Soft Matter

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Cite this: DOI: 10.1039/c0xx00000x

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ARTICLE TYPE

Shear-induced macroscopic "Siamese" twins in soft colloidal crystals

Supporting Information

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Received (in XXX, XXX) Xth XXXXXXXXX 20XX, Accepted Xth XXXXXXXXX 20XX
DOI: 10.1039/b000000x

Scanning Rheo-SANS

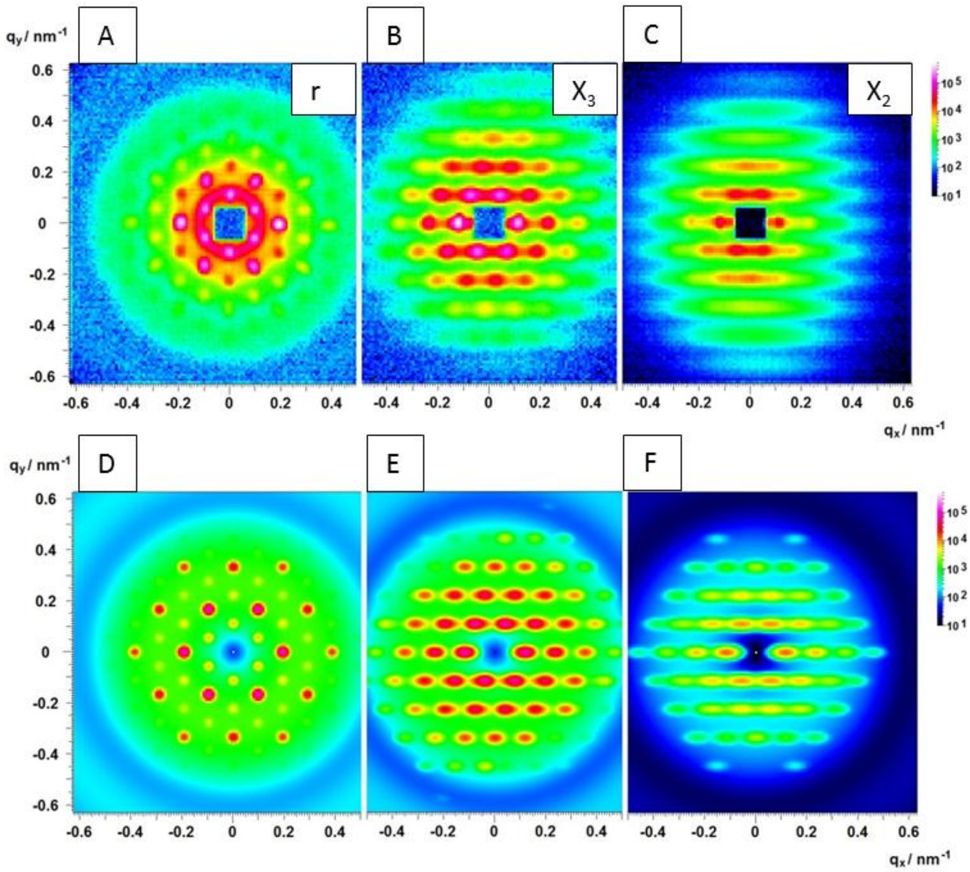

Fig. S1: Rheo-SANS scattering patterns of a shear-oriented FCC twin phase of a 13%wt PI(62)-PEO(193) in D_2O, presheared at 500 s^{-1}. **(A)** Measured scattering pattern in the "radial" beam position probing the [111]-direction. **(B, C)** Measured scattering patterns in the "tangential" beam position probing the [110]-direction at two different positions x_2 and x_3 in the gap. The distorted hexagonal array of the first-order reflections in **(B)** indicates the presence of twin "B" at the outer position x_3 of the gap of the Searle cell. The measurement at x_2 was taken close to the inner rotor of the cell and indicates the position of the twin boundary layer. The corresponding model calculations are shown in the lower panel.

