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## PERIODIC STRUCTURES IN DRIVEN COLLOIDAL CRYSTALS

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**Résumé** - La grande dimension de la maille des réseaux de cristaux colloïdaux est la cause de la petitesse de leurs constantes élastiques ( $10^{10}$  fois inférieures à celles des solides usuels). On peut donc aisément étudier des écoulements présentant des rapports contrainte/constante élastique supérieurs à ce que l'on connaissait jusqu'ici. On étudie un écoulement plastique hautement non linéaire en régimes oscillants et stationnaires, ce qui conduit à des structures spatialement périodiques. Ces structures correspondent à des régions alternées de cristallites qui sont l'image par réflexion de leurs voisines. La structure observée en régime oscillant dans un tube sort de l'ordinaire en ce que le cœur est liquide alors que contre les parois la structure est solide (ordonnée). Cette configuration, reconnue comme instable jusqu'ici peut être le résultat d'un comportement inhabituel de la relation déformation-contrainte à la frontière solide-fluide. Des expériences en géométrie de Couette produisent des bandes verticales qui correspondent à un mouvement cohérent de dislocations à la frontière de 2 structures images dans un miroir l'une de l'autre. Ces bandes se déplacent en sens inverse de celui de la rotation du cylindre intérieur et à vitesse proche de celle de la propagation des ondes élastiques transverses.

**Abstract** - The large lattice spacings in colloidal crystals produce elastic constants  $\sim 10^{10}$  less than conventional solids. It is therefore easy to study flow properties at stress/elastic constant ratios higher than previously available. The highly nonlinear plastic flow regime studied in oscillating and steady state flow yields periodic patterns. These patterns correspond to alternating regions of ordered crystallites which are mirror image structures. The pattern observed in oscillatory flow in a tube is also unusual in that the core is liquid while at larger radius one finds a solid on the tube wall. This traditionally unstable configuration may be the result of an anomalous stress-rate relation at the fluid-solid boundary. Experiments in couette geometry produce vertical stripes which correspond to coherent motion of dislocations at the boundaries separating two mirror image structures. The stripes move in the direction opposite from the rotation of the inner cylinder, at velocities close to the transverse sound velocity.

### I. Introduction

Colloidal crystals are characterized by lattice spacings which are

thousands of angstroms. The elastic constant, which roughly measures the energy density are therefore smaller than found in conventional solids by a factor of order  $10^{10} \text{ l}^{-4}$ . The yield stress and region of plastic flow are correspondingly small and one can easily attain stress/moduli ratio's which are not accessible in other systems.

In experiments which were originally aimed at measuring the shear stress-shear rate relationship we have found a series of instabilities which produce periodic ordered structures in the sheared colloidal crystal. These structures occur in the plastic flow regime somewhat below the shear melting transitions<sup>5</sup> and appear to involve the alignment of dislocations into grain or twin boundaries.

When the colloidal crystal is placed in a couette apparatus with the inner cylinder rotating, we can also observe the formation of Taylor vortices and the transition to the wavy Taylor states. However, although the colloidal crystal can flow with a relatively low viscosity we never observe the Taylor instability with the colloid in the crystalline state. For "strong" crystals we observe a suppression of the Taylor instability. With increasing shear, the crystalline phase shear melts with a spontaneous transition to Taylor or wavy Taylor flow.

As originally pointed out by Crandall and Williams, the characteristic elastic moduli of colloidal crystals can be understood using the approximate relationship

$$G \sim Vc \quad (1)$$

where  $V$  is an interparticle potential energy and  $c$  is the particle concentration. In Table I we note the characteristic interparticle potentials, densities and elastic constants of conventional materials and colloidal crystals. The potential energies are the same order of magnitude. Since the potential energy differences between liquid and solid state are what determine the melting temperatures, it is not surprising that the melting temperature of colloidal crystals and conventional solids are comparable and equal to several times room temperature. The elastic constants however differ by the factor of the particle concentration of  $\sim 10^{10}$ . This explains the extreme fragility of the colloidal crystals.

