

NEUTRON REFLECTOMETRY STUDY OF TRANSFORMER OIL-BASED MAGNETIC FLUID UNDER ELECTRIC FIELD

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ABSTRACT

This paper is focused on characterization of magnetic fluids (MFs) and experimental investigation of interfacial structural changes in transformer oil-based magnetic fluids (TOMFs) under the influence of electric field. Recently, such nanodielectric fluids have been proposed to be used as transformer liquid insulation as the addition of particles seeks to improve the dielectric and thermal properties of the base liquids. Ferrofluids typically respond to electric and magnetic fields and can be easily manipulated by such external fields. By means of neutron reflectometry (NR), we show impact of the electric field intensity on magnetic nanoparticle aggregates in a dilute (1.8 vol %) classical ferrofluid.

Keywords: external fields, magnetic fluids, magnetic nanoparticles, transformer oil-based ferrofluids, solid-liquid interface, neutron reflectometry.

1. INTRODUCTION

Magnetic fluids (MFs) are suspensions of magnetic nanoparticles (MNPs) in a liquid carrier. The study of MF systems is of both fundamental and applied interest. They are widely used in various fields, in particular, they have great potential for use in engineering and medicine [1]. One of the key equipment in the electrification system is an electric transformer. During the transformer operation the heat energy is generated in the transformer windings and core. It is necessary to transfer the heat from the transformer. Recently, nanodielectric fluids have been proposed to be used as transformer liquid insulation. The addition of particles seeks to improve the dielectric and thermal properties of the base liquids, giving rise to a new generation of insulating fluids. Despite the large number of works devoted to the study of the structure of magnetic fluids in the volume of the sample [2-10], still there are properties and phenomena, the understanding of which and subsequent practical applications in electrical engineering require deeper experimental study. A mechanism of the streamer development leading to the electrical breakdown, polarization, macroscopic structural changes of magnetic fluids in external fields and nanoscopic structure at magnetic fluid-solid interface belong to the currently discussed issues. Such fields play an important role in numerous self-assembly phenomena.

The interaction of MNPs with external magnetic and electric fields can lead to different structural reorganizations of ferrofluids including the formation of aggregates, chains and more complex patterns. Effect of an external magnetic field and the induced heterogeneous nanoparticle structure results in the magneto-viscous effect

and magneto-dielectric anisotropy [2]. Also, at a high magnetic flux density, the neutron reflectivity analysis shows the formation of two effective adsorption layers with different content of MNPs in them [3, 4]. In [5] via specular neutron reflectometry (NR) it was observed three-dimensional self-assembly of spherical and monodisperse MNPs from a MF with volume concentration less than 1% onto a silicon surface under magnetic field. The structure of the subsequent layers varies to a greater extent as a function of solution properties, surface interaction characteristics and applied magnetic field [6]. Thus, the magnetic field intensity, direction and distribution plays a key role in the study of MF.

Analogously, structural transitions in various colloidal suspensions can be induced by electric fields leading to particle-particle electrostatic interactions. Study [7] showed that the dielectric response of MF is dependent on the strength and frequency of the electric field. It was demonstrated visually observable pattern formation in a transformer oil-based magnetic fluid (TOMF) exposed to a DC electric field. Also, ferrofluid viscosity increasing with the increasing electric field intensity (that is analogous to the magnetoviscous effect) is associated with the formation of chains or aggregates [8]. Also, the electric field induced changes in the ferrofluid structure at nanoscale were recently confirmed by in situ small-angle neutron scattering (SANS) experiments [9]. The obtained theoretical results from [10] show that under the sufficiently strong applied electric field the homogeneous distribution of dielectric particles in the dielectric sample could become inhomogeneous.

The aim of this paper is to shed the light on the interface structural changes in TOMF with magnetite nanoparticles

as well as the magnetic fluid-solid interfaces under electric fields application. An important question to answer is whether electric field could lead to self-assembly of magnetic nanoparticles at the interface. For this purpose, we have performed NR measurements of TOMF on planar silicon surface under the different homogeneous electric fields. Analysis of the structural arrangement was based on comparison of the experimentally obtained scattering length density (SLD) profiles to those calculated from assumption of a certain NPs packing in a particular layer.

