

Study of Self-Assembly Processes of Polymer Nanocomposites Based on Cobalt Nanowires in a Magnetic Field by Magnetometry and NMR Methods

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Abstract

The self-assembly processes in magnetic polymer nanocomposites based on cobalt nanowires were studied during their polarization in an external magnetic field. These processes occur due to diffusion during polarization of magnetic nanowires, stimulated by the action of an external constant magnetic field. Experimental data obtained by complementary methods of NMR and magnetometry allow us to visually evaluate the degree of self-assembly of nanowires during their polarization. The obtained polymer composites are promising for practical applications.

Keywords: cobalt nanowires, polymer nanocomposites, polarization, magnetometry, NMR.

Introduction

In recent decades, the study of such new nanostructured forms as nanoparticles, nanotubes, and nanowires has become especially relevant. This is because nanostructured particles, due to their size and features of the atomic structure, exhibit such unique physical and mechanical properties that the range of their promising applications covers many areas of activity from microelectronics to medicine (Gubin et al., 2005).

Control of nano objects for their arrangement in rows, signatures, and grids is the key to obtaining new functional materials. Therefore, in recent years, many methods of self-assembly and synthesis have been developed to obtain structural units of various nanometric sizes. In this regard, the possibility of an ideal controlling nanoparticles' self-assembly and synthesis processes is a major challenge from the standpoint of both fundamental and applied research (Tretyakov, 2003).

Based on fundamental principles, the self-organization process requires the interactions between atoms and clusters, as well as thermodynamic and kinetic driving forces, so that the organization of atoms and clusters to create nanoscale domain structures can be realized. From this standpoint, magnetic nanoparticles particularly interesting due to their unique physicochemical properties and applicability in new functional material technologies.

In recent years, the magnetic field has been used to create nanoscale materials, which has led to significant achievements in the fabrication of macro- and microstructured synthesized materials with unique properties. Unlike other existing self-

assembly technologies, magnetic field-induced ordering determines the formation of magnetic nanoparticles into ordered structures with unique properties. Therefore, the scope of application of magnetic nanoparticles is large. It is enough to name such applications as magnetic fluids, scratch-resistant plastic glasses, magnetic storage media, sensors, biomedicine, etc. (Tracy & Crawford, 2013; Mourkas et al., 2021).

In Gavasheli et al. (2024) and Gegechkori and Mamniashvili (2025), the effects of amplification of the NMR signal intensity and the transverse relaxation rate T_2 , as well as magnetic susceptibility, were observed during magnetization reversal of a polymer nanocomposite sample with cobalt nanowires in an epoxy matrix polarized in an external magnetic field. For this, two complementary methods were used - the microscopic NMR spin echo method and the macroscopic resonant RF magnetometry method. These methods also allow obtaining information on the coercive force of cobalt nanowires, the pinning force of domain walls (DWs), and their anisotropy depending on the direction of the external magnetic field relative to the magnetization of the nanowire. This information may be useful for improving the production technology of cobalt nanowires for their practical use in sensors, memory devices, rare earth-free permanent magnets, and other applications.

In the work (Aneli et al., 2019), the self-assembly properties of carbon nanoparticles doped with cobalt nanoclusters in polymer nanocomposite films in a non-uniform field of a neodymium magnet were previously studied using NMR and magnetometry.

The aim of this work is a comparative study of the effect of the self-assembly process of polymer nanocomposites based on cobalt nanowires polarized in a uniform external magnetic field and prepared in its absence using NMR and magnetometry.

Experimental Results and Their Discussion

To characterize the magnetic properties of the obtained samples, the RF resonance magnetometry method was used in this work, which allows for a relatively quick assessment of the magnetic state (ferromagnetic state, superparamagnetism) of the samples, compared to longer measurements using the vibration magnetometry method (Mamniashvili et al., 2014). The magnetometer uses an LC resonant generator assembled according to a standard circuit using field-effect transistors, Fig. 1a.