With stresses that are easily attainable in the laboratory, it is possible to have ratios of shear stress to elastic constant which are extraordinary compared to conventional materials. A typical, laboratory experiment involves stresses of the order of  $\sigma \sim 10^3$  dynes/cm<sup>2</sup>, giving  $\sigma/G \sim 10^2$  for colloidal crystals. Noting that  $G_{\text{rock}}/G_{\text{cc}} \sim 10^{12}$  a similar  $\sigma/G$  for rock would involve stresses of  $\sim 10^{14}$  dynes/cm<sup>2</sup>. This would be the stress under a mountain  $\sim 10^6$  Km high or  $10^5$  x the height of Mt. Everest. Thus we are investigating matter in an entirely new shear regime.

The studies which come closest to these enormous stress/ modulus ratios involve geological flows, and it is interesting to examine those results to see what should be expected in the low shear-stress regime for our crystals. Figure 2 of Ref. 6 shows a typical deformation map (temperature and stress diagram appropriate for different flow mechanisms) for Olivine, a component of the earth's mantle. At low stress the solid is essentially elastic with the only flow coming from the creep of point defects. The transition to

Table I

	Metals, Rocks	Colloidal Crystals
Interparticle Distance	$\sim A^0$	$\sim \mu$
Interparticle Interaction	Coulomb $\frac{e^2}{r}$	Screened Coulomb $\frac{Z^2 e^2 e^{-Kr}}{\epsilon r}$ $\epsilon \sim 100$ $Kr \sim 1$
Charge	$e$	$Ze$ $Z \sim 10^3$
Interparticle Potential	$\sim 10^{-11}$ erg $\sim 10^5 K$	$\sim 10^{-11}$ erg $\sim 10^5 K$
Melting Temperature	$10^2 - 10^3 K$	$10^2 - 10^3 K$
Concentration	$\sim 10^{24}/(CM)^3$	$\sim 10^{12}/(cm)^3$
Elastic Constants	$\sim 10^{13}$ dynes/cm <sup>2</sup>	$\sim 10$ dyne/cm <sup>2</sup>

plastic flow, coming from dislocation creep, is characterized by a yield stress which varies from  $10^{-5}$  of  $G$  at melting to  $10^{-2}$  of  $G$  at low temperature. At temperatures above  $\sim .5$  times the melting temperature there is a region where the strain rate or shear rate  $\dot{\gamma}$  varies as a power law with applied stress  $\sigma$

$$\dot{\gamma} \propto \sigma^\alpha \quad \alpha = 3-7 \quad (2)$$

At higher shear stress there is an exponential variation known as the Dorn law<sup>6</sup>:

$$\dot{\gamma} \propto \exp(\sigma^2/T) \quad (3)$$

The general description (in scalar form) of the strain rate by motion of dislocations is (for edge dislocations):

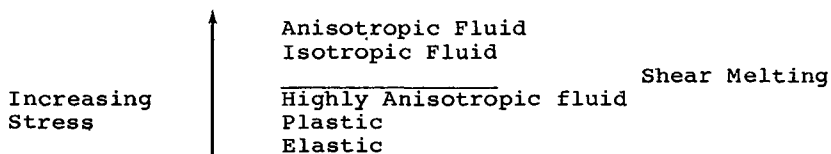
$$\dot{\gamma} = \rho b v \quad (4)$$

where  $\rho$  is the dislocation density,  $b$  is the Burgers vector and  $v$  is the dislocation velocity. Since both the velocity and density of dislocations can change with the stress, models are needed to explain the properties of different systems and these models can

produce the different power laws and exponential dependences<sup>7</sup>. The most "natural" relationship seems to take the power law with  $\alpha = 3$ .

It is worth noting that the yield stress of most single crystal materials is  $\sim 10^{-5} - 10^{-4}$  of  $G$ <sup>7</sup>. Olivine has a particularly high yield stress of  $\sim 10^{-2}$  at low temperatures due both to its small particle size and the fact that it is a mixture of many oxides, leading to a substantial increase the defect pinning. We should expect yield stresses of order  $10^{-4} - 10^{-5}$  for the colloidal crystals.

From the fact that we can obtain such high  $\sigma/G$  ratios for colloidal crystals, it should be expected that highly nonlinear flow is observable. In our studies of colloidal crystals we have indeed seen highly nonlinear behavior, which can roughly be grouped into the following regimes.



