

MAGNETIC NANOCOMPOSITE UNDER PRESSURE: A CASE STUDY OF Ni/C NANOPARTICLES IN POLYMER MATRIX

J. Typek¹, A. Krupska² and N. Guskos^{1,3}

¹Institute of Physics, West Pomeranian University of Technology, Al. Piastow 48, 70-311 Szczecin, Poland

²Institute of Molecular Physics, Polish Academy of Sciences, ul. M. Smoluchowskiego 17, 60-179 Poznan, Poland

³Solid State Section, Department of Physics, University of Athens, Panepistimiopolis, 15 784 Athens, Greece

Received: May 08, 2013

Abstract. In this paper pressure studies of magnetic nanoparticles will be reviewed and, as a particular case, pressure influence on the ferromagnetic resonance (FMR) spectra of Ni/C nanoparticles embedded in the PBT-block-PTMO polymer will be considered. Significant variations of the FMR parameters of Ni/C nanoparticles were observed as a function of an applied external pressure at both studied temperatures ($T = 142\text{K}$, 293K). It has been demonstrated that the pressure dependence of the FMR parameters (the resonance field, linewidth and integrated intensity) correlates well with the temperature dependence of these parameters. Comparison with the previous similar pressure study of two different samples containing $\gamma\text{-Fe}_2\text{O}_3$ agglomerates of different sizes in the PBT-block-PTMO polymer matrix has been carried out.

1. INTRODUCTION

Due to their wide technological applications the transition metal oxides, especially in the form of nanosize structures, are intensively studied in recent years [1,2]. They show a very interesting physical properties, which can be used to improve the mechanical and magnetic properties of materials e.g. [3-9]. Introduction of even small amount of magnetic particles into a polymer matrix can significantly shift the critical points and even increase its melting temperature [10,11].

Ferromagnetic resonance (FMR) technique allows to study such materials on an atomic level. Using small concentration of magnetic particles embedded into polymer matrix the FMR was applied to study the temperature dependence of magnetic properties [10,12]. With the decrease of temperature a decrease of FMR signal amplitude, an increase of

the linewidth and the shift of the resonance line towards smaller magnetic fields was observed. One of the processes responsible for the resonance line shift is the reorientation of the correlated system of spins. It was shown that the viscosity of the studied materials significantly depended on temperature and could greatly influence the processes of reorientation of the correlated spin system [11]. This is particularly important for such polymer matrices in which several critical points of the dynamical processes of freezing the motion of molecules exists. It was proposed that the internal pressure resulting from temperature changes has significantly influence on the electron orbital motion which in turn can change the magnetic moment of magnetic nanoparticles [13].

Recently, the results of measurements of pressure dependence of the FMR spectrum arising from

Corresponding author: J. Typek, e-mail: typjan@zut.edu.pl

Table 1. List of different kinds of studied nanoparticles and types of magnetic measurements used in pressure studies.

Nanoparticles	Type of magnetic measurement	Reference
γ -Fe ₂ O ₃	ZFC and FC dc magnetization, hysteresis loop	[15]
γ -Fe ₂ O ₃ /Fe ₃ O ₄ hollow, solid and dispersed in polymer	ac magnetic susceptibility, isothermal magnetization	[23]
γ -Fe ₂ O ₃	ac and dc magnetic susceptibility, isothermal magnetization	[16]
γ -Fe ₂ O ₃	ac magnetic susceptibility	[19]
γ -Fe ₂ O ₃ in polymer matrix	FMR	[14]
γ -Fe ₂ O ₃ surfactant coated	Magnetic susceptibility	[25]
FePt	ac magnetic susceptibility	[17]
FePt	ac magnetic susceptibility, isothermal magnetization	[22]
LaMnO _{3+x} in mesoporous silica	dc magnetic susceptibility	[24]
La _{0.7} Ca _{0.3} MnO ₃ with different sizes	ZFC and FC dc magnetization, isothermal magnetization	[20]
Magnetostrictive Ni-Zn and Ni-Zn-Co spinels	Complex susceptibility, FMR	[26]
Mn ₃ [Cr(CN) ₆] ₂ ·nH ₂ O, Ni ₃ [Cr(CN) ₆] ₂ ·nH ₂ O in an organic matrix	dc magnetization, isothermal magnetization	[21]
FeOOH·nH ₂ O	dc magnetic susceptibility, isothermal magnetization	[18]

0.1 wt.% and 0.3 wt.% of γ -Fe₂O₃ (maghemite) placed in a polymer matrix have been published [14]. The resonance line have been observed to shift towards stronger magnetic fields, the line broadened and the line amplitude decreased with pressure increase. A crucial role of the surface spins in formation of the FMR spectrum has been proposed.