The scheme based on the LC resonant generator (Fig. 1a) was previously used to measure the transverse susceptibility of magnetic samples (Mamniashvili et al., 2014). The samples were placed in the induction coil of the LC generator. Changes in its resonant frequency were measured by changing the external magnetic field H , perpendicular to the field of the coil H_{RF} . In this paper, we study the change in the resonance frequency $\Delta f(H)$ under the influence of an external magnetic field directed along, against and across the magnetization of a sample with cobalt nanowires.

Resonance methods have the advantage of accuracy and high sensitivity when it comes to detecting changes in the physical properties of materials depending on temperature and magnetic field.

The assessment of the DW pinning force in cobalt nanowires by the NMR method yielded an estimate of its value of about 100 Oe and higher (Gegechkori & Mamniashvili, 2025). It should be expected that the value of the external magnetic field required for the magnetization reversal (reversal) of the nanowires magnetization, pre-polarized in a magnetic field is close to the value of the DW pinning force in these nanowires. Since the residual magnetic field of the iron cores of the electromagnet has a value of about 200 Oe, Helmholtz coils were used to measure in the required range of the external magnetic field from 0 to 200 Oe. The process of magnetization reversal of nanowires was studied for three cases: in the case of an increase in the magnetic field along the magnetization of the nanowire and the opposite direction, as well as in the case of its direction transverse to the nanowires.

The scheme of the NMR spectrometer for studying the two-pulse echo (TPE) signal (Gegechkori & Mamniashvili, 2025) is shown in Fig. 1b.

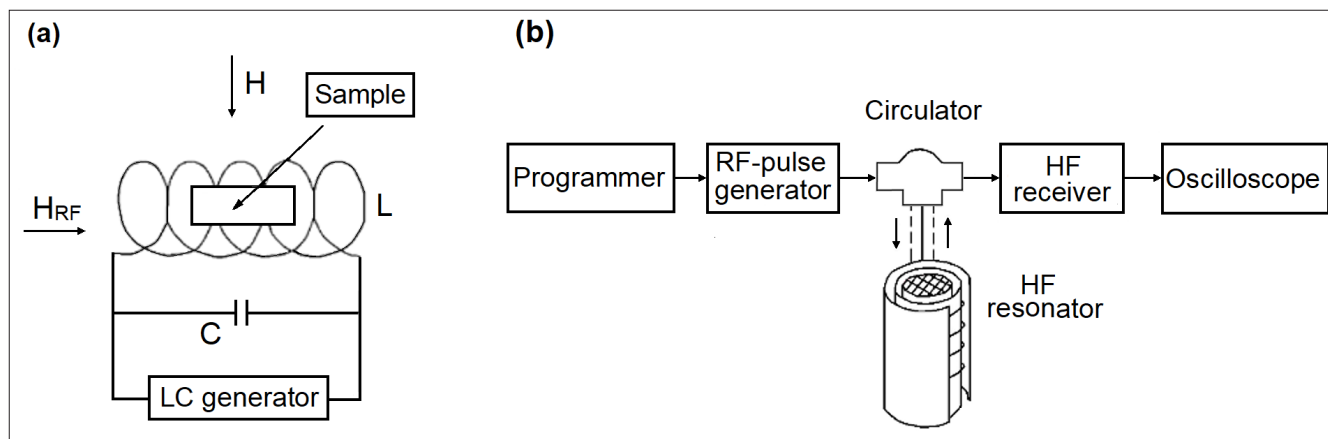


Figure 1: The scheme of the resonant magnetometer based on the LC generator, H_{RF} and H are the radio frequency field and the external field, respectively (a); scheme of the NMR spectrometer (b).

The NMR measurements were performed on a phase-incoherent spin echo spectrometer (Gegechkori & Mamniashvili, 2025) in the frequency range of 200–400 MHz at a temperature of 293 K. In the range of 200–400 MHz, a commercial Lecher-type generator with a two-wire line was used, including two inductors with different numbers of turns. With a pulse length of 0.1–50 μ s, the maximum amplitude of the RF field obtained on the sample is ~ 3.0 Oe, and the steepness of the fronts is no worse than 0.15 μ s. The dead time of the receiver is ~ 1 μ s.