Fig. S1 shows measured rheo-SANS patterns of a 13% PI(62)-PEO(193) solution after shearing at 500 s^{-1} and sudden cessation of shear. Fig. S1A displays the measured scattering pattern in the radial sample position, probing the [111]-direction of the lattice. A calculated scattering pattern is shown in Fig. S1D. The model calculation using Eq. (1) allows one to obtain the characteristic structural parameters of the FCC crystal. We observe a unit cell dimension of a = 70 nm (mean displacement σ_a = 0.03) and micellar core radius of R = 12 nm (mean rel. std. dev σ_R = 0.07). The peak shapes can be well described by an anisotropic Gaussian peak shape with longitudinal coherence lengths of l_{210} = l_{110} = 200 nm and l_{111} = 90 nm as derived from the Gaussian peak widths σ_{210} , σ_{110} and σ_{111} via Eq. (15). The calculated scattering patterns in Figs. S1E,F are calculated for the [110]-direction assuming nearly exclusively twin "B" (r=0.1, S1E) and twin coexistence (r=0.5, S1F) using the same structural parameters as in Fig. S1D.

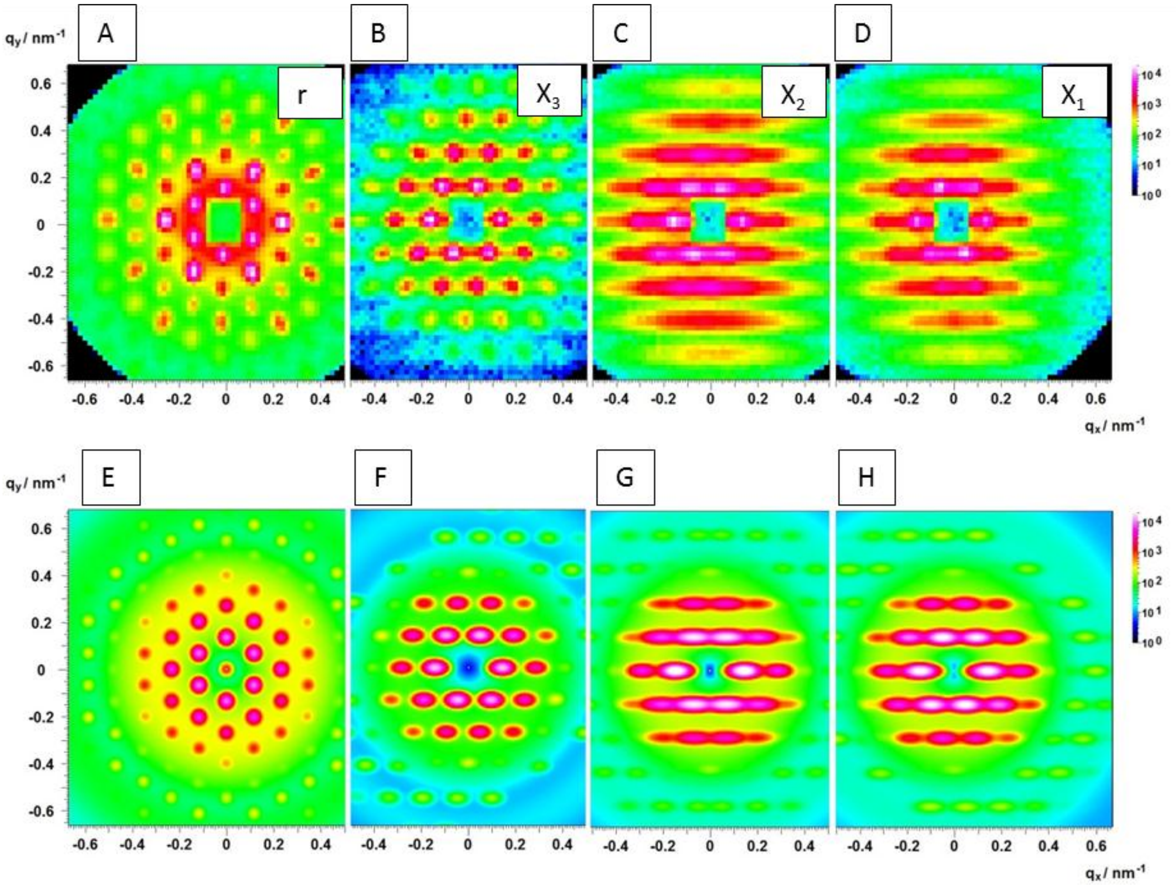

Fig. S2: Rheo-SANS scattering patterns of a shear-oriented FCC twin phase of a 15%wt PI(55)-PEO(170) in D_2O, presheared at 500 s^{-1} **(A)** Measured scattering pattern in the "radial" beam position probing the [111]-direction. **(B, C, D)** Measured scattering patterns in the "tangential" beam position probing the [110]-direction at three different positions $x_1 - x_3$ in the gap. The distorted hexagonal array of the first-order reflections in **(B)** indicate the presence of twin "B" in the outer region (position x_3) of the gap of the Searle cell. The measurement at x_2 **(C)** was taken in the middle of the gap indicating the position of the twin boundary layer. The measurement at x_1 **(D)** was taken close to the inner rotor in the gap indicating the presence of twin "A". The corresponding model calculations are shown in the lower panel **(E, F, G, H)**.

