxification of biological fluids, magnetically controlled transport of anti cancer drugs, magnetic resonance imaging (MRI) contrast enhancement and magnetic cell separation, radar absorption etc. [2-6].

The dielectric properties of ferrites depend on many factors, including the preparation method. Ferrites have quite low conductivity and this property is useful for microwave applications. The dielectric and magnetic behaviors of ferrites are greatly influenced by the conductivity [7]. Hence, it is quite convenient to study the dielectric properties of these materials in different frequencies and temperatures.

Spinel structure consists of a cubic close-packed array of oxygen atoms with tetrahedral A-site and octahedral B-site [8]. Cobalt ferrite is preferred due to its interesting magnetic and electrical properties such as strong anisotropy, high coercivity at room temperature, moderate saturation magnetization, high DC electrical resistivity, low dielectric losses, and good mechanical and chemical stability [3].

The method of preparation and the type of substitution play a decisive role on the physical properties of the Ni–Zn ferrite [9]. Various methods such as co-precipitation, the reverse micelle method, microwave plasma synthesis, sol-gel method, ultrasound irradiation, freeze drying, thermal decomposition of organo-metallic and coordination compounds, hydrothermal method and microwave assisted route have been reported to obtain nanocomposites [3,4,10].

So-called hopping semiconductor type mechanisms are more predominant for these types of ferrite groups and the conduction process is caused by hopping of thermally activated electrons from one cation to another [11].

In this paper we aimed to present the magnetic, electrical and dielectric properties of polymeric $Ni_{0,5}Zn_{0,4}Cu_{0,1}Fe_2O_4$ and $CoFe_2O_4$ composites such as ε' and σ' of different composition ratio as a function of frequency and temperature.

2. Experimental. Ferrite nanoparticles with the composition $Ni_{0.5}Zn_{0.4}Cu_{0.1}Fe_2O_4$ and $CoFe_2O_4$ were synthesized by the co-precipitation method and incorporated in PMMA polymeric matrix and X-ray powder diffraction was used to determine the crystalline phases [12].

Table 1 – Cobalt ferrite and complex ferrite ratios of the samples

NAME	Complex ferrites	Cobalt ferrites
3A	0	4.5
3B	1.125	3.375
3C	2.25	2.25
3D	3.375	1.125
3E	4.5	0

Magnetization measurements were performed by using a Quantum Design Vibrating sample magnetometer (QD-VSM). The sample was measured between ±10 kOe at room temperature and 50K. ZFC (zero field cooling) and FC (field cooling) measurements were carried out at 50 Oe and the blocking temperature was determined from the measurements.

The dielectric properties of the polymer nanocomposites containing different concentrations of ferrites (see table 1) have been investigated by Novocontrol Alpha-N High Resolution dielectric analyzer. Measurements were carried out in a frequency range of 1Hz-1MHz at a temperature increment of 20°C from -40°C to 150°C under rms 1V AC.

3. Results and Discussion

a) Magnetic Measurements

Hysteresis loops of the ferrite composites, obtained by using a Quantum Design PPMS VSM magnetometer, are presented in figure 1 for 300 K and in figure 2 for 50 K. All samples exhibit hysteretic behavior at room temperature with a rounded shape characteristic for superparamagnetic particles.

However, their coercive fields are decreased significantly with increase of complex ferrite concentration. At 50 K, the recorded *M–H* loops dramatically changes, especially for intermediate ferrite concentrations. Since these samples have both concentrations of complex and cobalt ferrites, one may expect the formation for two types of cluster from different materials. Indeed, the steps in the hysteresis loops of samples 3B, 3C and 3D are a clear sign for a two-component system. In addition, it is clear from the hysteresis curves that clusters of cobalt ferrites have a larger coercivity while complex ferrites have smaller one. With decrease of cobalt ferrite concentration, their contribution to the hysteresis curves decreases and thus coercivity in two-step loops decrease. The samples 3A and 3E contain one type of ferrite, cobalt ferrite for 3A and complex ferrite for 3E, respectively. For this reason, they exhibit characteristic hysteresis loop for one phase.