Characteristic parameters of RF pulses: duration of units of microseconds, delay between them of tens of microseconds. The carrier frequency of about 213 MHz at room temperature coincides with the frequency of the nuclei in the center of the DW of the face-centered cubic phase of cobalt.

Resonance methods have the advantage of accuracy and high sensitivity when it comes to detecting changes in the physical properties of materials depending on temperature and magnetic field.

Cobalt nanowire samples from Plasma Chem GmbH (Chemical plant in Berlin, Germany) with a diameter of 200–300 nm and a length of up to 200 μ m was used to manufacture the samples.

Epoxy capsules with cobalt nanowires were made. For this, the nanowires were placed in a polyethylene tube with epoxy resin. In particular, a transparent polyethylene tube 3 cm long and 6 mm in inner diameter is used. The required amount of cobalt nanowire (in our case 0.4 g) is placed in it and epoxy resin is added to make a capsule 2 cm long. Then, liquid epoxy

resin is mixed in the capsule until a homogeneous suspension of cobalt nanowires is obtained. Finally, the capsule is closed with a silicone plug and placed between the poles of a magnet for 24 hours until the epoxy resin completely hardens. Then, the direction of the magnetic field on the resulting capsule is noted and it is examined. In addition, capsules with polymer nanocomposites were made that were not polarized in an external field. In Fig. 2 shows the dependences of the change in the resonance frequency $\Delta f(H)$ under the action of a magnetic field (initial resonance frequency $f_0 = 10$ MHz) along and across the sample with polarized nanowires.