Fig. S2 shows measured rheo-SANS patterns of a 15% PI(55)-PEO(170) solution after shearing at 500 s^{-1} and sudden cessation of shear. Fig. S2A shows the measured scattering pattern in the radial sample position, probing the [111]-direction of the lattice. A calculated scattering pattern is shown in Fig. S1D. The model calculation using Eq. (1) allows one to obtain the characteristic structural parameters of the FCC crystal. We observe a unit cell dimension of a = 70 nm (mean displacement σ_a = 0.03) and micellar core radius of R = 12 nm (mean rel. std. dev σ_R = 0.07). The peak shapes can be well described by an anisotropic Gaussian peak shape with longitudinal coherence lengths of l_{210} = l_{110} = 200 nm and l_{111} = 90 nm as derived from the Gaussian peak widths σ_{210} , σ_{110} , and σ_{111} via Eq. (15). The calculated scattering patterns in Figs. S2F-H are calculated for the [110]-direction assuming twin "B" (r=0.0, S1F), twin coexistence (r=0.5, S1G), and twin "A"-dominance (r=0.8, S1H) using the same structural parameters as in Fig. S1E.

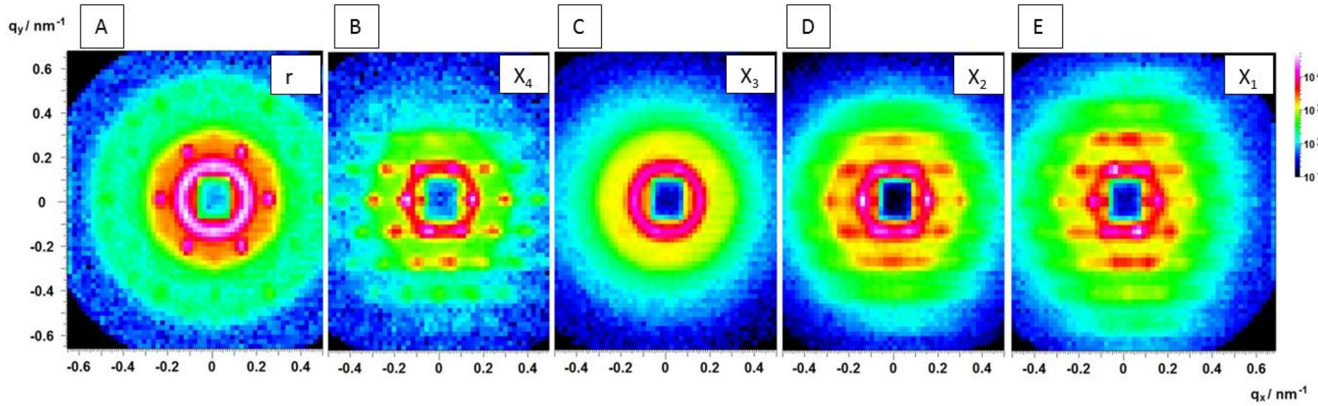

Fig. S3: Rheo-SANS scattering patterns of a shear-oriented FCC twin phase of a 16%wt PI(55)-PEO(170) in D_2O, presheared at 200 s^{-1} **(A)** Measured scattering pattern in the "radial" beam position probing the [111]-direction. **(B, C, D, E)** Measured scattering patterns in the "tangential" beam position probing the [110]-direction at four different positions $x_1 - x_4$ in the gap. The distorted hexagonal array of the first-order reflections in **(B)** indicate the presence of twin "B" at the outer position x_4 of the gap of the Searle cell. The measurement at x_3 **(C)** was taken further in the middle of the gap. We observe Debye-Scherrer rings indicating an irregular arrangement of twinned domains. In the subsequent position x_2 **(D)** both twins are observed in their oriented state. Close to the inner rotor **(E)** at position x_1 we observe twin "A". This measurement shows that high pre-shearing rates of > 200 s^{-1} are required to induce macroscopic twin growth.