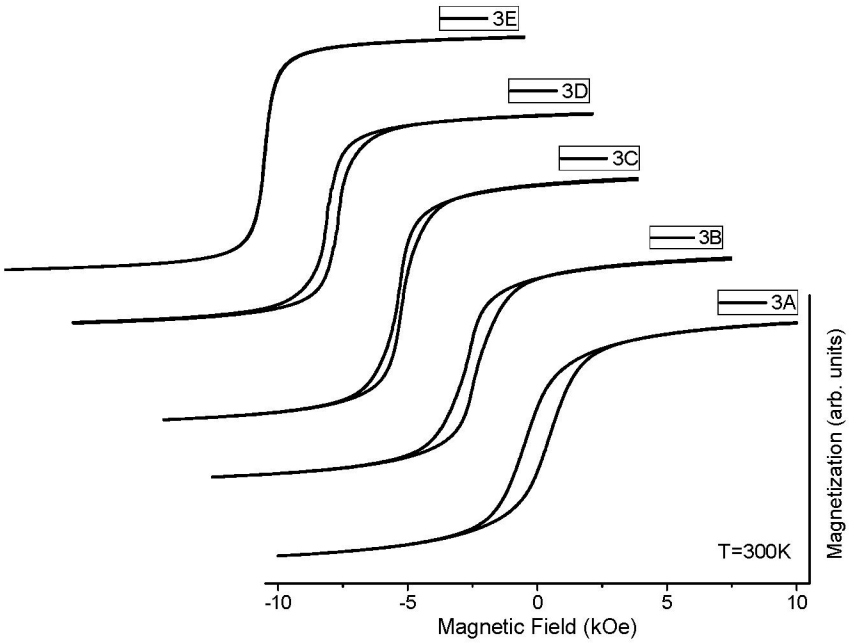


Figure 1 – Room temperature hysteresis curves of the composite samples

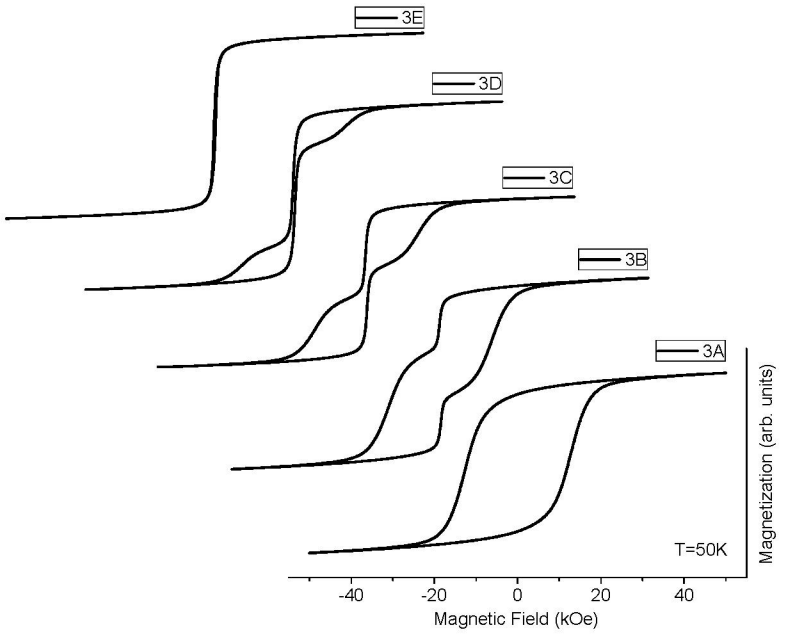


Figure 2 – *M-H* loops of the samples measured at 50 K

Figure 3 presents the saturation magnetization for room temperature and 50 K, which is plotted as a function ferrite concentration. The highest magnetic moment is observed for sample D, which contains %75 complex and %25 cobalt ferrites.

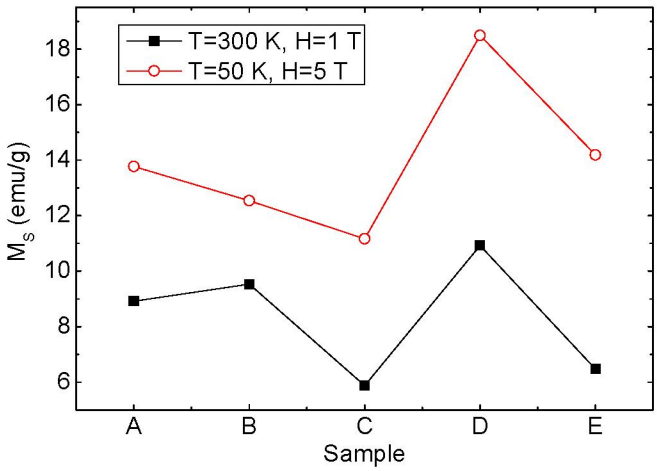
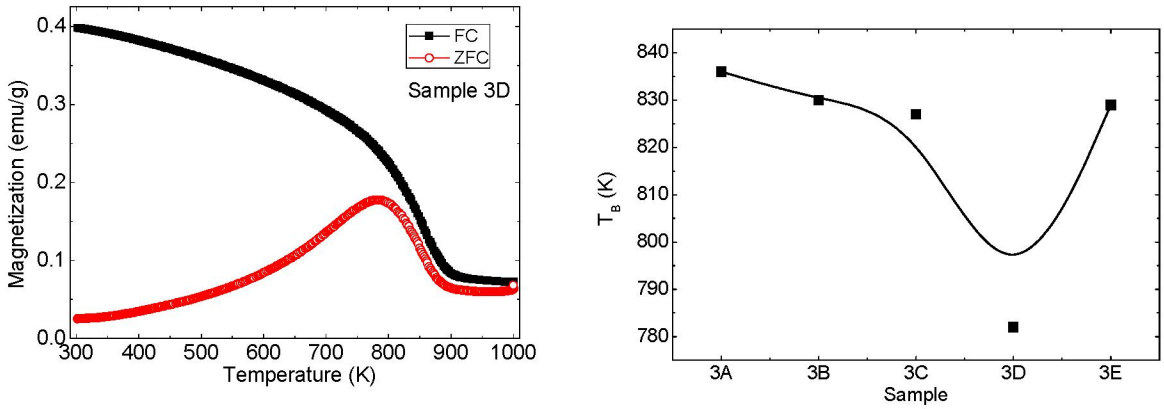