2. MATERIALS

The investigated MFs are based on a commercially available inhibited insulating transformer oil SHELL Diala S4 ZX-I with such parameters: density 0.805 g/cm³ (20 °C), kinematic viscosity 9.6 mm²/s, pour point 233 K and flash point 464 K. Magnetite MNPs were synthesized by chemical co-precipitation method from aqueous solution of ferrous and ferric ions in the presence of NH₄OH at 80–82 °C. After coprecipitation, the iron oxide NPs were sterically stabilized by chemisorbing of a single oleic acid layer (C₁₈H₃₄O₂, Merk) on the particle surfaces. Synthesis of MF was performed according to previous reports [11, 12]. Sample with the magnetic volume fraction of 1.8% (mass fraction 9.3 %) was studied.

The magnetic properties of the MF sample were measured by means of a vibrating sample magnetometer installed on a cryogen-free superconducting magnet from Cryogenic Limited (IEP SAS, Kosice). The obtained magnetization curve measured at 298 K in the field ranging up to 6 T and its fit are shown in Fig. 1. The magnetization of the diluted magnetic fluid can be expressed as a superposition of the Langevin functions:

$$M = M_S \int_0^\infty L(\xi) p(x) dx \quad (1)$$

where M_S is the saturation magnetization, $L(\xi) = \coth(\xi) - 1/\xi$ is the Langevin function with $\xi = \mu_0 \mu H / k_B T$, μ_0 is the magnetic permeability of vacuum, H is the magnetic-field strength, k_B is the Boltzmann constant and T is the absolute temperature. The mean particle size derived from the fitting of the magnetization curve by the superposition of Langevin functions is 10.3 nm.

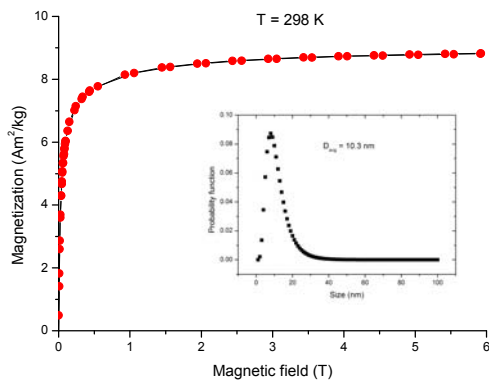


Fig. 1 Magnetization curve of the MF. Inset 1: distribution of MNPs in MF calculated from the magnetization

3. METHODS

Neutron reflectometry measurements were performed at the GRAINS instrument of the IBR-2 pulsed reactor (FLNP, JINR). GRAINS is a multifunctional reflectometer with the horizontal sample plane,[19] operating in the time-of-flight regime. For both X-ray and neutron reflectometry, the reflectivity was plotted as a function of the momentum transfer q :

$$q = \left(\frac{4\pi}{\lambda}\right) \sin(\theta) \quad (2)$$

where: θ – is the fixed (5 mrad) incident grazing angle and λ is the neutron wavelength varied in the interval of 0.1–0.6 nm (cold mode of the moderator at IBR-2).

The calibration for the incident spectrum was made by measuring the empty beam. The NR measurements were carried out in the ‘beam from the bottom’ configuration in the time-of-flight mode that is standard positioning to study the solid-liquid interfaces when a plane neutron beam passes through a single-crystal silicon substrate (thickness about 10 mm) to meet and scatter from the interface between a liquid sample and electrode film (thickness below 1000 Å) prepared on the substrate. The electrode was represented by a copper layer on the single crystal parallelepipedic silicon block (Holm GmbH) with dimensions $8.5 \times 5 \times 1.5$ cm³ and roughness <0.5 nm for the working surface (plane orientation <111>). The silicon monocrystal is applied for guiding the neutrons to the interface. Metal-layered electrode with Cu (500 Å) was used to reach the most suitable and possible (considering the instrument features) neutron contrast conditions for the reflection.