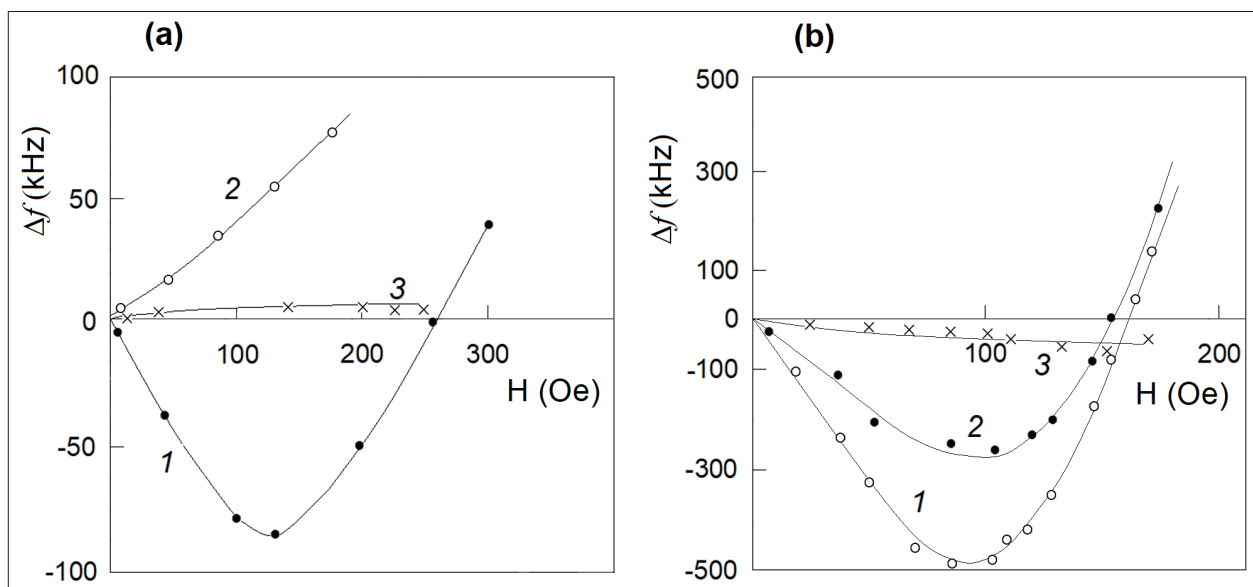


Figure 2: Change in the resonance frequency of the magnetometer with a polarized polymer nanocomposite sample based on cobalt nanowires: a magnetic field increases in the direction opposite to its magnetization (1), a magnetic field increases in the direction of magnetization (2), the magnetic field increases in the direction perpendicular to the nanowire (3) for a polarized sample; similar dependences for non-polarized sample (b).

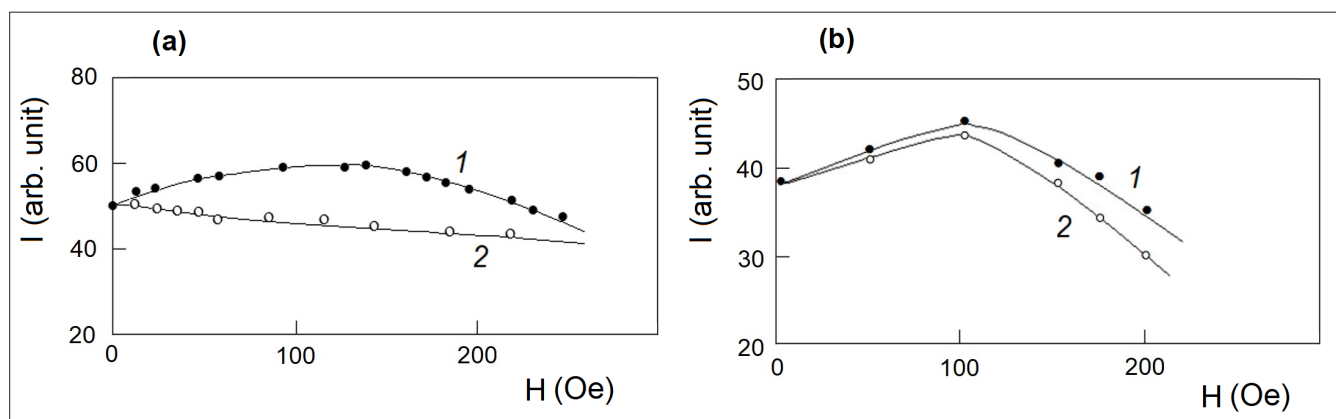


Figure 3: Dependence of the intensity I of the TPE on the external magnetic field H for its directions opposite (1) and along (2) the the direction of magnetization of the nanowire (a); similar dependences for the non-polarized sample (b).