Figure 3 – The saturation magnetization is plotted as a function ferrite concentration. The data were taken at RT ant 50 K using VSM magnetometry

In order to further investigate the magnetic properties, we have performed temperature dependent magnetization (*M–T)* measurements using a VSM magnetometer. Figure 4a presents field cooled (FC) and zero field cooled (ZFC) plots for sample 3D. For ZFC measurements, the samples are heated up to 1000 K. Then, they are cooled in zero field to 300 K and the magnetization is recorded during warming up to 1000 K in an applied field of 50 Oe. For FC measurements the applied field of 50 Oe is kept constant during cooling from 1000 K to 300 K and the magnetization is recorded during field warming within the same field value. The FC and ZFC curves diverge substantially for all samples. This behavior is not expected for a ferromagnet and suggests the presence of magnetic nanoparticles in the samples. The peak in the ZFC curves (shows the blocking temperature) progressively shifts to lower temperatures with increase of complex ferrite concentration (Fig. 4b). For sample E, in which the complex ferrite concentration reaches maximum and the cobalt ferrite concentration is 0, the T_B increases again.



(a) (b)

Figure 4 – (a) FC and ZFC curves refer to field cooled and zero-field cooled protocols and are presented by closed and open symbols, respectively. In both cases the data were taken in a field of 50 Oe during the heating up cycle. (b) The blocking temperature (T_B) for different samples

b) Dielectric Measurements

Temperature dependent dielectric spectroscopy (DS) is employed for acquiring information about the electrical behaviors of ferrite doped polymer samples of various ratios. The complex dielectric permittivity is given as, $\varepsilon = \varepsilon' - i\varepsilon''$, where ε' is the real part, and ε'' is the imaginary part of the dielectric constant. The real part of the dielectric constant was calculated from the equation $\varepsilon' = C_p d/(\varepsilon_0 A)$, where C_p is the parallel capacitance, d is the inter-electrodes distance, ε_0 is the permittivity of free space and A is the plate area. Our main fictions in these measurements are focused primarily on the peculiarity of real dielectric constant, ε', of PMMA composite. It is clearly seen from fig.5 that the real part of the dielectric permittivity decreases as frequency increases showing usual dielectric dispersion which is due to Maxwell-Wagner type interfacial polarization. The decrease is quite rapid at low frequencies and becomes quite slow at higher frequencies. The decrease ε' takes place when the jumping frequency of electric charge carriers cannot follow the alternation of the applied electric field beyond a certain critical frequency.

The origin of ε' can be explained on the basis of Maxwell–Wagner theory [13,14] which is a result of the inhomogeneous nature of dielectric structures. This type of dielectric structures are supposed to be composed of two layers [15], where first layer is the large ferrite grains of fairly well-conducting materials which is separated by the second thin layer (grain boundaries) of relatively poor conducting substances [15]. The ferrite grains are found to be more effective at higher frequencies while grain boundaries are more effective at lower frequencies [16,17].

Actually these variations are also depending on the ratios of the compositions. In order to account for this variation, one can call the difference between the minimum and maximum values of dielectric constants as dielectric strength (Δε), which can be interpreted as followings;

$$\Delta\varepsilon = \varepsilon_0 - \varepsilon_\infty \quad (1)$$

where ε_0 is the dielectric constant at lowest frequency, and ε_∞ is the dielectric constant at highest frequency. Table 2 depicts the Dielectric Strength (Δε) values of these composites.