The aim of this report is to review the results of pressure studies on magnetic nanoparticles and to present our new results of hydrostatic pressure effects on the FMR spectra arising from magnetic nanoparticles of Ni/C embedded at concentration of 0.1 wt.% in the PBT-block-PTMO polymer. The pressure study has been carried out at two temperatures: 293K and at 142K. The pressure dependence of the FMR parameters determined at these two temperatures will be compared with our previous pressure study of two different samples containing γ -Fe₂O₃ agglomerates of different sizes in a polymer matrix [14].

2. REVIEW OF PRESSURE STUDIES OF MAGNETIC NANOPARTICLES

Pressure is an important thermodynamical parameter that could change structural and magnetic properties of materials. In the case of nanoparticles, pressure might modify the transition

temperature to magnetically ordered phase, change the susceptibility and magnetization, alter hysteresis loops and modify the effective anisotropy energy barrier [14-26]. One important advantage of pressure measurements is the possibility of disentangling of the core and shell magnetic properties of nanoparticles. It follows from the different response of these components to the applied pressure. Despite its importance, pressure measurements of magnetic nanoparticles are rare. In Table 1 different kinds of studied nanoparticles and types of magnetic measurements used in pressure studies are presented. As could be noticed a large portion of papers in Table 1 is devoted to the study of γ -Fe₂O₃ nanoparticles [14-16,19,23,25].

Among iron oxides, maghemite nanoparticles are the most attractive materials for industrial applications because of their chemical stability and interesting magnetic properties. The first paper on pressure study of maghemite nanoparticles by J. Dai et al. reported on linear increase of the blocking temperature with applied pressure [25]. It was explained as an effect of reduction of an average interparticle distance between nanoparticles caused by pressure. A detailed study of pressure effect on maghemite nanoparticles with different sizes was published by group of Y. Komorida [15,16,19]. Of

special importance was application of the core/shell structure model used for ferrimagnetic nanoparticles. Pressure study allows to separate anisotropy constants for the core and the shell. The experimental findings indicated that the core/shell structure is unstable against external stress and under sufficiently high pressure the core could be restructured [15,16]. Silva et al. have made pressure measurements on three types of maghemite nanoparticles: hollow capped by oleylamine, solid capped by oleic acid and solid dispersed in a polymer [23]. The anisotropy energy of solid and hollow maghemite nanoparticles has shown different pressure dependence. This difference was due to the different geometry of nanoparticles and with larger pressure response of the shell.

Pressure study of magnetic properties of the iron oxide hydroxide ferrihydrite ($\text{FeOOH}\cdot n\text{H}_2\text{O}$) nanoparticles with the particle size of 4.7 nm was described in Ref. 18. The dc magnetic measurements as a function of temperature or magnetic field have been performed that have shown decrease of the blocking temperature with applied pressure. Pressure has also decreased the effective magnetic moment. On the other hand, the pressure dependence of magnetic susceptibility has shown a behaviour contrary to that of magnetic moment. Together, these findings indicated the decrease of the anisotropy energy associated to the decrease of particles uncompensated magnetic moment [18].

Pressure-induced changes in magnetic properties of FePt nanoparticles are presented in Ref. 17 and 22. FePt has attracted much attention of material scientist for the last decade because it is a candidate material for magnetic recording media. Its magnetic properties are very sensitive to nanoparticle sizes. In Ref. 17 the size of FePt nanoparticles was 2.0 nm, in Ref. 22 it was 2.6 nm. Change of the blocking temperature T_B under the applied pressure (linear increase with pressure) was investigated by ac susceptibility measurements. As particle size and lattice parameters has hardly changed in the considered pressure region, the experimental results suggested the effective magnetocrystalline anisotropy increased due to the applied pressure [17]. For larger FePt nanoparticles (2.6 nm) the blocking temperature was reduced by applied pressure [22]. The study revealed that anisotropic unit-cell contraction reduced the effective anisotropic energy and the magnetic nature of nanoparticles could be controlled by changing the contraction manner [22].

Investigation of two different kinds of nanoparticles, based on prussian blue analogues,

$\text{Mn}_3[\text{Cr}(\text{CN})_6]_2\cdot n\text{H}_2\text{O}$, and $\text{Ni}_3[\text{Cr}(\text{CN})_6]_2\cdot n\text{H}_2\text{O}$ embedded in an organic matrix was described in Ref. 21. The nanoparticles have had an average diameter of 4 nm. The Curie temperature T_C of both nanoparticles types was significantly reduced in comparison with bulk materials due to dilution in an organic matrix. Pressure effect on the Curie temperature was quite opposite for both nanoparticles - in case of $\text{Mn}_3[\text{Cr}(\text{CN})_6]_2\cdot n\text{H}_2\text{O}$ an increase of pressure caused an increase of T_C , while for $\text{Ni}_3[\text{Cr}(\text{CN})_6]_2\cdot n\text{H}_2\text{O}$ nanoparticles it caused decrease of T_C . The applied pressure also increased the magnetic moment of both samples it this was attributed to the reduction of inter-particle distances by compression of the organic matrix [21].