Fig. S3 shows measured rheo-SANS patterns of a 16% PI(55)-PEO(170) solution after shearing at 200 s^{-1} and sudden cessation of shear. We observe the presence of twin "B" at the outer gap position x_4 (S3B). Further in the middle of the gap in position x_3 we obsere Debye-Scherrer rings indicating isotropic irregular layer arrangements (S3C). Further towards the inner part of the gap at position x_2 we observe twin coexistence, whereas at the inner position x_1 close to the rotor we observe a dominance of twin "A". This measurement shows that high pre-shearing rates of > 200 s-1 are required to induce macroscopic twin growth. At a shear rate of 100s^{-1} no orientation of the sample is observed.

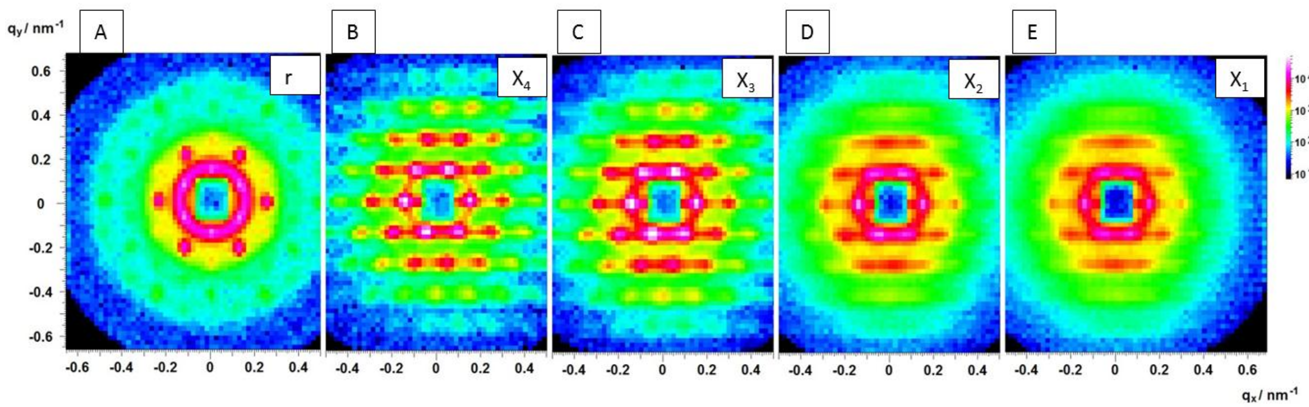

Fig. S4: Rheo-SANS scattering patterns of a shear-oriented FCC twin phase of a 16%wt PI(55)-PEO(170) in D_2O, presheared at 1000 s^{-1} **(A)** Measured scattering pattern in the "radial" beam position probing the [111]-direction. **(B, C, D, E)** Measured scattering patterns in the "tangential" beam position probing the [110]-direction at four different positions $x_1 - x_4$ in the gap. The distorted hexagonal array of the first-order reflections in **(B)** and **(C)** indicate the presence of twin "B" at the outer positions x_3 and x_4 of the gap of the Searle cell. The measurement at x_2 **(D)** was taken in the middle of the gap indicating the position of the twin boundary layer. The measurement at x_1 **(D)** was taken close to the inner rotor in the gap indicating the presence of twin "A".

Fig. S4 displays measured rheo-SANS patterns of a 16% PI(55)-PEO(170) solution after shearing at 1000 s^{-1} and sudden cessation of shear. Fig. S4A shows the measured scattering pattern in the radial sample position, probing the [111]-direction of the lattice. We observe the presence of twin "B" at the outer and central gap positions x_2 - x_4 (S4B,C,D), whereas at the inner position x_1 close to the rotor we observe a regular hexagon with the coexistence of both twins.

Scanning rheo-SAXS

Fig. S5 shows measured rheo-SAXS patterns of a 13% PI(62)-PEO(193) solution in H_2O after shearing at 500 s^{-1} and sudden cessation of shear. Here we show the scattering pattern measured in the radial sample position (Fig. S5A) and all eleven positions (Fig. S2B – K) in the tangential sample position. We observe twin "B" on all positions x_3 – x_{11} in the outer region of the gap. At position x_2 the scattering pattern is symmetric, containing the twin boundary surface, and at location x_1 we observe twin "A".