Table 2 – Dielectric Strength (Δε) values of the samples at various temperatures

Δε	-40 °C	-20 °C	0 °C	20 °C	40 °C	60 °C	80 °C	100 °C	120 °C	140 °C
3A	3.66	4.81	6.18	7.55	8.5	9.12	11.56	17.53	31.39	40.58
3B	5.36	7.77	10.69	13.51	15.82	17.13	17.85	21.87	40.23	47.58
3C	7.36	11.07	17.26	25.02	31.9	35.89	41.31	52.83	73.19	93.75
3D	3.11	4.81	7.17	9.68	11.69	13.56	18.08	27.63	34.8	41.19
3E	2.76	4.38	6.79	9.52	11.97	13.66	16.2	23.36	33.15	44.79

Firstly, it is noteworthy to distinguish the maximum values of sample 3C. This sample contains Complex ferrite and Cobalt Ferrite at the same ratios and dielectric peculiarity of this sample is simply increasing with the increase in temperature. In order to make comments on relative situation of the other samples merely Cobalt Ferrite and Complex Ferrite containing 3A and 3E samples were analyzed and it was observed that Δε of Cobalt Ferrit-3A is more effective at temperatures below 0 °C while Ferrite-3E is more effective above 0 °C. Some further optimization is performed by exploiting 3B and 3D along the temperatures until 80 °C. Actually 3B shows strong and 3D shows weak dielectric strength effect in this interval. The situation is just reciprocal above 80 °C, namely 3B shows weak and 3D shows strong behavior.

The best dielectric peculiarity is exhibited in 3C and this is valid for all temperatures. Also one should look for the imaginary part of dielectric behavior for the energy dependency of materials. In fact imaginary dielectric constant could be distinguished as the energy loss and here imaginary part of ferrite doped PMMA gives us information about the order of AC conductivity [18].