At very low shear (and finite frequency of  $\sim 10^{-2}$  Hz) the colloidal crystal responds elastically. Increasing the shear stress we find a plastic flow regime where the shear stress/shear rate relationship is described by a power law indicating dislocation creep. The particular exponent observed depends upon the crystal studied (density, charge, ionic strength) and ranges from 3 to 7 (as in rocks)<sup>8</sup>. For higher shear stress the dislocations are so dense and moving so rapidly that the samples behave as a highly anisotropic fluid with rigid planes gliding over one another. Just above this region the sample "shear melts" into an approximately isotropic fluid<sup>5</sup>, but at higher shear the colors observed in different shear directions indicate a small anisotropy in the liquid structure factor induced by the shear<sup>9</sup>.

As a consequence of this highly nonlinear behavior and also because of a transition between different regimes, we have often observed spatially periodic patterns, either stationary or moving, and in a variety of geometries.

The first instability we observed was for oscillatory flow in a tube<sup>10</sup>. A capillary is filled with the colloidal crystal and then an oscillating pressure gradient is applied. At first the crystal slips at the tube radius and we have plug flow. Then dark circles (perpendicular to the axis) are observed to move from the top to the bottom of the tube, in phase with the meniscus but with much greater velocity and excursion. These are dislocation loops, which by their motion tend to align the crystallites. We then observe a pattern forming on the inner glass wall of the tube. This pattern corresponds to alternating mirror-image regions of fcc crystallites with an orientation which best resists the shear<sup>10</sup>.

In trying to understand this pattern we run into the problem that pressure applied to a solid in a tube should cause yield of the solid at the largest radius first, and then plug flow (like toothpaste squeezed from a tube). Elementary arguments show that the pressure on a solid crystal of radius  $r$  gives a force  $\pi r^2 P$  while the restoring force from the boundary is  $\pi \sigma r l$  (where  $\sigma$  is the shear stress). Thus for larger radius cylinders the ratio increases and a solid will yield first at its outer boundary.

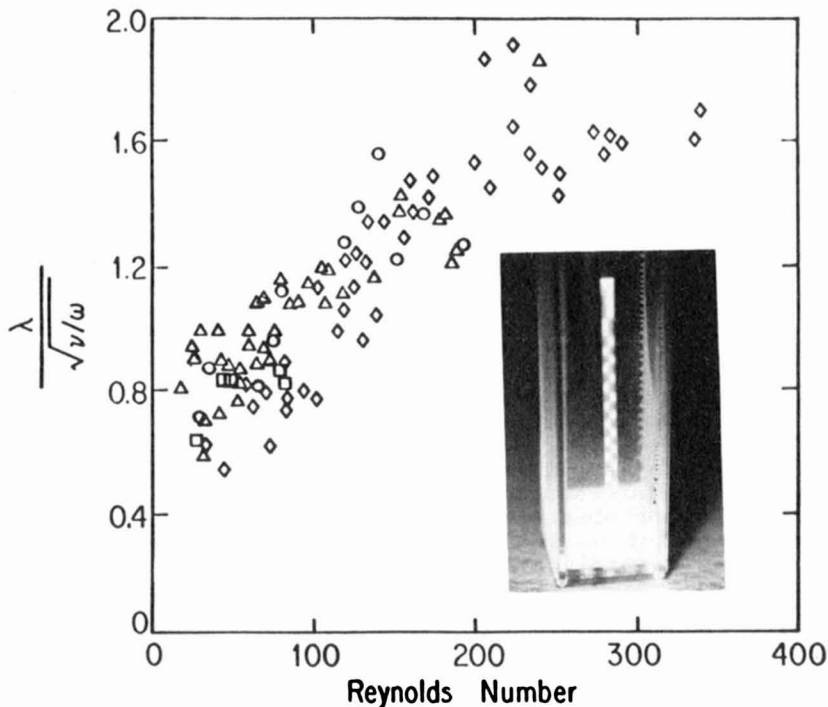


Fig. 1 - Wavelength of periodic pattern, normalized by the viscous penetration depth vs. Reynolds number for a series of colloidal crystals of varying elastic constant ( $10-60$  dynes/cm<sup>2</sup>), tube diameter (1-4 mm), drive frequency (2-30 Hz) and drive amplitude. Inset is a picture of the pattern formed by oscillatory flow in a capillary.