The specialized experimental cell for the proposed experiment has already been prepared and successfully applied before [13]. To apply the voltage on the electrodes, control the intensity and form of the signal, a signal generator (Agilent 33210 A) was employed. The sample in the experimental cell was exposed to various direct current (DC) voltage (0, 60, 300 and 600 V), which are close to the operating values commonly used in high voltage power engineering.

The X-ray reflectometry (XRR) measurements of pure crystal with electrode were made using reflectometry mode of the Empyrean diffractometer (Malvern PANalytical, Almelo, the Netherlands) at FLNP (Dubna, Russia). The wavelength used was 1.54 Å from the Cu source. The measurements were carried with the fixed sample position and orientation. The range of measured angles was chosen to cover the reflectivity fringes until they were smoothened by the quality of the interfaces.

Motofit package [14] of the IGOR Pro environment (www.wavemetrics.com), was used for modelling of the reflectivity curves. The characteristics of the formed layers at the interface were obtained in a homogeneous approximation.

The parameters for each layer include the thickness, interlayer roughness and mean nuclear scattering length density (SLD):

$$\rho = \frac{\rho_m N_A}{M} \sum n_i b_i \quad (3)$$

where b_i is an atomic incoherent scattering length, M - atomic mass, ρ_m material density, N_A - Avogadro's number.

For X-ray scattering SLD is:

$$\rho = \frac{\rho_m N_A r_e}{M} \sum n_i Z_i \quad (4)$$

where r_e is radius of electron and Z_i is atomic number of a chemical element.

Theoretical SLD values of electrode and MF materials are presented at Table 1.

Table 1 SLD values of electrode and MF materials

material	density (g/cm ³)	SLD (10 ⁻⁶ Å ⁻²) neutron	X-ray
Si	2.65	2.07	22.7
SiO ₂	2.2	3.57	18.9
Cu	8.96	6.55	64.7
Cu ₂ O	6	5.36	44.2
CuO ₂	2.77	3.38	21.2
CuO	6.61	6.77	49.5
Fe ₃ O ₄	5.15	6.91	40.3
MF	0.908	0.2	9.7
oil, oleic acid	0.895	0.083	9.1

The reflectivity is calculated using the Abeles matrix method for stratified interface. Although the scattering length density profile is normally a continuously varying function, the interfacial structure can often be well approximated by a slab model in which layers of thickness (d_n), SLD (ρ_n) and roughness ($\sigma_{n,n+1}$) are sandwiched between the super- and sub-phases. One then uses a refinement procedure to minimise the differences between the theoretical and measured reflectivity curves, by changing the parameters that describe each layer. In this description the interface is split into n layers. Since the incident neutron beam is refracted by each of the layers the wavevector, k , in layer n , is given by:

$$k_n = \sqrt{k_0^2 - 4\pi(\rho_n - \rho_0)} \quad (5)$$

Since the interface between each layer is unlikely to be perfectly smooth the roughness/diffuseness of each interface modifies the Fresnel reflection coefficient between layer n and $n+1$:

$$r_{n,n+1} = \frac{k_n - k_{n+1}}{k_n + k_{n+1}} \exp(-2k_n k_{n+1} \sigma_{n,n+1}^2) \quad (6)$$

A phase factor, β is introduced, which accounts for the thickness of each layer as $\beta_n = k_n d_n$.

A characteristic matrix, c_n is then calculated for each layer:

$$c_n = \begin{bmatrix} \exp(\beta_n) & r_n \exp(\beta_n) \\ r_n \exp(-\beta_n) & \exp(-\beta_n) \end{bmatrix} \quad (7)$$

The resultant matrix is defined as the product of these characteristic matrices, from which the reflectivity is calculated:

$$R = \prod_0^n c_n \quad (8)$$

4. RESULTS

X-ray reflectometry measurements for pure crystal with electrodes were made for using the results of fit as a preliminary information for better treatment of neutron data. During the experiment, the crystal was fixed at normal conditions. The obtained experimental dependencies are plotted in Fig. 2, where the presence of layered structures is clearly seen because of oscillations. Gained SLD profile (Fig. 3) confirm the passport thickness and roughness of elements but also reveal existing of silicon oxide and copper oxide layers with thickness about 5 nm and 17 nm respectively and roughness 0.4-0.9 nm.