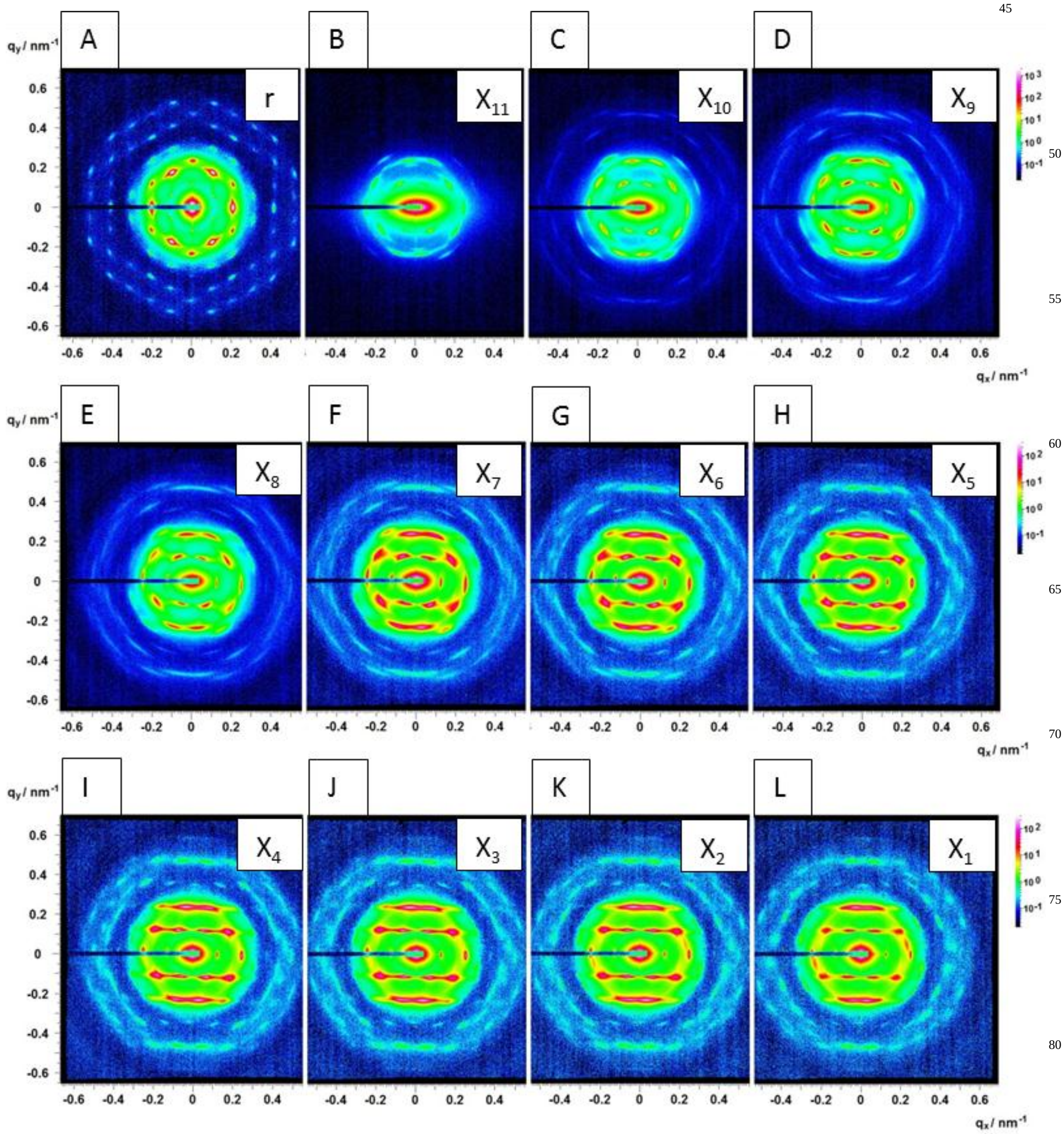

Fig. S5: Synchrotron rheo-SANS scattering patterns of a shear-oriented FCC twin phase of a 13%wt PI(62)-PEO(193) in H_2O, presheared at 1000 s^{-1}. **(A)** Measured scattering pattern in the "radial" beam position probing the [111]-direction. **(B - L)** Measured scattering patterns in the "tangential" beam position probing the [110]-direction with a complete scan over eleven different positions x_{11} – x_1 through the gap of the Searle cell. The distorted hexagonal array of the first-order reflections in **(B-J)** indicates the presence of twin "B" at the outer position x_{11} until position x_3 in the gap of the Searle cell. In the subsequent position x_2 **(K)** both twin are observed in their oriented state. Close to the inner rotor we observe the presence of twin "A".