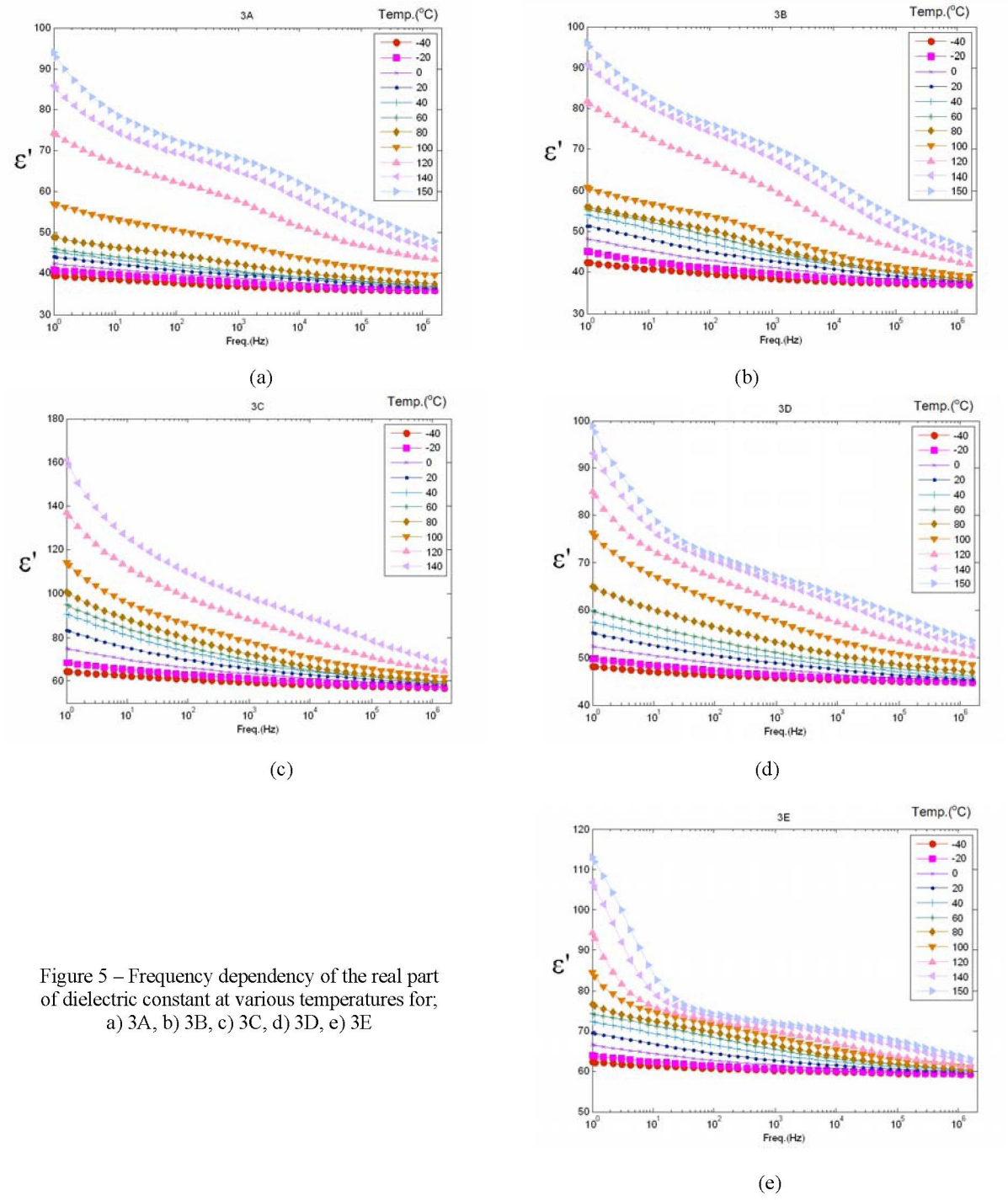


Figure 5 – Frequency dependency of the real part of dielectric constant at various temperatures for; a) 3A, b) 3B, c) 3C, d) 3D, e) 3E

Complex dielectric equation can be written as [19],

$$\varepsilon^{*} = \varepsilon_{\infty} + \frac{(\varepsilon_0 - \varepsilon_{\infty})}{1 + (i\omega\tau)^{1-\alpha}} \qquad (2)$$

where ε_o is the low frequency dielectric value and ε_{∞} is the high frequency dielectric value in the measured frequency interval, ω is the angular frequency, τ is the relaxation time and α is the absorption coefficient. Debye equation can be inspired from Eq. (2) for the merely real dielectric portions and it can be written as [14].

$$\varepsilon'(\omega) = \varepsilon_\infty + (\varepsilon_s - \varepsilon_\infty)\frac{1+(\omega\tau_0)^{1-\alpha}\sin\frac{1}{2}\alpha\pi}{1+2(\omega\tau_0)^{1-\alpha}\sin\frac{1}{2}\alpha\pi+(\omega\tau_0)^{2(1-\alpha)}} \quad (3)$$

These measured values of real dielectric constant are fitted to above function for different temperatures (-40 °C, 20 °C, 80 °C, 140 °C) and relaxation time τ and the absorption coefficient α were acquired from this fit and these values are given in Table 3. Absorption coefficient values are between $0 \leq \alpha \leq 1$ and if it is 0 then Debye type behavior is dominant if it is nonzero then non-Debye behavior is dominant [20]. It is obvious from α values that all ferrite samples demonstrate non-Debye character and their values are between 0.5 and 0.7.

Table 3 – The values of absorption coefficient α and the relaxation time, τ for all samples

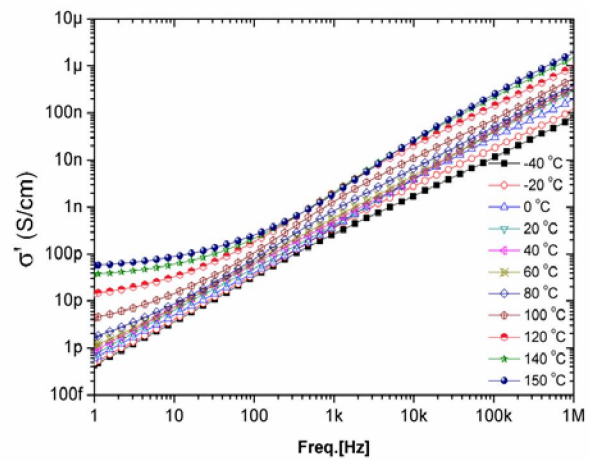
α	-40°C	20°C	80°C	140°C
3A	0.58252	0.69516	0.66238	0.70143
3B	0.56159	0.61779	0.61243	0.6748
3C	0.60027	0.64698	0.63112	0.70037
3D	0.56251	0.66468	0.63544	0.70324
3E	0.68591	0.58286	0.63737	0.65048
τ				
3A	0.02421	0.00149	0.002	0.00186
3B	0.00633	0.00421	0.00155	0.00117
3C	0.00457	0.00817	0.00694	0.01015
3D	0.01275	0.00337	0.00567	0.01037
3E	0.00163	0.00656	0.00279	0.07475

3B exhibits the lowest relaxation time value among the samples for all temperatures. Also the relaxation time τ is decreasing with temperature rise for 3B and this is simply an indicator of short term dipole-dipole interactions, namely higher conductivity values with the mentioned temperature rise. This kind of temperature dependency is actually revealing the semi-conducting behavior caused by ferrite concentration [20]. Fig. 6 confirms this argument explicitly, and 140 °C is the best response temperature for PMMA with Cobalt Ferrite.