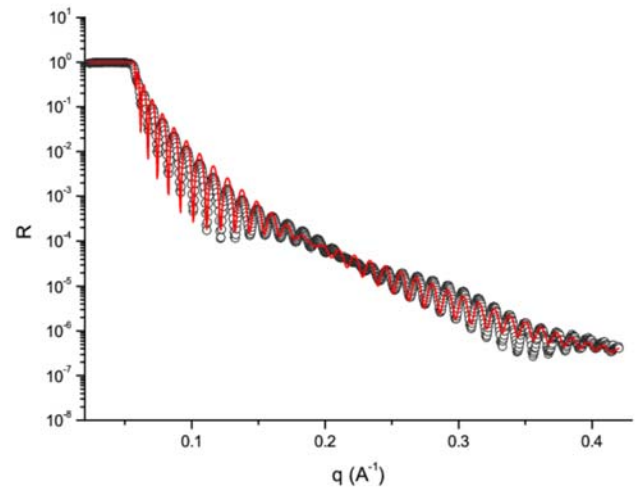


Fig. 2 XRR data taken from crystal with electrodes without MF and without electric field. The solid lines represent fits to the data

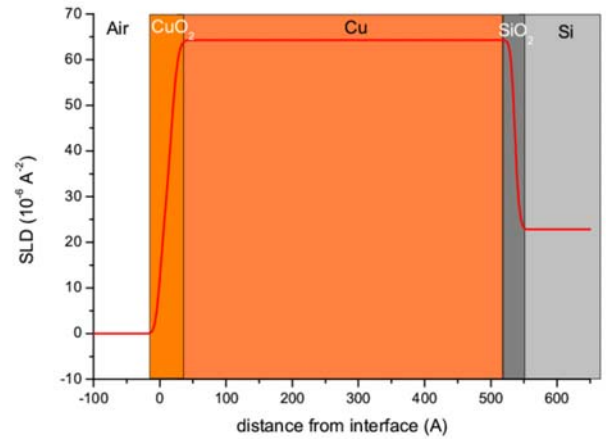


Fig. 3 Profiles of SLD plotted as a function of distance z from the air determined from the results of the fits to the XRR data taken from crystal with electrodes without MF and without electric field and shown in Fig. 2

Experimental NR measurements were performed for a cell fully filled with the ferrofluid. During the experiment, the sample was fixed and only the intensity of the applied electric field was gradually varied. The obtained data and fit for the initial state (without electric field) are presented in Fig. 4. Since in the experiment neutrons passed from an optically denser medium towards a less dense medium, the

reflectivity curves do not have any total reflection region and show a typical power-law decrease modulated to various extents by different kinds of the SLD depth profile. Thus, in the simplest case, when incident neutrons are reflected from the boundary between two homogeneous semi-infinite media, the reflectivity decrease is monotonous and has an asymptotic behavior of $\sim q_z^{-4}$ (Fresnel law). However, if there is at least one optically different layer between the semi-infinite media, oscillations in the reflectivity curves appear.

The smallest χ^2 -values were achieved if the parameters of system are varied within narrow (compared to the final error) intervals around the nominal value. The corresponding SLD profile is presented in Fig. 5 according to the parameters from the Table 2 ($\chi^2=0.007$). The discrepancies between the SLD values in the table and Fig. 5 is due to the influence of roughness, which for some layers is higher than their thickness.

Table 2 Parameters of the layered structure of the crystal and liquid component (MF) in the SLD profile of the interface without electric field derived from neutron reflectivity curves in Fig. 4. Errors are in the last digit given

layer	thickness (Å)	SLD (10^{-6} Å^{-2})	roughness (Å)
fronting		2.38	
1	10.8	4.06	35.5
2	555.8	4.36	26.2
3	16.8	3.23	87.8
4	47.7	3.63	71.6
5	27.1	0.13	2.7
6	106.8	5.86	49.1
backing		0.62	44.7