Pressure effects on magnetic properties of two $\text{La}_{0.7}\text{Ca}_{0.3}\text{MnO}_3$ nanoparticle samples with different particle sizes (12 and 49 nm) were investigated in Ref. 20. Sample with larger nanoparticles was obtained from the first sample (black porous ash) by annealing it at 900 °C for 2 h in air. The Curie temperatures T_C at ambient pressure were very different for both samples (120K and 261K) and the saturation magnetization for the annealed sample was almost two times higher than for non-annealed sample. Differences in magnetic behavior of these samples became more pronounced under the applied pressure. The pressure coefficient dT_C/dP was positive for the annealed sample and negative for the other sample. Other magnetic parameters (coercivity, remanence, saturation magnetization) underwent increase with pressure for annealed sample, while for non-annealed sample coercivity and remanence increased with pressure but at same time saturation magnetization decreased. These differences in magnetic behavior under the applied pressure were explained by different contributions of two types of disorder: surface disorder introduced by the particle outer shell, and structural disorder of the particle core. The larger, annealed sample has had small shell thickness and thus its behaviour closely resembled the one of the bulk counterpart. For small size nanoparticles sample the magnetic behaviour is governed by both particle shell contribution and large core disorder [20].

In Ref. 24 application of anisotropic stress to $\text{LaMnO}_{3+\delta}$ nanoparticles synthesized in one-dimensional pores of mesoporous silica was presented. The authors have investigated the effects of the anisotropic strain on the nanoparticles, which consisted of ferromagnetic and antiferromagnetic particles. Both the ferromagnetic and antiferromagnetic transition temperatures for the $\text{LaMnO}_{3+\delta}$ nanoparticles initially increased with

increasing pressure and then remained constant at around the critical strain. These results indicated that anisotropic stress caused distortion of the shape of nanoparticles. This induced an increase in the lattice strain and the anisotropic compression of the crystal structure, which, in turn, resulted in an increase in the transition temperatures [24].

Pressure dependence of the frequency permeability spectra of soft ferrite composite materials was presented in Ref. 26. Soft magnetic composite materials were prepared by mixing epoxy resin with nanosize particles with diameter of 30 nm of magnetostrictive Ni-Zn and Ni-Zn-Co spinels. The measured FMR frequencies of samples were obtained from permeability spectra (maximum of the imaginary part) for various applied pressures. The FMR frequency increased linearly with the compression strength only beyond a pressure threshold. Beyond this threshold value, FMR remained insensitive to variations of the applied pressure. This region corresponds to permanent internal stress that existed before application of external pressure.

3. EXPERIMENTAL

The procedure for obtaining nanocomposite with a small amount (0.1 wt.%) of magnetic Ni/C nanoparticles embedded in the PBT-block-PTMO polymer was described previously [27,28]. An average size of agglomerates was in the 10-20 nm range.

RADIOPAN SE/X spectrometer was used for study of the pressure dependence of the FMR spectra. FMR measurements at a frequency of 9.4 GHz under high hydrostatic pressure were carried out using a special appliance connected to a standard spectrometer [29]. This equipment, made at the Institute of Molecular Physics in Poznan (Poland) is a modified version of the apparatus used previously [30]. Technical details are described in our previous paper [14]. The studied sample was placed into the 2 mm diameter hole of corundum resonator. The pressure measurements were carried out at constant temperature of 293K and 142K.

4. RESULTS AND DISCUSSION

Fig. 1 presents temperature dependence of the FMR spectrum parameters at ambient pressure in the investigated sample of Ni/C nanoparticles embedded in PBT-block-PTMO polymer. The FMR spectrum consists of a single intense, broad and anisotropic line. The FMR parameters: resonance field B_r , peak-to-peak linewidth ΔB_{pp} , and integrated