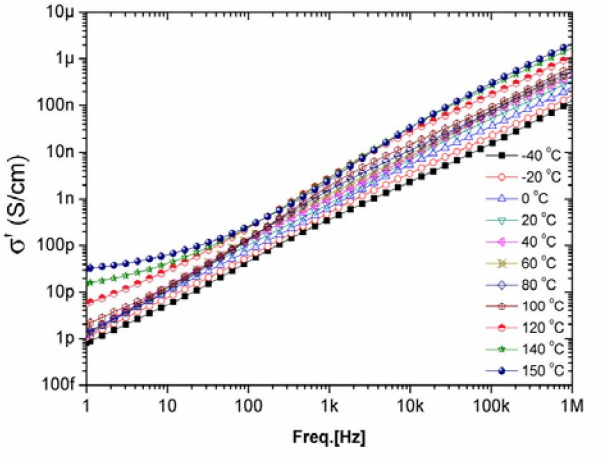
The real part of conductivity σ' is in consistency with the imaginer permittivity values according to, $\sigma' = \omega\varepsilon''\varepsilon_o$ here ω is the angular frequency. Fig. 6 demonstrates the variations of AC conductivity as a function of frequency at different temperatures.

Furthermore, Fig. 6 shows AC conductivity depends on the frequency plots in the temperature range of -40^{0}C to 140^{0}C with 20 °C ramps. As it can be seen from this analysis, weak temperature dependency of conductivity at high frequencies and strong temperature dependency at low frequencies indicate semi-conducting properties of the ferrite. The increase in the ratio of complex ferrite to cobalt ferrite causes stronger temperature dependency at higher temperatures, again verifying the dominancy of the semi-conducting property.

Fig. 7 shows AC conductivity plots of ln (σ) versus 1000/Temperature (K^{-1}) and it was calculated by the equation $\sigma = \sigma_0 \exp(-E/(kT))$ for the concerned samples at 1 Hz spot frequency. The figure shows that the AC conductivity increases almost linearly with temperature for all the samples. The increase in the electrical conductivity as temperature increases may be related to the increase in drift mobility of the thermally activated charge carriers according to hopping conduction mechanism. The semilogarithmic relations for the conductivity represents almost a straight line by changing its slope at the curie temperature T_c, indicating a change of magnetic ordering from ferromagnetism to paramagnetism.



(a)



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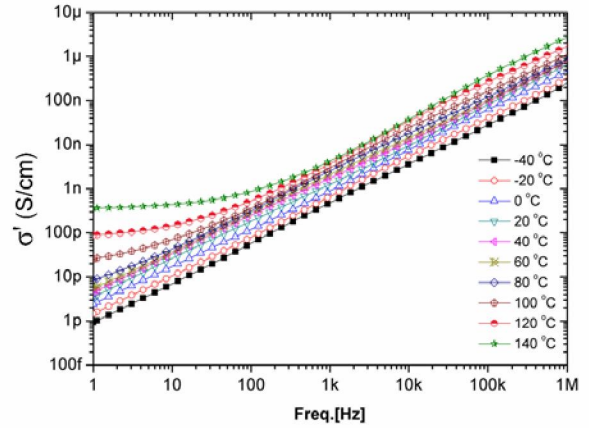

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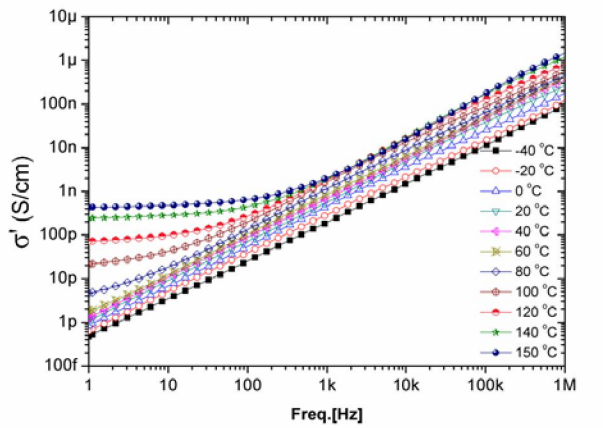

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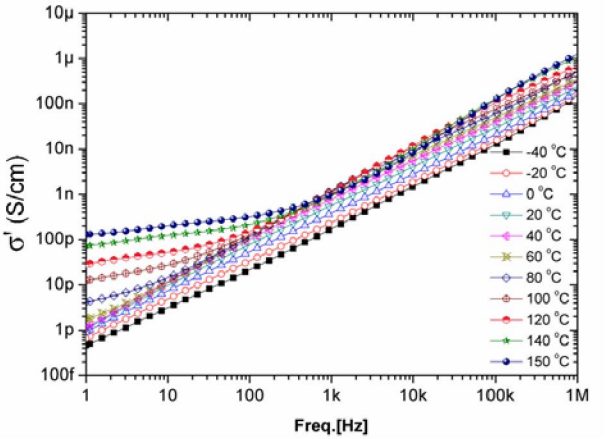