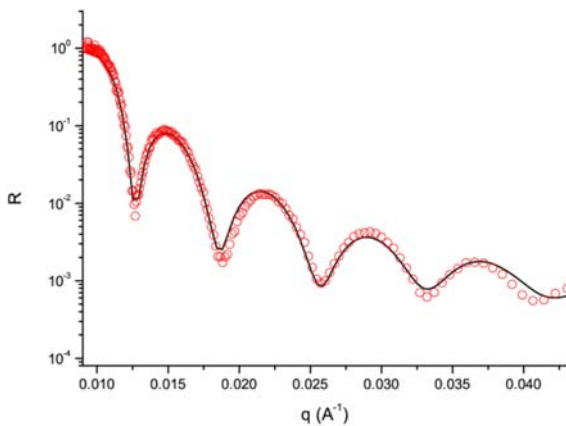


Fig. 4 NR data taken from crystal with electrodes and MF without electric field. The solid lines represent fits to the data

Compared to XRR neutrons results show more part of copper oxides in electrode film. For the initial state the good fits are obtained when assuming one 'wetting' layer of MNPs between the surface and bulk MF which means 3 layers on the SLD profile. The first layer ('buffer') is associated with the average size of a coating around MNP absorbed on the substrate. This reflects the natural fact that MNP cannot come to the Si surface closer than the thickness of the coating. The found thickness of the first layer corresponds well to the effective ~ 1.5 nm thickness

of the oleic acid monolayer on magnetite nanoparticles, which is known from the small-angle neutron scattering research [15], and its low SLD is consistent with SLD of oleic acid or its possible mixture with the oil. The next two layers of MF are characterized by higher thickness and SLD values, which is indicative of a significant fraction of magnetite in them. The second layer size (10.7 nm) with the roughness of 5 nm is in a good agreement with the NPs diameter value gained from magnetization measurements. It is possible to estimate the mass fraction of magnetite in the layer, ϕ_m , using the equation for the mean SLD of the layer:

$$SLD = \phi_m SLD_m + (1 - \phi_m) SLD_s \quad (9)$$

where, SLD_m , SLD_s are SLD of magnetite and solvent (surfactant), respectively. It gives for bulk $\phi_m = 7.9\%$, thus showing about 10 times larger concentration of MNP in the adsorption layer as compared to the bulk.

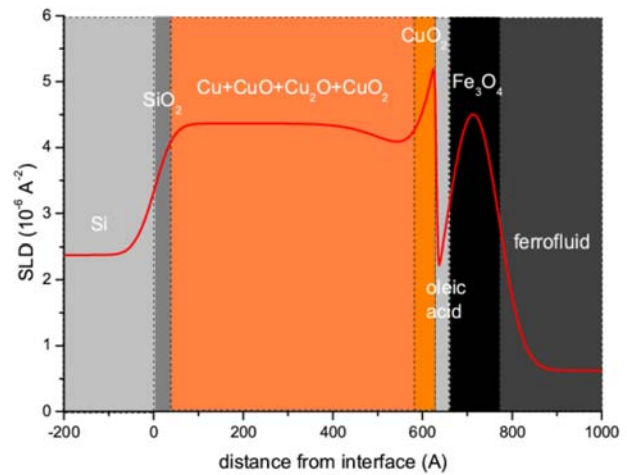


Fig. 5 Profiles of SLD plotted as a function of distance z from the Si(100) surface determined from the results of the fits to the NR data taken from crystal with electrodes and MF without electric field and shown in Fig. 4

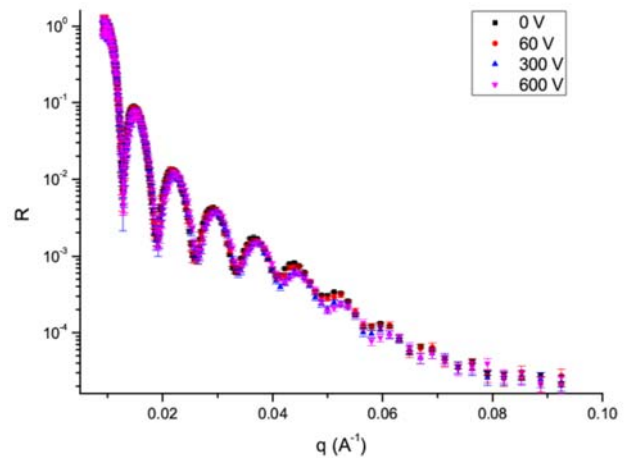


Fig. 6 NR data taken from MF when applying electric field from 0 V to 600 V

The obtained experimental NR curves for different electric fields applied are presented in Fig. 6, where the NR effect is clearly observed. The best result for smaller