However if we consider a fluid flowing in the tube and then ask where the crystal phase can nucleate we find first that the mass flow must be conserved. If we crystallize a region into the liquid from the glass walls we find that the velocity and shear force decrease with the radius of the hole which is left. Thus the crystalline region can grow in from the glass wall until the stress is the yield stress. This configuration, solid outside and liquid inside, is then stable as far as radial displacements are concerned. However further analysis shows that in this configuration an increase in flow decreases the pressure - an unstable situation<sup>10</sup>. Thus the system is unstable against modulations of the radius of the liquid hole along the axis of the tube. We expect that this is what causes the periodic structure. But the complication of periodic motion of the fluid with the Poiseuille geometry makes further mathematical investigation difficult.

The solution to the periodic Poiseuille flow problem has not been accomplished even for Newtonian fluids. Some indication that we may be seeing purely hydrodynamic effects comes from the fact that all of our data for different size tubes, and crystalline elastic constants collapses to the same curve (Figure 1) when the periodicity is normalized by the viscous penetration depth ( $\sqrt{\nu/\omega}$ , with  $\omega$  the drive frequencies and  $\nu$  the dynamic viscosity) and plotted vs. the Reynold's number. Together with Dr. G Maret (Grenoble) we there-

fore studied oscillatory motion of a pure liquid using shear birefringence. No stationary, spatially periodic modes were seen. Thus the periodicity in the Poiseuille geometry remains an unresolved question.

We now turn our attention to the couette geometry where the flow field and shear rate is more uniform than in Poiseuille flow. A picture of the apparatus is shown in Figure 2. The outer wall is fixed, the inner cylinder of radius 4.52 cm can be rotated and the gap is 3.175 mm ( $\emptyset$ .125 inch). At low shear rates the colloidal crystal samples (typically  $.091\mu$  diameter spheres with 1-3% volume fraction) flow and shear align producing brilliant Bragg scattering. At higher shear rates new and rather unusual long range patterns appear in the flow. They consist of periodic vertical stripes of alternating green and red color, with a wavelength on the order of one cm. These stripes circulate around the tube in a sense contrary to the velocity field. The formation of this pattern and its properties will be elaborated shortly.

As the shear rate is further increased the observations depend on the strength of interaction of the particles in the colloid. For "soft" crystals one observes shear melting into a liquid state with consequent loss of opalescence. At higher shear Taylor rolls<sup>11</sup> appear at approximately the same Reynolds number as for water in this geometry. The Taylor rolls are easily observed with the sheared colloid as bright, narrow opalescent bands which appear horizontally around the cylinder wall. The opalescence results from the recrystallization of the colloid in the nodes of the Taylor rolls where the total shear is decreased (Figure 2). When shear is increased further we see the onset of wavy Taylor rolls. These are characterized by the disappearance of the sharp opalescent lines and the appearance of a colored dynamic shear pattern. This results from the shear distortion of the structure factor of the colloid liquid and the alignment of these distortions in the shear flow.

For stronger crystals the Taylor instability is suppressed and does not occur until the sample shear melts - often at Reynolds numbers, calculated using the viscosity of the sheared colloidal crystal, which are several times the critical Reynolds number for an isotropic liquid. In these samples the shear melting and the transition to the Taylor instability happens simultaneously. If the crystal is sufficiently strong the shear melting occurs directly into the wavy Taylor state. We have never observed the Taylor instability before the colloidal crystal is shear melted. We suspect that this is the result of the extreme anisotropy of the viscosity in the sheared colloidal crystal. It has a low viscosity along the direction of flow but is essentially a rigid solid in the direction along the shear gradient. Since the Taylor instability involves flow both radially and tangentially it is suppressed by this anisotropy. We would expect similar effects with Smectic Liquid Crystals.