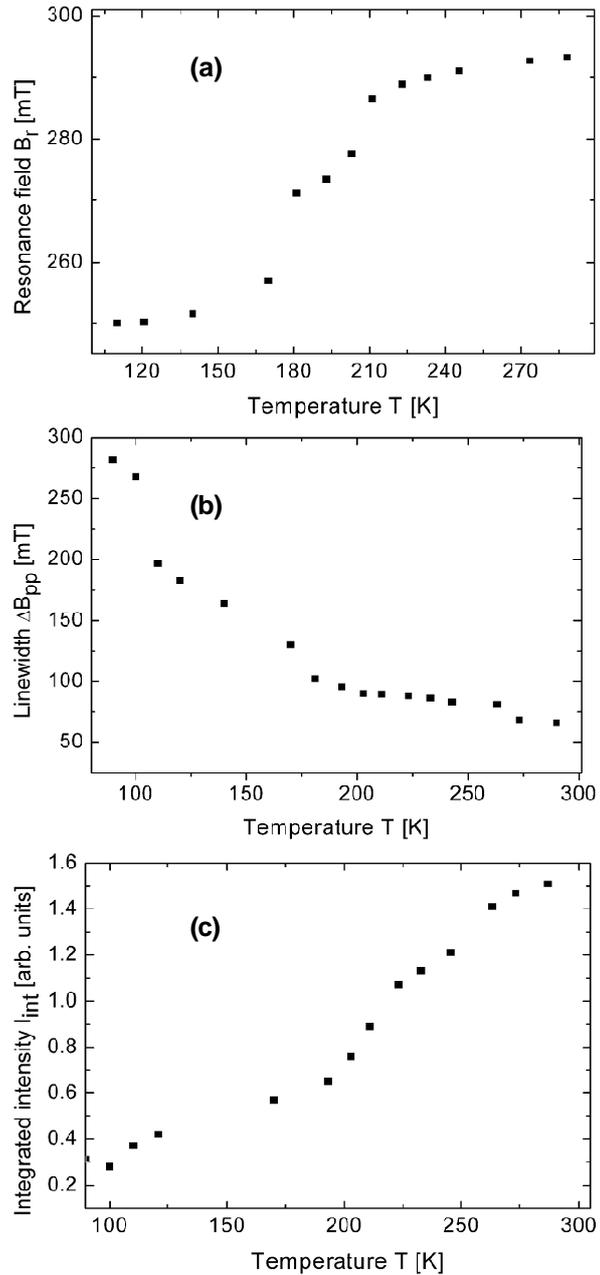


Fig. 1. Temperature dependence of the FMR parameters at ambient pressure: (a) resonance field, (b) peak-to-peak linewidth, (c) integrated intensity, for investigated sample of Ni/C nanoparticles in the PBT-block-PTMO polymer.

intensity I_{int} have been calculated by using procedure described by Koksharov [31]. The FMR integrated intensity I_{int} was calculated as the product of the first derivative line amplitude A_{pp} and the square of the peak-to-peak linewidth ΔB_{pp} : $I_{int} = A_{pp} \cdot \Delta B_{pp}^2$. It is proportional to the magnetic susceptibility (at microwave frequency) of the investigated spin system. At ambient pressure the resonance line is centered at $B_r = 293.5$ mT, with linewidth $\Delta B_{pp} = 63.9$ mT at 293K, and $B_r = 252.2$ mT, with linewidth $\Delta B_{pp} =$

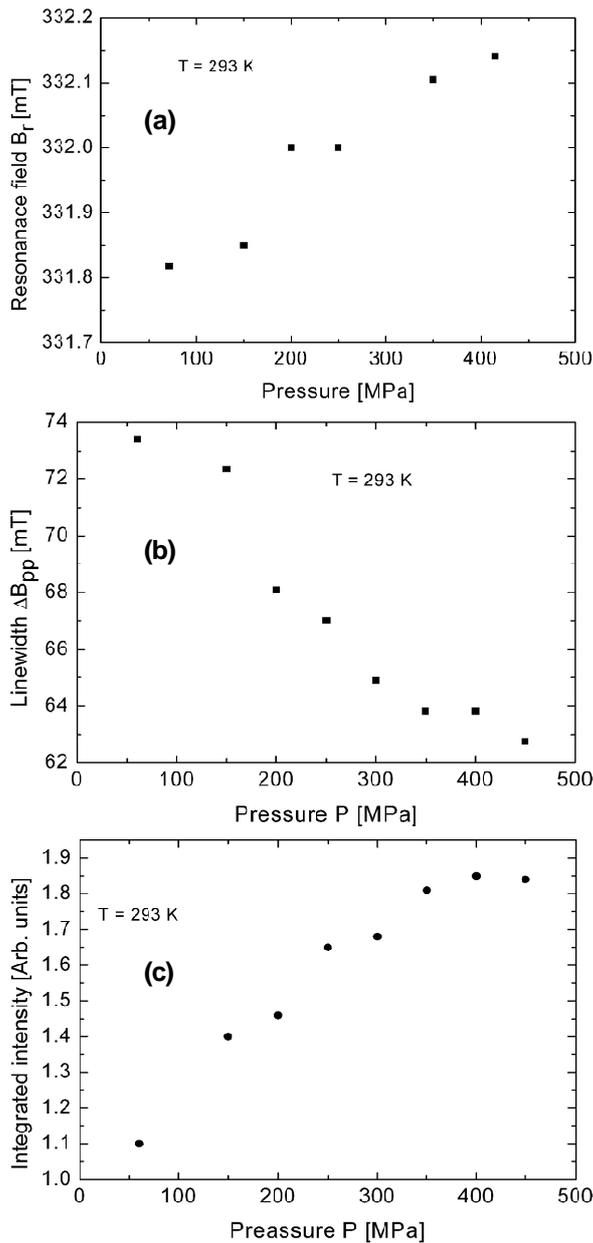


Fig. 2. Pressure dependence of the FMR parameters at $T = 293\text{ K}$: (a) resonance field, (b) peak-to-peak linewidth, (c) integrated intensity, for investigated sample of Ni/C nanoparticles in the PBT-block-PTMO polymer.

