Experimental report

21/05/2019

Proposal: **1-04-130**

Council: 4/2017

Title: Investigation of magnetic field induced structuring of soft magnetoactive elastomers.

Research area: Soft condensed matter

This proposal is a new proposal

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Samples: silicone rubber filled with iron particle
Magnetorheological fluid based on a polycarbosilane matrix and iron microparticles

Instrument	Requested days	Allocated days	From	To
S18	10	10	18/06/2018	28/06/2018

Abstract:

The purpose of this proposal is to provide experimental evidence for formation of chain-like magnetic-particle structures in highly filled magnetorheological (MR) elastomers (MREs) and magnetorheological fluids under external magnetic fields. The elastomeric samples will consist of carbonyl iron particles (diameter about 4 - 5 µm) embedded into a compliant silicone matrix. Another set of samples will consist of MR fluids based on a polycarbosilane matrix (or similar) and iron microparticles. Using MR fluids, it can be verified if the formation of chains depends on the carrier medium. Similarly to MR fluids, the change in MR properties is thought to be due to the magnetic polarization induced in each particle by the external field, with the resulting interaction forces between the particles leading to the formation of elongated aggregates in the direction of the field. If an external magnetic field is applied to the MRE composite, the magnetic filler particles may move inside the compliant polymer matrix and build magnetic-network aggregates. We are interested in verifying this hypothesis.

Experimental Report to the proposal 1-04-130 “Investigation of magnetic field induced structuring of soft magnetoactive elastomers”.

Following our proposal we fabricated several samples of compliant magnetoactive elastomers (MAEs) with the same silicone matrix and different iron (Fe) content: 10 wt%, 30 wt%, 60 wt% and 80 wt%. We also used a sample with 67 wt% of iron and somewhat harder elastomer matrix. Additionally, we had a sample of an empty elastomer (matrix) and several MAE samples fabricated by colleagues from State Institute of Chemistry and Technology of Organoelement Compounds and Lomonosov State University in Moscow, Russia. They are quite similar to samples from the OTH Regensburg but are fabricated using different technology and also included magnetite (Fe_3O_4) filled specimens. They were intended to be used for comparison. Due to the lack of experimental time, theses samples were not investigated.

Due to the previous experience obtained from a preliminary experiment made at S18 instrument at the ILL, the thickness of the fabricated samples was drastically reduced. This has been done in order to increase transmission and to reduce the influence of multiple scattering. The resulting transmission of all filled samples was more than 90%. Table 1 gives the overview of the samples from the OTH Regensburg used in experiments.

Table 1.

Sample	Mass fraction of Fe, %	Volume fraction of Fe, %	Thickness, mm
Matrix	0	0	0.70
MAE10	10	1.3	0.35
MAE30	30	5.0	0.30
MAE60	60	15.5	0.20
MAE67	67	19.6	0.26
MAE80	80	32.9	0.30

First, we assembled an experimental setup incorporating an ILL-build electromagnet with a power supply (see Figure 1). The electromagnet required external water cooling. The maximum magnetic flux density achievable with the electromagnet was equal to 0.35 T. This magnitude of the magnetic field allowed us to observe the restructuring effects, but it is relatively low, since large magneto-mechanical effects are expected in MAEs at flux density of 500 to 600 mT. In our previous experiments on the KWS-3 setup in Garching, significant restructuring effects were observed at approximately 500-600 mT and came to the saturation in larger fields (up to 2 T).

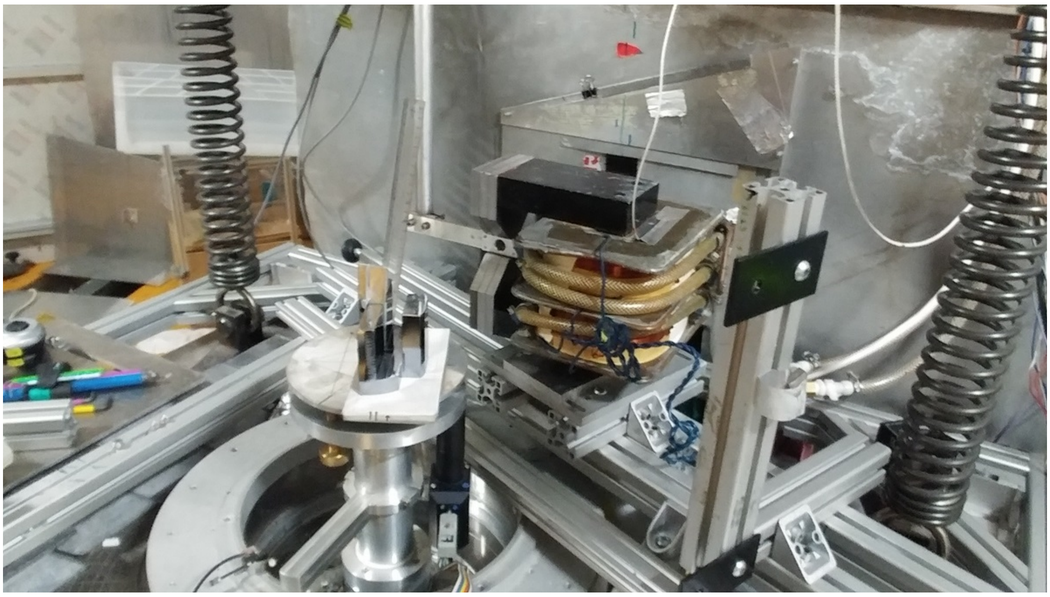

Figure 1. A photograph of the experimental setup. A sample holder between the poles of an electromagnet is seen in the center of the picture. The water cooling is also clearly seen.

The scattering experiments were made in the field of 0, 200 and 350 mT. The filled samples and the empty polymer matrix have been measured. The measurements of the polymer matrix were required in order to eliminate its influence on the experimental results. All investigated filled samples demonstrated the dependence of scattering curves on the magnetic fields. As expected the maximum effect was observed in the maximum magnetic field of 350 mT. Finally, we measured the hysteresis behavior of the MAE80 sample, which we expected from our magnetorheological and magnetodielectric measurements The field was first increased in two steps to the maximium field and the decreases to the zero in two steps. Two such cycles have been recorded. We believe that we found the confirmation of hysteresis behavior in scattering curves obtained.

It is probably too early to make quantitative conclusions from our experiments, but we can now claim that we observed strong influence of a magnetic field on the USANS characteristics of MAE samples. The evaluation of data and interpretation of the results are currently under way. The challenge is that the scattering curves do not easily fit into the most of conventional modes, we observe a local maximum in the Q-region of approximately $(2 – 5)\times10^{-4}$ Å^{-1}.

It became also clear which experiments are necessary and should be made next. First, one has to be able to apply larger magnetic fields (ideally, up to 800 mT) in order to increase the observed effect. This could be achieved, for example, by bringing our own electromagnet from Regensburg and installing it at S18. Second, one could produce MAE samples with smaller particles (≤ 1 μm) in order to better use the working range of the S18 instrument. Third, one should fabricate MREs with the same concentration of the filler but different rigidity of the polymer matrix. To investigate the influence of anisotropy of the filler, isotropic and anisotropic samples must be compared. To summarize, the first experimental results on MAEs obtained at S18 instrument look promising and pave the way for further experimental and theoretical advances.