We now return to the vertical stripes which are observed in the plastic flow regime. This pattern of flow has, to our knowledge, never been seen before in the couette geometry, and, indeed, has totally different symmetry from the instabilities normally seen. From the Bragg scattering properties of the crystals, we learn that the stripes represent alternating regions of shear aligned fcc crystals with mirror image symmetry, as was observed for the Poiseuille flow. The pattern is somewhat unstable - it develops but does not always remain indefinitely. Instead it will appear, disappear, then reappear. The frequency and persistence of this

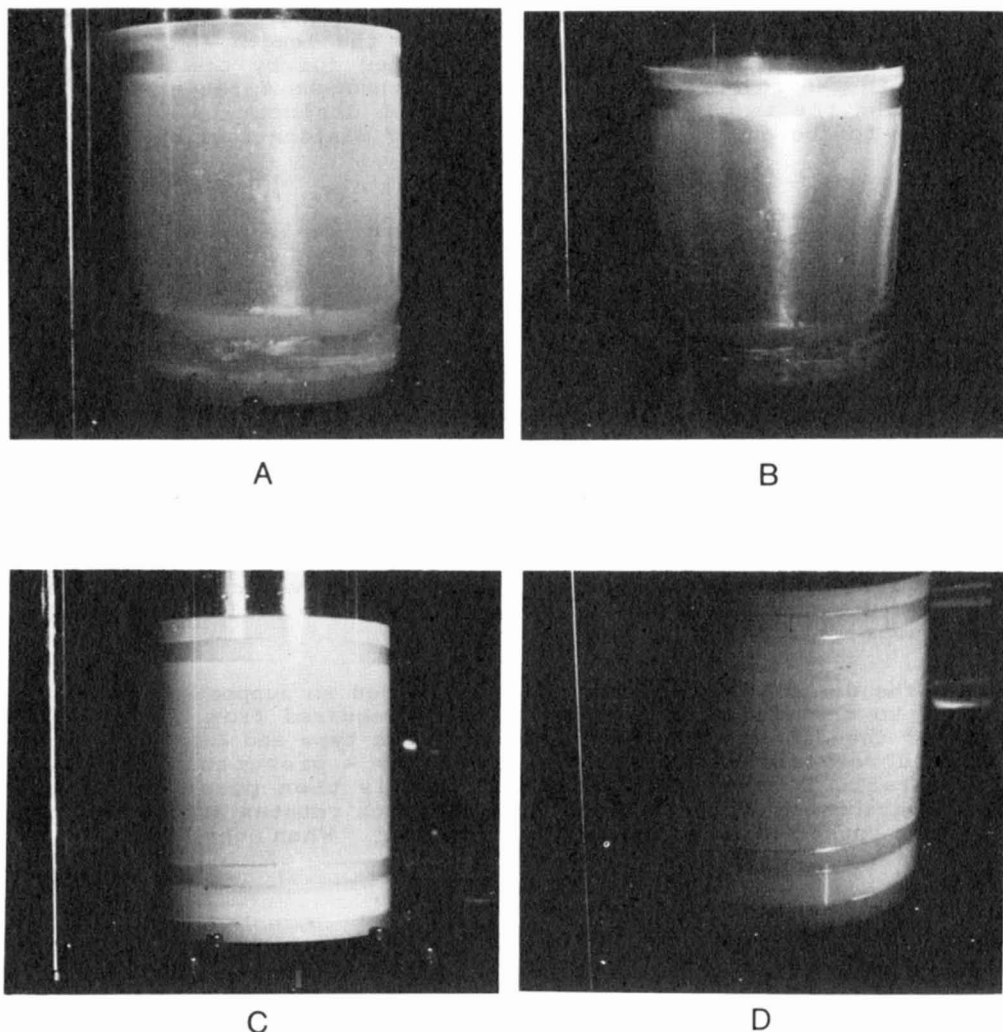


Figure 2 -- a) Couette system at rest, with deionized polystyrene latex  $d$ ,  $0.091 \mu\text{m}$ ,  $\phi = 3\%$ ; b) Periodic stripes at a shear rate of  $\sim 10 \text{ Hz}$ ; c) Taylor rolls at a shear rate of  $\sim 130 \text{ Hz}$ ; d) Wavy Taylor rolls at a shear rate of  $\sim 150 \text{ Hz}$ .

