DYNAMIC SAXS CORRELATION AS METHOD OFSTRUCTURAL RESEARCH

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Composite-type material consisted of polyurethane gel, and carbonyl iron (CI) of 7 μm size spheres. The gel constituents were: MDI/OAE+DCDA with molar ratio 70/30. The material was processed with 11.5 vol.% of CI under magnetic field, during 24 h at 25ºC.

SEM images of the composite showed short chains of CI formed inside the material.

- XPCS (X-ray Photon Correlation Spectroscopy) measurements were performed on the Troika beamline (ID10A) of the ESRF in SAXS configuration. The meaurements depending on time (t) and angle (q), using X-beam wavelength of 1.745 Å, and detector type CCD were used. The sample was placed between electromagnet poles with magnetic lines direction perpendicular to the X-beam and parallel to the sample surface and CI chains inside it. Measurements were performed for: 0, 300 and 600 mT. It occurred that correlation of scattered intensity took place in the time range: from 5×10^2 to 100×10^2 sec.

- *Correlation curves, $g_2(t)$:* The correlation function $g_2(t)$, according to the formula $g_2(t)=\langle I(t_o+t)I(t_o)\rangle/\langle I(t_o)^2\rangle$ was obtained from $I(t, q)$ CCD measurements using masks to get them in directions: parallel (par), isotropic (iso) and perpendicular (per) to the magnetic field lines.

- The reference curves at 0 mT are of extremely slow dynamics behavior with correlation reached for about 10^4 sec and of weak differentiation between the measurement directions. For 300 mT, the correlation is faster (~ 1.2×10^3 sec) with the dependence on different directions. For 600 mT the correlation is the fastest, of less than 10^3 sec.

- *Correlation rates, $\Gamma(q)$.* The correlation rates, $\Gamma(q)$, were found by fitting exponential function: $y = a\, e^{2\Gamma x}$ to the subsequent $g_2(t)$ curves, in their region of correlation behavior. Fig. 1 shows $\Gamma(q)$ curves for subsequent magnetic field value, with sets of three curves for each directions (par, iso and per). When magnetic field increases, from 0 through 300 to 600 mT, correlation rate also increases. It occurred that the magnetic field essentially influences the correlation rate, up to ten times (Fig. 1). Besides, within each set the highest curve is for direction parallel to the magnetic field (par), the lowest one for direction perpendicular (per) and the middle one is for isotropic (without mask) measurement. The straight-line shape of all $\Gamma(q)$ curves are of no significant deviations. The correlation process is thus not due to structural reorganization on measured length scale.

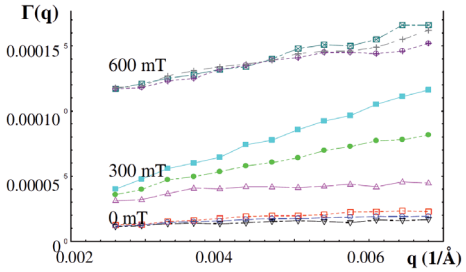

Figure 1. Correlation rates $\Gamma(q)$ for different magnetic field values. At each three curve set: bottom – perpendicular, middle – isotropic, top – parallel to the field lines.

In the case of linear $\Gamma(q)$ shape, the dependence it on diffusion coefficient is: $\Gamma(q) = D \times q$. The D [Å/sec] values were found from slope of straight lines fitted to $\Gamma(q)$ (Table 1).

- For the reference curves for 0 mT the very moderate values of the $\Gamma(q)$,and D as well as no differentiation from direction seem to be an evidence of very strong structure.

- For the middle value of the field (300 mT), the same parameters are strongly dependent on the magnetic field direction and are of much bigger values (Fig. 1 and Table 1). Here dependence on the material dynamics from direction of the outer magnetic field is very clear, with strongly privileged parallel direction (par).

- But for the magnetic field of 600 mT, despite the highest correlation rate, the dependence on magnetic field direction became weak, as well as D values (Fig. 1 and Table 1). Simultaneously, on the $\Gamma(q)$ curves the bigger fluctuation around the fitted straight lines are observed. There probably mechanism of dynamics behavior altered.

Table 1. Diffusion coefficients, D [Å /sec].

	0 mT	300 mT	600 mT
Par	0.0027	0.0180	0.0119
iso	0.0018	0.0109	0.0096
per	0.0011	0.0029	0.0083

Reference

[1] J. Lal, D. Abernathy, L. Auvray, O. Diat, G. Gruebel, "Dynamics and correlations in magnetic colloidal systems studied by X-ray photon correlation spectroscopy", *Eur. Phys. J.* **E4** (2001) 263.