

Thixotropic Behaviour of Petroleum Jelly

If the term 'thixotropy' be restricted to Freundlich's original definition of reversible sol-gel transformation, a peculiar manifestation of this property is strikingly shown by petroleum jelly at varying rates of shear. The phenomenon can be demonstrated with no more elaborate apparatus than a 100-c.c. glass cylinder and a perforated disk as piston, mounted centrally on a metal rod terminating in a ring, a similar arrangement to the familiar 'malted milk mixer'. The plunger not only serves to shear the contents of the cylinder, but also as a rough consistometer, the time of travel of the disk between any two graduations on the freely suspended cylinder serving as a measure, provided the rigidity component of consistency of the material is less than the hydrostatic load of the suspended cylinder.

A petroleum jelly of medium consistency serves for the observation. This should be melted, poured into the cylinder and allowed to set for 24 hours, when the consistency will be generally such that no movement of the disk relative to the jelly takes place when the apparatus is suspended by the ring.

If now the plunger be worked up and down very slowly between its lowest position and, say, the 25 c.c. mark, the shearing resistance will be felt to diminish until suspension from the ring will result in a steady upward travel of the disk through the jelly in the sheared zone. Timing is recorded between any two graduated points within the zone of shear. Measurement is repeated by *slowly* returning the disk to its lowest position once more and again noting the time of travel. Repeated measurements lead eventually to a low constant value of consistency, increasing very slowly indeed on resting the material.

When this sheared jelly is now re-sheared by pushing the plunger up and down as quickly as possible, a rapid and progressive growth of consistency is observed, a few churnings resulting in such an increase of rigidity that the disk remains fixed in position as it did in the original un-sheared jelly. The transformation from high to low consistency may be repeated as often as desired.

The optical picture is not very clear. The un-sheared material shows thin needle-shaped crystals under polarized light, but rather less in amount than the known heterogeneity would lead one to expect. This may, of course, be due to the bulk of the crystals being of a dimension below optical resolution. The field under paraboloid condenser illumination is quite void, indicating similarity of refractive indices of the component phases. The slowly sheared jelly under polarized light shows a great increase of crystallinity, the crystals, however, apparently having the form of shorter, thicker double cones on a common base. The quickly sheared material again shows needles, but of a shorter length, and, while the field of the original jelly shows some incomplete rosettes, these are here absent.

These experiments followed on qualitative observations on a sample of petroleum jelly which had been variously treated by the alternative methods of melting and quickly chilling to a low and a normal temperature respectively, passing through a three-roller mill, a cone mill, a milk homogenizer and a Werner-Pfleider mixer, the subjective consistencies being in the descending order named.

Precision measurements of mobilities by a capillary plastometer and of rigidities by a parallel plate plastometer are in progress of determination.

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Recrystallization of Metals under Stress

IN *Nature* of September 11, 1948, Prof. E. N. da C. Andrade and also Messrs. Sully, Cale and Willoughby discuss the recrystallization of metals under stress. This phenomenon has been under observation¹⁻³ in the Metallurgical Laboratories of the University of Melbourne for some fourteen years; and we have reached the following conclusions relating to lead and its dilute alloys.

(1) Industrial virgin lead, so long as it contains less than 0.001 per cent silver, will, in the annealed state, recrystallize under stress when it has reached 4-5 per cent elongation at strain-rates of the order of 10^{-4} - 10^{-5} per day. (2) In the 'as rolled' condition, the recrystallization frequently occurs with less than this amount of deformation. (3) The recrystallization is always accompanied by an increase in grain size, the linear dimensions of the grains increasing by a factor of ten. (4) There is a marked increase in creep-rate during recrystallization. (5) The recrystallization starts at one or more centres and spreads throughout the mass. It can be observed at different stages by intercepting the test and re-etching. (6) The progress of recrystallization can be followed without interrupting the test by observing the slip-line system on the test piece. At first, slip lines change direction at the boundaries of the original small crystals; after recrystallization in any area the slip lines are seen to continue across many of the areas defined as crystals by the original etch. (7) Annealing lead at 120° C. for 24 hours removes the facility for recrystallization at creep-rates of the above order. (8) The phenomenon is noted in alloys of lead with less than 0.05 per cent copper, zinc, bismuth, silver, antimony, respectively. In general, the strain needed is higher: an alloy containing 0.001 per cent silver recrystallized after 10 per cent extension in 300 days caused by a load corresponding to an initial stress of 500 lb./sq. in. (9) Additions of less than 0.05 per cent of calcium, magnesium or tellurium respectively appear to eliminate the recrystallization under the conditions used. (10) In pure lead, vibration facilitates recrystallization. With a creep-rate of 10^{-4} - 10^{-5} a day, the phenomenon occurs with about 2.5 per cent elongation when a 50-cycle vibration is superposed on a steady stress.

The creep-rates employed by us have always been much lower than those reported by Prof. Andrade. In our opinion the rate of strain has a fundamental effect on the mechanism of creep.

I do not agree that the transition from constant creep-rate to accelerating creep-rate in uniform load tests is necessarily due either to the increased stress or to recrystallization at this stage, except in the case of single crystals. Hirst⁴ has shown that single crystals of lead do fail in this manner.

It is interesting to note from the work in the Fulmer Research Institute that the compression -