seems to be a complicated function of the crystal strength and inner rotor speed. However, when the pattern is fully developed, the inner rotor can be stopped suddenly and the pattern will be "frozen in". We believe that the occurrence of the pattern may be understood in terms of the static and dynamic dislocation density and motion. For simplicity we will discuss the phenomena as if the structure were simple cubic rather than the much more complicated fcc or bcc structure actually formed. Due to the confinement of the colloidal crystal in a cylindrical geometry there is a minimum



density of dislocations that must be present even statically. At the inner cylinder the path length corresponding to an angle  $\Delta\theta$  is  $R_1 \Delta\theta$  (see Figure 3); at the outer cylinder the length is  $R_2 \Delta\theta$ . The difference in length  $(R_2 - R_1)\Delta\theta$  is accounted for by the addition of planes in the form of edge dislocations throughout the area defined by  $\Delta\theta$  and the two cylinders. Since each dislocation adds a distance  $b$  to the outer length, the number of dislocations needed is

$$n = \frac{\Delta\theta (R_2 - R_1)}{b} \quad (5)$$

The area is  $\sim (R_1 - R_2)R_1 \Delta\theta$  and the minimum dislocation density is then

$$\rho_s = \frac{n}{R\Delta\theta(R_2 - R_1)} = \frac{1}{bR} \quad (6)$$

where  $R = (R_1 + R_2)/2$ . On the other hand, independent of geometry, there is a dynamic dislocation density needed to maintain the shear rate and given by equation 4 or

$$\rho = \frac{\dot{\gamma}}{vb} \quad (7)$$

As  $\dot{\gamma}$  increases from zero the dislocation density increases (in plane Couette flow) as does the dislocation velocity. This dislocation increase coupled with the velocity increase gives rise to the highly nonlinear behavior of sheared solids. Usually the dislocation velocity can not be higher than the sound velocity<sup>12</sup>  $v_s$ . Thus at some point the velocity is fixed and only the dislocation density increases.

When the dislocation density which is needed to support the shear is equal to the static dislocation density required from our geometry all of the dislocations can be of the same type and can move in the same direction and at the same velocity - presumably something comparable to the speed of sound. It is then possible for the dislocations to form a spatial pattern which rotates around the tube but is otherwise independent of time. When the dislocation

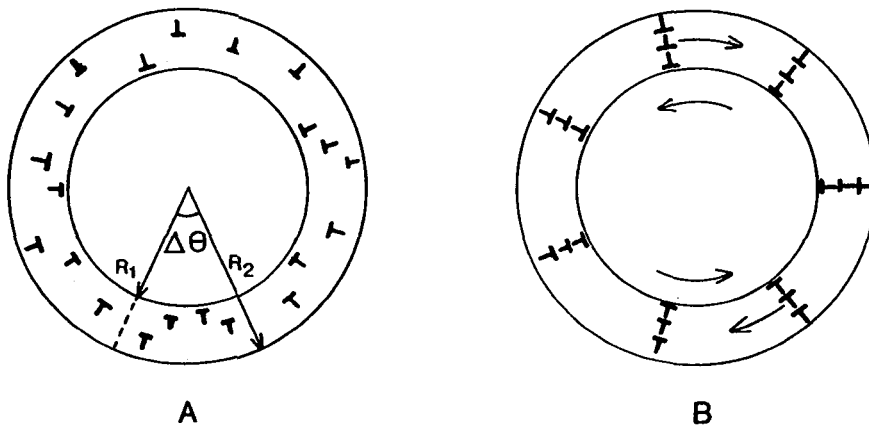


Figure 3 -- a) Random dislocations in the Couette cell showing minimum dislocation density. b) Model for periodic stripes with coherent dislocation motion.

densities are equal the shear rate is

$$\dot{\gamma} = \frac{v}{R} \sim \frac{v_s}{R} \quad (8)$$

If the dislocations align into grain boundaries as shown in Figure 3 then we can calculate the spatial period which would be observed. The alignment of the dislocations from energetic considerations is favorable, but is highly unlikely due to the small forces involved as compared to the large stresses involved in the shear. However, F. Rothen at this conference has suggested that the dissipative shear forces themselves favor alignment for rapidly moving edge dislocations.