(e)

Figure 6 – The variations of AC conductivity as a function of frequency at different temperatures for samples; a) 3A, b) 3B, c) 3C, d) 3D, e) 3E

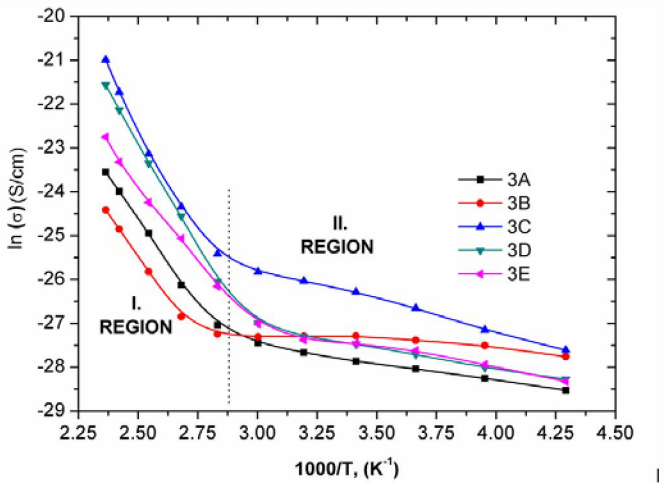

Figure 7 – Arrhenius plots of AC conductivity for 3A, 3B, 3C, 3D, 3E coded samples at 1 Hz spot frequency

The observed increase in conductivity with temperature is normal behavior for semiconductors which follows the Arrhenius relation. It is clear from the figure that the conductivity exhibits a ferritic-semiconductor behavior along the temperature rise. Here we have investigated the panorama in two main regions exhibiting different tendency. One can call these regions as high temperature and low temperature regions according to activation energies whose values are determined from the so called Arrhenius law:

$$\sigma = \sigma_o \left[\exp(-E_I/(kT)) + \exp(-E_{II}/(kT))\right]. \quad (4)$$

Here σ_o is conductivity constant, E_I and E_{II} are activation energies in the Region I and Region II, respectively and k is the Boltzmann constant.

The calculated activation energies of these two regions were depicted in Table 4. In fact, activation energy is a function of temperature, and it gives information about the required energy for keeping the material altogether in the matrix structure. In addition, we tried to acquire information about the trend of the change of Activation Energy at high and low temperatures at low frequencies, which is the critical region in polymers [21]. Activation energies are very low at low temperatures and it is more effective at high temperature region. This activation of charge carrier is indicative of a hopping conduction mechanism. The obtained results suggest that the conduction may be dominated by hopping of carriers between localized states at lower temperatures [22]. The values of activation energies in paramagnetic region are greater than those observed in ferromagnetic region, this suggest that the process of conduction is also affected by the change in magnetic ordering. The activation energy decreases with increasing frequency. This suggests that the applied field frequency enhances the electronic jumps between the localized states and, that is why, activation energy decreases with frequency.

Again the sample 3B is dominant according to activation energy. This situation is also caused by the Cobalt Ferrites. As it was mentioned before, Cobalt Ferrite is more anisotropic and exhibits a better conductivity while the Complex Ferrite shows cohesive behavior in the prepared matrixes. If we compare the activation energy of 3B in high and low temperature regions it is obvious that so called activation energy is higher at high temperatures so the polymer matrix is more accommodating at higher temperatures for the previously optimized 3B sample.

Table 4 – Activation energy dependency at the 1 Hz spot frequency of Ferrites

Ea(eV)	1 [Hz]	
	E_I	E_{II}
3A	0.84216	0.06988
3B	0.87693	0.02985
3C	0.77585	0.12179
3D	0.78628	0.08363
3E	0.80615	0.08093

4. Conclusions. Various informations were acquired by temperature dependent Magnetization and Dielectric Spectroscopy studies of the samples. Equivalently doped Cobalt and Complex Ferrite demonstrated the compatible behaviors to PMMA in all measured temperature ranges according to obtained results, which were discussed in previous section in detail. Contribution of Cobalt ferrite on the magnetization and conductivity is more dominant while Complex ferrite establishes cohesively stronger structure in the constructed matrixes in all temperatures. It was also shown that higher temperatures are more promising for the samples. As an overall evaluation, such kind of polymer matrix designs can be proposed as novel ferritic-semiconductors, which can be explicitly understood from temperature dependency of the material.

The variation of AC conductivity as a function of temperature is almost linear up to the Curie temperature and thereafter a break occurs indicating a change of magnetic ordering from ferromagnetism to paramagnetism. The values of activation energy in paramagnetic region are found to be greater than those in ferromagnetic region, which suggests that the process of conduction is affected by the change in magnetic ordering.

