SAXS investigations on shear orientated nanoparticle lattices

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The formation of oriented lattices such as face centred cubic (fcc) is well known from block copolymer gels [1]. We succeeded to create a similar gel system with Fe_xO_y nanoparticles surrounded by polystyrene with a diethylentriamine anchor group. This nanocomposite is forming gels in a certain concentration range in toluene. Depending on the molecular weight of the polystyrene and the characteristics of the nanoparticle, fcc or body centred cubic (bcc) lattices are formed. Furthermore the distance of the nanoparticles can be varied by using polymers of different molecular weight.

With reference to previous experiments performed at the HASYLAB [2] we succeeded to characterise various Fe_xO_y nanocomposite gels under application of defined shear with a Linkam shear cell CSS450 (figure 1).

The characterisation of the gels has been done by small-angle x-ray scattering (SAXS). For the measurements we used the SAXS setup of the beamlines A2 and BW4 and a 2-dimensional CCD detector at a distance of 2 m behind the sample. A volume of some hundred µL sample was placed in the shear cell. The orientation was achieved with large amplitude oscillatory strain (LAOS) or shear. The Shear cell was controlled from outside the hutch and allowed in-situ measurements. Due to the µ-focus beam at the BW4 high resolution small-angle diffraction patterns could be determined.

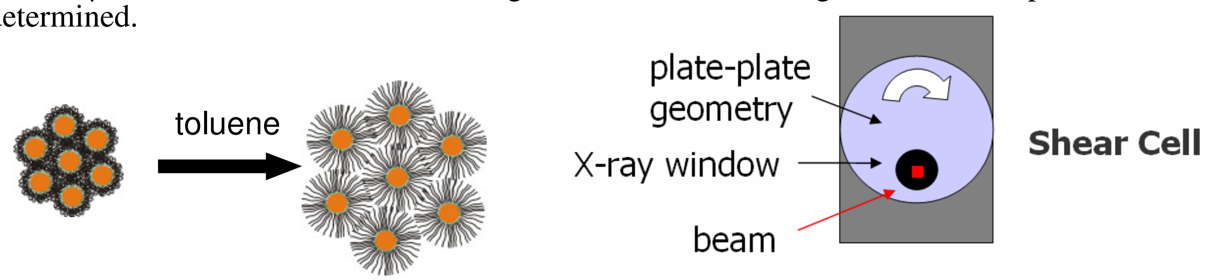

Figure 1 Left: Formation of a gel. Right: Application of shear stress with a Linkam shear cell on the sample.

Table 1 gives an overview on the used functionalised polystyrenes obtained by living anionic polymerisation.

	M(PS) [g/mol]	n (styrene)
PS-1	3300	31
PS-2	7600	72
PS-3	16100	153

Table 1: Molecular weight and number of monomers for the used polystyrenes.

Figure 2 shows the anisotropic patterns for Fe_xO_y nanoparticles (D = 15.4 nm) surrounded with functionalised polystyrenes of different molecular weights.

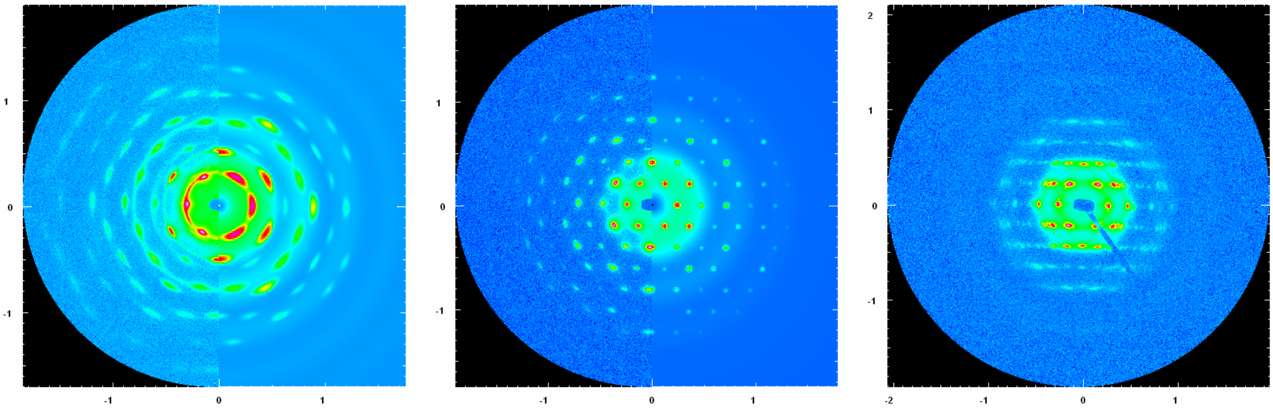

Figure 2 Left to right: Patterns of nanocomposite gels of Fe_xO_y (15.4 nm) with PS-1, PS-2 and PS-3 after oscillation. For the first two patterns the right side shows model calculation.

Figure 3 shows the anisotropic patterns for Fe_xO_y nanoparticles (D = 5.2 nm) surrounded with functionalised polystyrenes. For the left image no accurate hkl for the orientation could be determined. The isotropic sample indicates a bcc lattice.

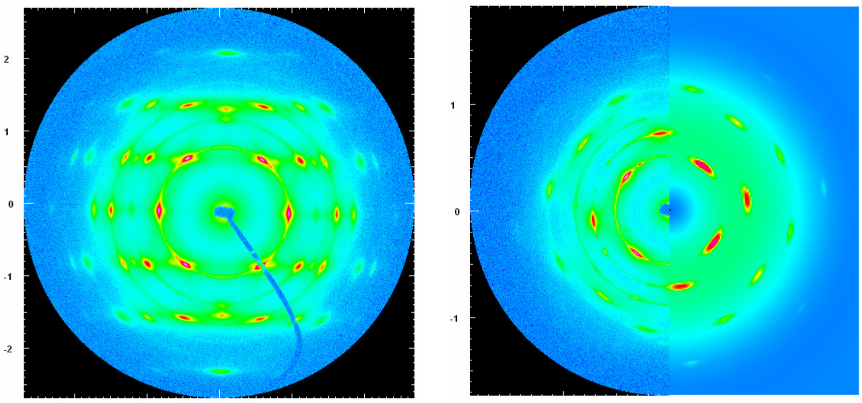

Figure 3: Left to right: Patterns of nanocomposite gels of Fe_xO_y (5.2 nm) with PS-1 (under shear), PS-2 (after oscillation) with a model calculation on the right side.

The obtained anisotropic pattern can be used to determine the lattice type, lattice constant, and nearest neighbor distance (NND). For the characterization the *Scatter* software was used [2].

	Lattice type	Lattice constant (nm)	NND (nm)
Fe_3O_4 PS-1 (15.4 nm)	fcc	35	25
Fe_3O_4 PS-2 (15.4 nm)	fcc	44	31
Fe_3O_4 PS-3 (15.4 nm)	bcc	36	31
Fe_3O_4 PS-1 (5.2 nm)	bcc	14.1	12.2
Fe_3O_4 PS-2 (5.2 nm)	bcc	17.3	15.0

These promising results show the possibility to enlarge the nearest neighbor distance with greater functionalized polystyrenes. Various anisotropic patterns could be obtained and determined. The nanocomposite Fe_xO_y (D = 15.4 nm) with PS-3 and Fe_xO_y (D = 5.2 nm) with PS-1 showed anisotropic patterns which needs further study.

References

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