157.4 mT at 142K. The linewidth increases, the integrated intensity decreases and the resonance field shifts towards smaller magnetic fields with decrease in temperature.

Figs. 2 and 3 show the pressure dependence of the same FMR parameters at two different temperatures. At both temperatures the resonance line shifts towards stronger magnetic fields (Figs. 2a and 3a) with increase in pressure. A similar behavior of the resonance line was recorded for the FMR spectra of $\gamma\text{-Fe}_2\text{O}_3$ nanoparticles embedded in

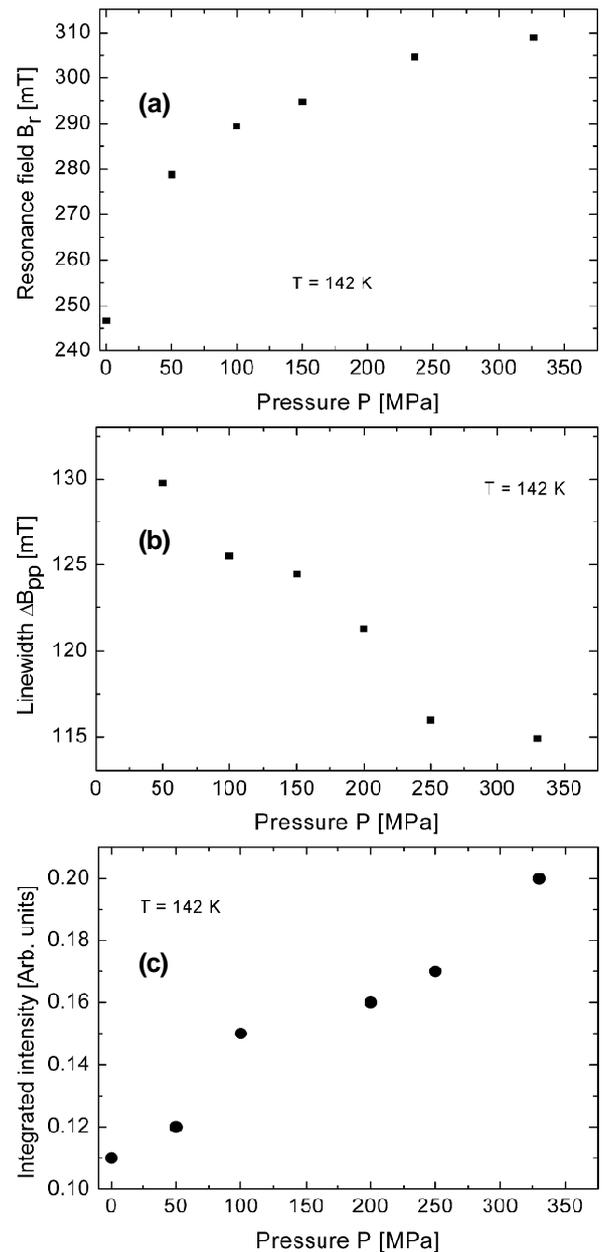
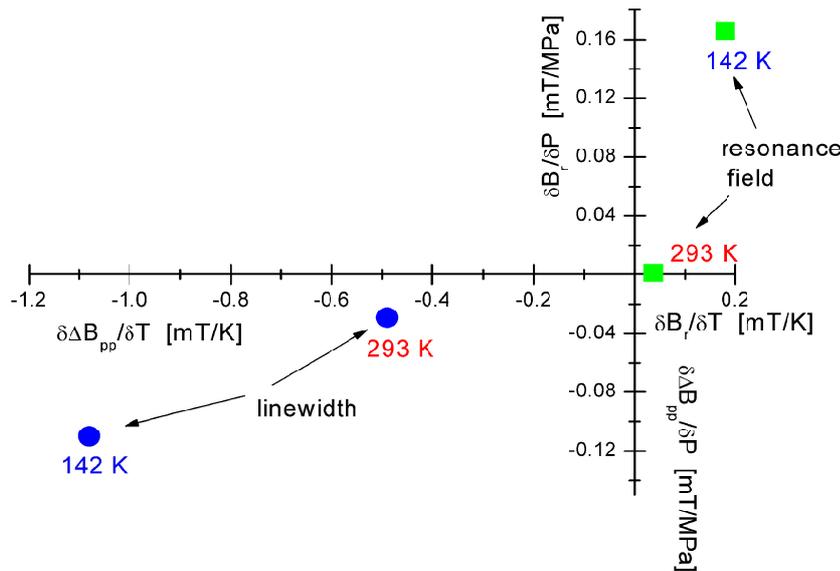