electric field was used as initial configuration for higher voltage. One can see that with increasing of electric field intensity become smaller and all curves slightly go right in the range of greater q . It also reflects in the SLD profiles gained from the fit (Fig. 7). It can be seen that with application of electric field adsorption layer become more concentrated and magnetite NPs lower from bulk MF. Starting from 300 V the SLD value derived from the model calculations performed for 1 wetting layer can no longer describe the experimental data. Instead, new layer could be recognised between initial layer and bulk and reorganisation continue with increasing voltage. The second adsorption layer is almost 2 times thicker but about 7 times less concentrated than an initial one. The effect could be associated with the migration of some small space charge, which engenders a weak vertical fluid flow. And it is similar with situation with applying magnetic field [3, 5], when at a higher magnetic flux density, the analysis of SLD gives the formation of two effective adsorption layers with different content of magnetic nanoparticles in them. In that case the shape anisotropy generates a force that twists the elliptical particles and reorients them with their major axis parallel to the sample surface (Fig. 8).

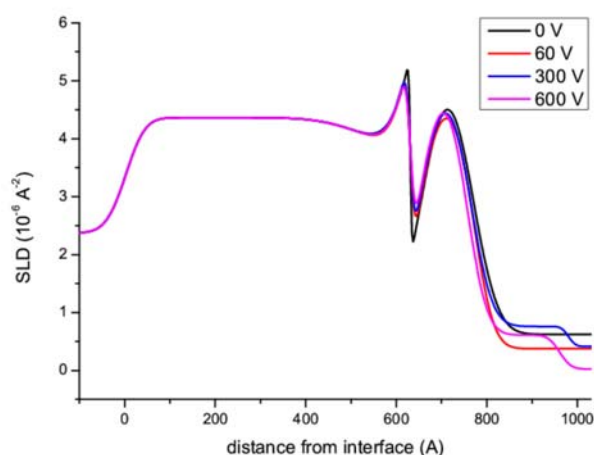


Fig. 7 Profiles of SLD plotted as a function of distance z from the Si(100) surface determined from the results of the fits to the NR data taken from MF at electric field from 0 V to 600 V and shown in Fig. 6

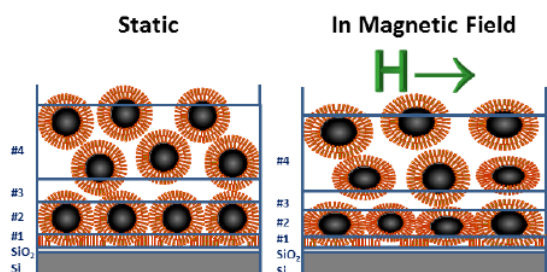


Fig. 8 Schematics showing the layers that form in static and magnetic fields for a MF in direct contact with a SiO₂ hydrophilic surface [5]

5. CONCLUSIONS

We report self-assembling of superparamagnetic polydisperse (10.3 nm) nanoparticles in magnetic fluid - magnetite coated with oleic acid in transformer oil. Forming of NPs layers was found under perpendicular homogeneous electric field by neutron reflectometry. At the electrode surface in the low electric field, we observed only wetting MNPs layer with size $\sim$ magnetite NPs. From electric field 300 V additional MNPs layer was detected and their parameters was calculated by comparing the measured SLDs of MF components. The reason of such development is polarization of the particles and their interaction as dipoles. The observed self-assembled layering could be used as additional barrier at the inner surface of transformer to increase dielectric breakdown voltage of working fluids layer and advance heat transfer. As the next step for using of such materials in electric power engineering investigation of temperature effect on MF in electric field is needed.

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