From Figure 3 we see that the spacing between boundaries depends on the spacing between dislocations in the boundaries which we have called mb. The fixed dislocation density gives us the relation

$$\begin{aligned} \rho &= \frac{R_2 - R_1}{mb} / \lambda (R_2 - R_1) \\ &= \frac{1}{bR} \\ \Rightarrow \lambda &= R/m \end{aligned} \quad (9)$$

One final question relating to the experiment is the direction in which the dislocation moves relative to the velocity field. In Figure 4 we illustrate the movement of edge dislocations in shear flow for "positive" and "negative" dislocation. In the figure the upper surface corresponds to the inner rotating cylinder while the outer stationary cylinder is the lower surface. Because of the curvature of the cylinder (see Figure 3) we have only negative dislocations (when the static and dynamic dislocation densities are equal) and therefore the dislocations and the domain wall they form will move in a direction opposite to the velocity field.

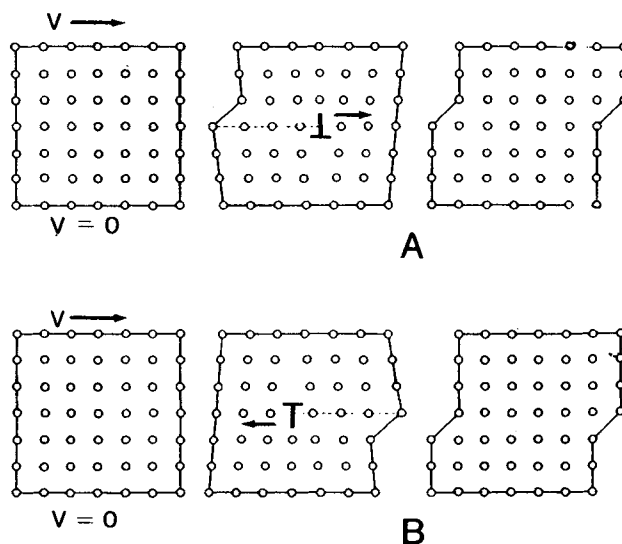


Figure 4 -- a) Direction of dislocation motion for a "positive" dislocation. b) Direction of dislocation motion for a negative dislocation, opposite to velocity of flow. This corresponds to our geometry.

Thus the model which we propose suggests

- pattern forms when  $\dot{\gamma} \sim V/R \sim V_s/R$
- pattern moves with  $v \sim v_s$
- $\lambda = R/m$
- pattern moves opposite to rotation of inner cylinder.

Experimentally the pattern does indeed move opposite to the direction of rotation. The spacing between the "stripes" is measured to be  $1 \text{ cm} \pm 0.05$  and appears independent of the shear modulus of the colloidal crystal. This would imply that the aligned dislocations in a wall are  $\sim 10$  lattice constants apart. To test the other predictions we have measured the velocity of the pattern as a function of shear rate for several samples with different elastic constants. The results are shown in Figure 5. The measurements were performed by scattering a monochromatic laser beam from the sheared sample and placing a photodiode at one of the Bragg scattered spots. As the pattern rotates the Bragg spots oscillate, reappearing at the photodiode every other "stripe". The spectrum is then Fourier analyzed to obtain the frequency  $\omega$  and the velocity calculated from  $\lambda\omega$ .

As we can see from Figure 5 the velocity of the pattern is approximately the velocity of sound, although there appears to be an entire range of values for which the pattern is stable. As the sound velocity of the sample is increased the pattern velocity increases. The shear rate is about a factor of four larger than the predicted value ( $R \sim 5 \text{ cm}$  and we expected  $\dot{\gamma} = v/R$ ) and the relationship between  $v$  and  $\dot{\gamma}$  is not linear with zero intercept. This is the biggest discrepancy with our model but may be due to the increase of  $\rho$  throughout the region where we observe the pattern, rather than the model of a single  $\dot{\gamma}$  for its formation exactly where  $\rho_s = \rho_d$ . There may also be some geometrical factors from the different dislocation structure in the fcc structure.