Fig. 3. Pressure dependence of the FMR parameters at $T = 142\text{ K}$: (a) resonance field, (b) peak-to-peak linewidth, (c) integrated intensity, for investigated sample of Ni/C nanoparticles in the PBT-block-PTMO polymer.

nonmagnetic polymer matrix [14] and for sample investigated in this work in the high temperature range [14,27]. The linewidth decreases with an increase in applied pressure at both temperatures (Fig. 2b and 3b) and it is an opposite effect to what was observed for $\gamma\text{-Fe}_2\text{O}_3$ nanoparticles embedded in nonmagnetic polymer matrix [14]. Probably this is due to the difference in magnetic interactions because in contrast to the maghemite nanoparticles the Ni nanoparticles are covered with carbon layer which increases the distance between the metal

Table 2. Calculated values of the pressure gradients of the FMR spectrum parameters in three different samples.

Sample	Temperature [K]	$\delta B_r/\delta P$ [mT/MPa]	$\delta B_{pp}/\delta P$ [mT/MPa]	$\delta I_{int}/\delta P$ [1/MPa]	Reference
0.1 wt.% Ni/C	293	$0.1 \cdot 10^{-2}$	$-2.95 \cdot 10^{-2}$	$17 \cdot 10^{-4}$	This work
	142	$16.6 \cdot 10^{-2}$	$-11 \cdot 10^{-2}$	$26 \cdot 10^{-4}$	This work
0.1 wt.% γ -Fe ₂ O ₃ (large size agglomerates)	82	$2.8 \cdot 10^{-2}$	$1.2 \cdot 10^{-2}$	$3.0 \cdot 10^{-4}$	[14]
0.1 wt.% γ -Fe ₂ O ₃ (small size agglomerates)	92	$4.4 \cdot 10^{-2}$	$5.0 \cdot 10^{-2}$	$18 \cdot 10^{-4}$	[14]

**Fig. 4.** Presentation of values of the pressure and temperature gradients of the resonance field (full squares) and linewidth (full circles) at $T = 293$ and 142 K for investigated sample of Ni/C nanoparticles in the PBT-block-PTMO polymer.

nanoparticles and has a profound influence on the state of the surface spins. The integrated intensity of the FMR line increases with increase in an applied external pressure at both temperatures (Figs. 2c and 3c). A similar behavior was also observed for maghemite nanoparticles [14].

In the Table 1 the values of pressure gradients of changes in resonance field, linewidth and integrated intensity for the Ni/C nanoparticles embedded in the PBT-block-PTMO polymer are shown. For comparison, similar parameters for two samples of the γ -Fe₂O₃ nanoparticles embedded in nonmagnetic polymer matrix are also given [14]. Two previously studied samples at roughly the same temperature contained maghemite nanoparticles in a very different agglomeration state so it was possible to elucidate the role of the surface spins [14]. It was found that for smaller maghemite agglomerates the pressure gradient of the resonance field was bigger than for larger agglomerates. In case of our sample of Ni/C

nanoparticles we have the same agglomerates but they were investigated at two different temperatures. As could be seen in Table 1 at higher temperature the value of $\delta B_r/\delta P$ gradient is much smaller than at lower temperature. It would be interesting to compare the pressure and temperature gradients of the resonance field at both temperatures. The temperature gradients of the resonance field have the values $\delta B_r/\delta T \sim 3.7 \cdot 10^{-2}$ mT/K above 150 K and $\delta B_r/\delta T \sim 0.18$ mT/K below 150 K (Fig. 1a). It seems to be a strong correlation between the values of pressure and temperature gradients at both studied temperatures. It indicates on a common cause underlying the pressure and temperature changes of the resonance field. This correlation could be easily noticed in Fig. 4 (right hand side), where the values of the pressure and temperature gradients are presented. The variation of $\delta B_r/\delta T$ could reflect an increase of magnetic anisotropy or even interparticles interactions that might be connected

with the reorientation processes in the correlated spin system. An elastic stress could also strengthen the interactions by reducing an average interparticle distance. These differences could be related to the value of the saturation magnetization M_s as the resonance field B_r is expected to vary with applied external pressure P according to the formula [26]:

$$B_r \approx \frac{\lambda_s}{M_s} P,$$

where λ_s is the magnetostriction constant. Saturation magnetization of the nanoparticles varies strongly in comparison to the bulk and this behaviour is mainly related to the presence of surface anisotropy and disorder. The surface layer of a nanoparticle could be in a paramagnetic state due to the interaction with the matrix [32].