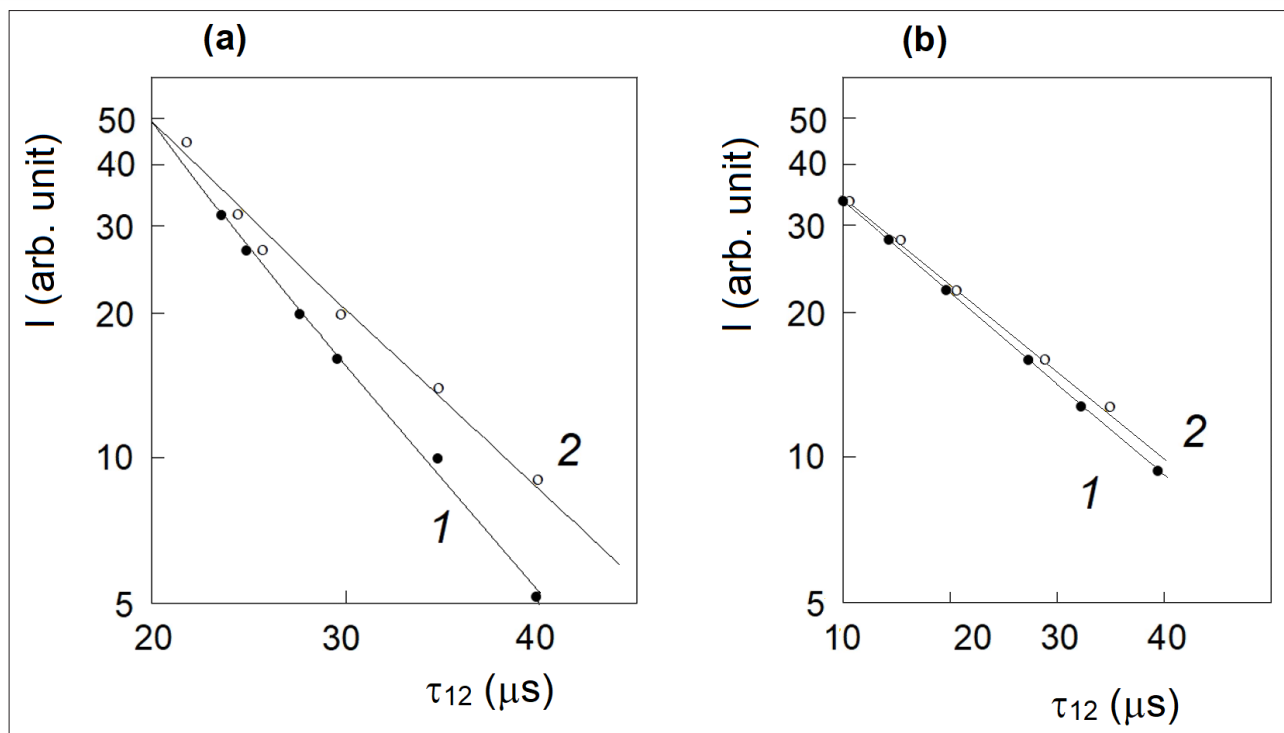


Figure 4: Dependence of the transverse relaxation rate of the TPE signal on the direction of the magnetic field H : opposite (1) or along (2) the magnetization of cobalt nanowires for the polarized sample (a); similar dependences for the non-polarized sample (b). Magnetic field value is $H=130$ Oe, τ_{12} is the time interval between RF pulses.

The increase in the intensity of the TPE signal is associated with an increase in the magnetic susceptibility χ_{dis} associated with the displacement of the DWs, which leads to an increase in the gain η_{eff} of the transverse RF field H_{RF} acting on the nuclei, $\eta_{\text{eff}} \sim A \cdot \chi_{\text{dis}}$, where A is the hyperfine coupling constant and an associated increase in the absorbed NMR power: $P \sim \eta_{\text{eff}}^2 H_{\text{RF}}^2$ (Turov, Petrov, 1972).

The decrease in the transverse relaxation time is apparently due to the growth of longitudinal hyperfine field fluctuations on the nuclei in the DW (Gavasheli et al., 2024; Turov & Petrov, 1972) with an increase in the external magnetic field in the direction opposite to the nanowire magnetization, due to the processes of electron magnetization reorientation, leading to additional dephasing of the transverse magnetization of the isochromats of the nuclei created by the action of the first RF pulse, and the associated decrease in the intensity of the TPE signal.

Comparison of experimental dependences obtained by methods of resonant RF magnetometry and NMR for polarized and non-polarized samples shows the possibility of their use for visual assessment of the degree of their self-assembly when they are polarized in an external magnetic field.

Conclusion

In this work, the process of self-assembly of pointed nanocomposites based on cobalt nanowires during their polarization in an external field in an epoxy matrix with cobalt nanowires oriented in an external magnetic field in an epoxy matrix was studied. For this purpose, the macroscopic method

of RF resonance magnetometry and the complementary microscopic NMR method were used. These methods provide interesting information on the degree of self-assembly of cobalt nanowires when polarized in a magnetic field. This information may be useful for improving the technology of manufacturing cobalt nanowires in sensors, memory devices, permanent magnets that do not contain rare earth elements, and other applications.

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