High quality magnetic properties of these samples were previously revealed and these additive electrical performances of the samples can cause novel material and device inspirations, which could be of critical importance in new generation electronics.

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Резюме

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ДИЭЛЕКТРИКАЛЫҚ СПЕКТРОСКОПТЫҚ ТАЛДАУ АРҚЫЛЫ $Ni_{0.5}Zn_{0.4}Cu_{0.1}Fe_2O_4$ ЖӘНЕ $CoFe_2O_4$ ҚОСПАЛАНҒАН PMMA ПОЛИМЕР МАТРИЦАЛАРЫНЫҢ МАГНИТТІК ЖӘНЕ ДИЭЛЕКТРИКАЛЫҚ ҚАСИЕТТЕРІ

VSM және диэлектрикалық спектроскоптық талдау арқылы $Ni_{0.5}Zn_{0.4}Cu_{0.1}Fe_2O_4$ және $CoFe_2O_4$ қоспаланған PMMA полимер матрицаларының магниттік және диэлектрикалық қасиеттері зерттелді. Олар бөлме температурасында домалақ пішінді суперпарамагниттік бөлшектер түрінде гистерезистік қасиетін көрсетеді. Коэрцитивтік өріс күрделі феррит концентрациясын өсірумен айтарлықтай бәсеңдеген. Төмен температураларда, әсіресе феррит концентрациясы орта болса M-H сақиналары едәуір өзгереді. Бұл күрделі және кобальт ферриттердің концентрациясы себебімен әртүрлі материалдан тұратын екі түр кластердің жасалуы арқылы түсіндіріледі. Кобальт ферритінің және күрделі ферритпен қоспаланған PMMA инсуляторының

диэлектрикалық күші мен белсеңдену энергиясы жасалған талдау барысында есептелді. Олардың ең жақсы үйлесімі оңтайландырылды. Кобальт ферритінің өткізгіштікке себептесуі нәтижелі екені байқалды. Сонымен қатар күрделі феррит барлық температураларда матрицаларда мықты құрылымдар жасаған. Электр өткізгіштігінің температураны көтергенде өсуі термальды белсеңдеген заряд тасушылардың жылжыту мобильділігінің өсуіне байланысты. Ауыспалы өткізгіштіктің уақыттың функциясы ретінде вариациясы түзу сызық береді. Сызықтың көлбеулігі кюри температурасында T_c ауысады, бұдан кейін сызық үзіледі. Бұл магниттік реттің ферромагнетизмнен парамагнетизмге ауысқанын көрсетеді.

Тірек сөздер: спектроскоп, ферриттер, температура, заряд, талдау, күш, энергия.

Резюме

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ДИЭЛЕКТРИЧЕСКИЙ СПЕКТРОСКОПИЧЕСКИЙ АНАЛИЗ МАГНИТНЫХ И ДИЭЛЕКТРИЧЕСКИХ СВОЙСТВ $NI_{0.5}ZN_{0.4}CU_{0.1}FE_2O_4$ И ПОЛИМЕРНЫЕ МАТРИЦЫ PMMA ЛЕГИРОВАННЫЕ $COFE_2O_4$

Исследованы с помощью VSM и диэлектрического спектроскопического анализа магнитные и диэлектрические свойства $Ni_{0.5}Zn_{0.4}Cu_{0.1}Fe_2O_4$ и полимерные матрицы PMMA легированные $CoFe_2O_4$. Они показывают гистерезисное поведение при комнатной температуре округленной формой суперпарамагнитных частиц. Коэрцитивные поля значительно сбавлены с повышением концентрации сложных ферритов. При низких температурах кольца M-H сильно меняются, особенно если концентрация феррита средняя. Это объясняется образованием двух типов кластеров из разных материалов из-за концентрации сложных и кобальтовых ферритов. Диэлектрическая сила и энергия активации кобальтового феррита и инсулятора PMMA легированного сложным ферритом были получены в ходе проделанного анализа. Лучшее сочетание было оптимизировано в рамках этой работы. Было замечено, что содействие кобальтового феррита к проводимости более эффективно, тогда как сложный феррит образовывал сильные структуры в матрицах при всех температурах. Повышение электрической проводимости при повышении температуры связано с повышением мобильности смещения термально активированных носителей заряда. Вариация переменной проводимости как функции температуры представляет почти прямую линию. Наклонная линия меняется на температуре кюри T_c, после происходит разрыв, который указывает на изменение магнитного порядка с ферромагнетизма на парамагнетизм.

Ключевые слова: спектроскоп, ферриты, температура, заряд, анализ, сила, энергия.

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