We should also mention that the two cases described above are only a few of many pattern formations we have observed in different geometries and flow conditions. In steady flow in a capillary tube, we often observe a series of colored rings which move at velocities near the sound velocity (and much greater than the flow velocity) down the tube in the same direction as the flow. In the couette geometry for "weak" crystals we also observe a metastable pattern of "stripes" with spacing  $\sim 3 \text{ mm}$  (approximately equal to the gap size) which rotates considerably slower than the sound velocity and in the direction of the flow. This pattern is seen when the sample is quickly slowed down from the shear melted state to the same  $\dot{\gamma}$  as would produce the oppositely rotating pattern described at length above. This short stripe pattern decays in  $\sim$  one minute to the more stable contrary rotating pattern.

In conclusion the high shear stress/shear modulus ratios easily obtained in colloidal crystals lead to highly nonlinear flow properties similar to those seen in geological flows and to entirely new regimes far beyond those available previously for laboratory experiments. As a consequence of these large nonlinearities there are a variety of instabilities which lead to the formation of periodic spatial structures under shear. One particular pattern can be associated with the shear alignment of edge dislocations into a boundary which moves at the transverse sound velocity in the crystal. The appearance of this pattern illustrates the role of dislocations in the flow of the colloidal crystals at shear rates approaching the shear melting transition.

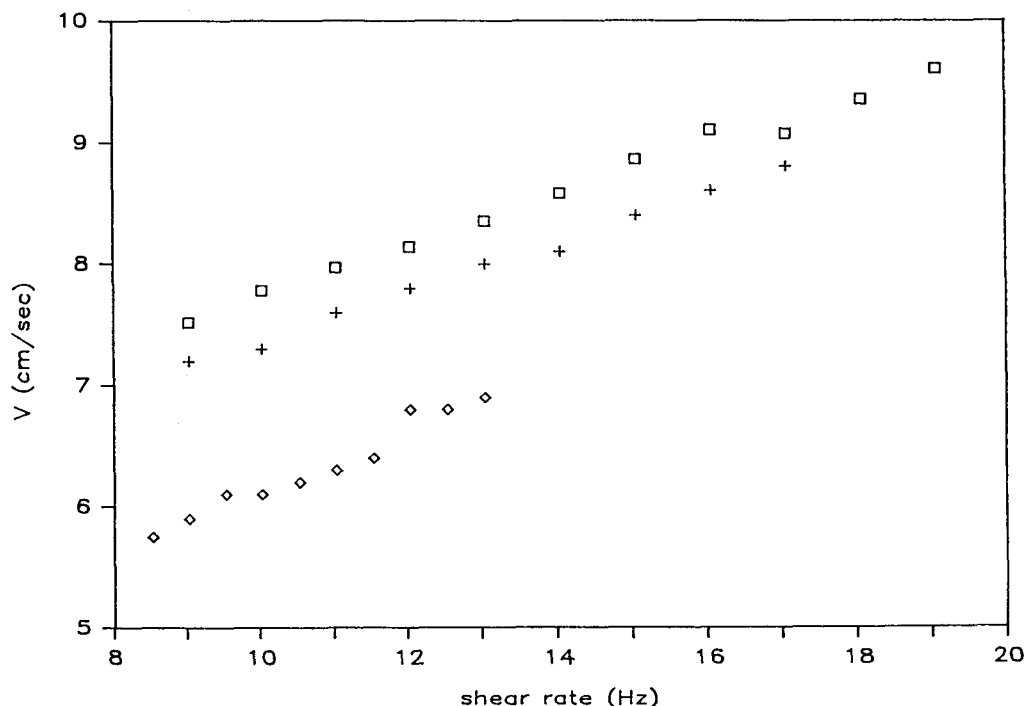


Figure 5 -- Velocity of observed pattern as a function of shear rate for colloidal crystals with different sound velocities. In the graph, the squares are  $v_s = 7$  cm/sec, the pluses are  $v_s = 6.1$  cm/sec and the diamonds are  $v_s = 3.9$  cm/sec.

Experiments on Taylor instabilities indicate that Taylor rolls are never formed before the crystals shear melt. This results from the anisotropy of the viscosity of the sheared colloid. With sufficiently strong colloidal crystals the inner crystal can be rotated to produce a Reynolds number (including the increased viscosity of the polyballs) several times that which would be necessary to produce Taylor rolls. In this case the shear melting leads immediately to the formation of wavy Taylor rolls. We expect that other anisotropic fluids such as liquid crystals may show similar effects.

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