Significant changes under the influence of pressure are also observed in the linewidth of the resonance line and the effect of pressure is much more stonger in low temperature range than at high temperaure (Table 1). Likewise as for the resonance field, in case of the linewidth its pressure and temperature gradients are also strongly correlated. As could be calculated from Fig. 1b the temperature gradients of linewidth have the following values: $\delta\Delta B_{pp}/\delta T = -49 \cdot 10^{-2}$ mT/K above 150K and $-108 \cdot 10^{-2}$ mT/K below 150K. An increase of pressure has a similar effect on the linewidth as has an increase of temperature and this correlation is easily seen in Fig. 4 (left hand side). The observed FMR line is the sum of many individual component lines reflecting the different orientation of each nanoparticle relative to the external magnetic field. Thus the unresolved component lines determine the observed linewidth that would depend directly on the magnetocrystalline anisotropy field of the nanoparticles system [33]. The magnetocrystalline anisotropy field H_{mc} could be given as [27]:

$$H_{mc} = \frac{K_1}{M_s},$$

where K_1 is the first-order uniaxial anisotropy constant. This field, and in consequence the resulting linewidth, would increase as the applied pressure decreases the saturation magnetization. As the pressure gradient $\delta\Delta B_{pp}/\delta P$ is roughly four times bigger at low temperatures it follows that pressure has that much more effect on M_s at 142K than at 293K.

Figs. 2c and 3c show the pressure dependence of the FMR integrated intensity. To determine the change of the integrated intensity with increase of

applied pressure, the $\delta I_{int}/\delta P$ gradients have been calculated and the values are placed in Table 1. At lower temperature the value of $\delta I_{int}/\delta P$ is one and a half times greater than at 293K. The values of the temperature gradients of the integrated intensity $\delta I_{int}/\delta T$ for our sample at different temperatures could be calculated from Fig. 1c. It was found that the values of $\delta I_{int}/\delta T$ are $30 \cdot 10^{-2}$ 1/K and $32 \cdot 10^{-2}$ 1/K at 293K and 142K, respectively. Comparison of $\delta I_{int}/\delta P$ and dI_{int}/dT values at different temperatures indicate once again that there is a significant correlation of pressure and temperature changes of the integrated intensity. On the other hand comparison of $\delta I_{int}/\delta P$ values for Ni/C and maghemite nanoparticles shows that larger vales are obtained for smaller agglomerates. This is consistent with small sizes of Ni/C nanoparticles in the current study.

5. CONCLUSIONS

The influence of an external pressure on the FMR spectra of Ni/C nanoparticles embedded in a nonmagnetic polymer has been investigated at two different temperatures. Pressure dependence of the FMR parameters has shown that the shift of the resonance line, and the changes of linewidth and the FMR integrated intensity due to applied external pressure are similar to what was observed previously for these parameters as the temperature was varied. This indicates on a common cause of pressure and temperature variations of the FMR parameters. An opposite effect of temperature and pressure variations was observed for the linewidth changes in case of maghemite nanoparticles. This might be explained by assuming an important role played by the surface spins. At low temperature the calculated values of $\delta B_r/\delta P$, $\delta\Delta B_{pp}/\delta P$ and $\delta I/\delta P$ pressure gradients for Ni/C nanoparticles were consistently larger than at high temperature. In general the pressure and temperature changes of the FMR parameters of nanoparticles embedded in polymer matrix appear to be strongly correlated but in cases when spins on the surface of nanopartiles play a significant role this correlation might be weakened or even reversed.

REFERENCES

- [1] M. Fernandez-Garcia and J.A. Rodriguez, In: *Nanomaterials. Inorganic and Bioinorganic Perspective*, ed. by Ch.M. Lukehart and R.A. Scott (Wiley, Chichester (UK), 2008), p. 453.
- [2] C. Burda, X. B. Chen, R. Narayanan and M.A. El-Sayed // *Chem. Rev.* **105** (2005) 1025.

