



	Experiment title: Creep and unjamming of a soft columnar crystal – Time-resolved experiment on a soft glassy material under mechanical stress	Experiment number: SC - 1832
Beamline: ID02	Date of experiment: from: 18-11-05 to: 21-11-05	Date of report: June 06
Shifts: 9	Local contact(s): Dr. Pierre PANINE, Dr. Emanuela DI COLA	<i>Received at ESRF:</i>
Names and affiliations of applicants (* indicates experimentalists): Laurence RAMOS*, Julian OBERDISSE*, Teresa BAUER* Laboratoire des Colloïdes Verres et Nanomatériaux, UMR CNRS-UM2 5587, Montpellier, France		

Report:

We have investigated the coupling between the time evolution of the rheological properties of a soft hexagonal columnar crystal and its structural rearrangement. Upon application of a very small stress to a polycrystalline sample, the measured strain varies as a power law with time (with an exponent smaller than 1): the material deforms plastically. By contrast, under high stress, the system behaves as a fluid and flows. At intermediate stress however, an intriguing regime can be observed when the material evolves suddenly from creep to flow: one observes first a fluidification stage characterized by a continuous decrease of viscosity; this stage is followed by a stationary flow state characterized by a constant and low viscosity. We were essentially interested in this intermediate regime.

We used a stress-controlled rheometer and measured simultaneously the scattered intensity, and the mechanical properties, when a constant stress is applied to the sample. The sample was loaded in a Couette cell, and spectra were recorded in both tangential and radial geometries (incident beam in tangential and radial position with respect to the cell axis). In this report, we only present results obtained in radial geometry as more information is obtained in this case. We typically measure the azimuthal profile of the scattered intensity around the first Bragg peak.

Experimentally, in order to start with a polycrystalline sample with no preferred orientation of the crystallites, we used the following procedure: once loaded in the Couette cell, we cooled the sample. At low temperature, a phase transition from a hexagonal phase to a fluid isotropic phase occurs. By warming up the sample until room temperature, an isotropic polycrystalline sample was recovered. Note that the phase transition temperature varies with the elasticity of the samples. We initially planned to investigate several samples with different elastic moduli (more than one order of magnitude range), but due to the limited

cooling capacities of the set-up (no temperature below 11° C could be reached), we could only study the very soft samples.

A typical set of data is given in Fig. 1. The rheological quantities, the strain γ and the instantaneous viscosity η , defined as the stress over the time-derivative of γ , are shown in a). In b) we show the time evolution of the angular position and intensity of the local maxima of the azimuthal scan. We clearly observe that, as soon as the systems flows, the cylinders are essentially aligned along the flow (maxima around 270 deg). In c) we display the contrast \mathcal{C} , defined as $(I_{\max} - I_{\min})/(I_{\max} + I_{\min})$, where I_{\max} and I_{\min} are respectively the maximal and minimal intensity along the azimuthal scan profile. \mathcal{C} is a useful indicator for the homogeneity of the orientation of the cylinders. We note that at the beginning of the experiment \mathcal{C} is small (~ 0.2). After a slight decrease as the stress is applied (which reflects that one reaches a quite good isotropic orientation), it increases steadily up to a value of the order of 0.9 when the sample flows. The variation of \mathcal{C} directly reflects the scattered intensity at 0 and 180 deg (which corresponds to the scattering for tubes perpendicular to the flow). We indeed show I_{180} , the scattered intensity around 180 deg in b): I_{180} decreases continuously with time and reaches a small and constant value once the sample flows. Note that, in the creep regime, the strain is very low, and essentially one probes the distribution of

orientations of crystallites contained in one given small region of the sample (containing several crystallites), by contrast once the sample flows, different parts of the sample are probed with time. This explains the higher fluctuations of \mathcal{C} and I_{180} in the flow regime than in the creep regime.

By analyzing several sets of data in the same way, we conclude that (i) the evolution from creep to flow regime is due to a collective rearrangement of all crystallites rather than to wall slippage, or shear-banding, (ii) one condition for the sample to flow may be that I_{180} reaches a threshold value; i.e. that the number of crystallites for which the cylinders are perpendicular to the flow (cylinders parallel to both the vorticity direction and the velocity gradient direction contribute to I_{180}) is sufficiently low.

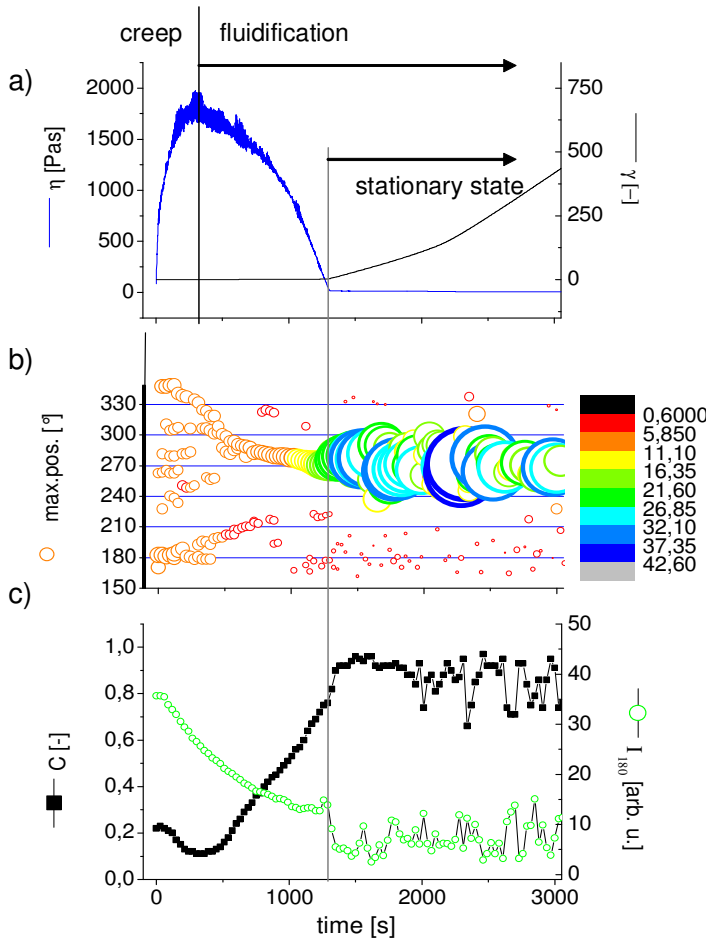


Fig. 1: typical set of data in radial configuration as a function of time. A constant stress of 2Pa is applied from $t=0$. a) rheology data: viscosity (—) and strain (—). b) azimuthal position of the local intensity maxima (○). bubble colour and size scale with the intensity. c) contrast (■) and intensity around 180° (○).