- [3] I.A. Ovid'ko and T.G. Langdon // *Rev. Adv. Mater. Sci.* **30** (2012) 103.
- [4] L.J.L. Dormann, D. Fiorani and E. Tronc // *Adv. Chem. Phys.* **98** (1997) 283.
- [5] B. Martínez, X. Obradors, L. Balcells, A. Rouanet and C. Monty // *Phys. Rev. Lett.* **80** (1998) 181.
- [6] R.H. Kodama and A.E. Berkowitz // *Phys. Rev. B* **59** (1999) 6321.
- [7] P. Dutta, A. Manivannan, M.S. Seehra, N. Shah and G.P. Huffman // *Phys. Rev. B* **70** (2004) 174428.
- [8] M. Maryniak, N. Guskos, J. Typek, I. Kucharewicz, U. Narkiewicz, Z. Roslaniec, M. Kwiatkowska, W. Arabczyk and K. Aidinis // *Rev. Adv. Mater. Sci.* **12** (2006) 200.
- [9] N. Guskos, V. Likodimos, S. Glenis, J. Typek, M. Maryniak, Z. Roslaniec, M. Baran, R. Szymczak, D. Petridis and M. Kwiatkowska // *J. Appl. Phys.* **99** (2006) 084307.
- [10] N. Guskos, V. Likodimos, S. Glenis, M. Maryniak, M. Baran, R. Szymczak, Z. Roslaniec, M. Kwiatkowska and D. Petridis // *J. Nanosci. Nanotech.* **8** (2008) 2127.
- [11] M.R. Dudek, N. Guskos, E. Senderek and Z. Roslaniec // *J. Alloy. Compd.* **504** (2010) 289.
- [12] N. Guskos, J. Typek, U. Narkiewicz, M. Maryniak and K. Aidinis // *Rev. Adv. Mater. Sci.* **8** (2004) 10.
- [13] N. Guskos, J. Typek and M. Maryniak // *phys. stat. sol. (b)* **244** (2007) 859.
- [14] N. Guskos, A. Krupska and J. Typek // *Rev. Adv. Mater. Sci.* **32** (2012) 19.
- [15] Y. Komorida, M. Mito, H. Deguchi, S. Takagi, A. Millan and F. Palacio // *J. Magn. Magn. Mater.* **310** (2007) e800.
- [16] Y. Komorida, M. Mito, H. Deguchi, S. Takagi, T. Tajiri, A. Millan, N.J.O. Silva, M.A. Laguna and F. Palacio // *J. Magn. Magn. Mater.* **322** (2010) 2117.
- [17] Y. Komorida, M. Mito, H. Deguchi, S. Takagi, T. Iwamoto and Y. Kitamoto // *J. Phys.: Conf. Ser.* **200** (2010) 072054.
- [18] Y. Komorida, N.J.O. Silva, M. Mito, H. Deguchi, S. Takagi, F. Palacio and V.S. Amaral // *J. Phys.: Conf. Ser.* **150** (2009) 042098.
- [19] Y. Komorida, M. Mito, H. Deguchi, S. Takagi, A. Millan, N.J.O. Silva and F. Palacio // *Appl. Phys. Lett.* **94** (2009) 202503.
- [20] V. Kusigerski, D. Markovic, V. Spasojevic, M. Tadic, M. Zentkova and M. Mihalik // *J. Nanopart. Res.* **12** (2010) 1299.
- [21] Zentko, M. Zentkova, V. Kavecansky, M. Mihalik, Z. Mitrova, Z. Arnold, J. Kamarad, M. Cieslar and V. Zelenak // *Acta Phys. Pol. A* **113** (2008) 489.
- [22] M. Mito, Y. Komorida, H. Deguchi, T. Tajiri, T. Iwamoto and Y. Kitamoto // *J. Appl. Phys.* **113** (2013) 044302.
- [23] N.J.O. Silva, S. Saisho, M. Mito, A. Milla, F. Palacio, A. Cabot, O. Iglesias and A. Labarta // *J. Magn. Magn. Mater.* **335** (2013) 1.
- [24] T. Tajiri, S. Saisho, Y. Komorida, M. Mito, H. Deguchi and A. Kohno // *J. Appl. Phys.* **110** (2011) 044307.
- [25] J. Dai, J.Q. Wang, C. Sangregorio, J. Fang, E. Carpenter and J. Tang // *J. Appl. Phys.* **87** (2000) 7397.
- [26] Chevalier, E. Le Guen, A.C. Tarot, B. Grisart, D. Souriou, P. Queffelec, A. Thakur and J.L. Mattei // *IEEE Trans. Magn.* **47** (2011) 4132.
- [27] N. Guskos, M. Maryniak, J. Typek, P. Podsiadly, U. Narkiewicz, E. Senderek and Z. Roslaniec // *J. Non-Cryst. Solids* **355** (2009) 1400.
- [28] N. Guskos, J. Typek, B.V. Padlyak, Yu.K. Gorelenko, I. Pelech, U. Narkiewicz, E. Senderek, A. Guskos and Z. Roslaniec // *J. Non-Cryst. Solids* **356** (2010) 37.
- [29] J. Stankowski, A. Galezewski, M. Krupski, S. Waplak and H. Gierszal // *Rev. Sci. Instrum.* **47** (1976) 128.
- [30] M. Krupski // *Rev. Sci. Instrum.* **67** (1996) 2894.
- [31] Yu.A. Koksharov, S.P. Gubin, I.D. Kosobudsky, M. Beltran, Y. Khodorkovsky and A.M. Tishin // *J. Appl. Phys.* **88** (2000) 1587.
- [32] C. Caizer // *Physica B* **327** (2003) 27.
- [33] Helminiak, W. Arabczyk, G. Zolnierkiewicz, N. Guskos and J. Typek // *Rev. Adv. Mater. Sci.* **29** (2011) 166