

Über fließende Krystalle.

Von

O. Lehmann.

(Mit Tafel III und 3 Holzschnitten.)

Fließende Krystalle! Ist dies nicht ein Widerspruch in sich selbst — wird der Leser der Überschrift fragen —, wie könnte denn ein starres, wohlgeordnetes System von Molekülen, als welches wir uns einen Krystall vorstellen, in ähnliche äussere und innere Bewegungszustände geraten, wie wir sie bei Flüssigkeiten als „Fließen“ bezeichnen und durch mannigfache Verschiebungen und Drehungen der ohnehin schon des Wärmezustandes halber äusserst lebhaft durcheinander wimmelnden Moleküle zu erklären pflegen?

Wäre ein Krystall wirklich ein starres Molekularaggregat, dann könnte von einem Fließen desselben in der That ebensowenig die Rede sein als beispielsweise vom Fließen eines Mauerwerks, das allerdings bei Einwirkung starker Kräfte in rutschende Bewegung geraten kann, welche Bewegung aber nur dann einigermaßen dem Strömen einer flüssigen Masse entspricht, wenn die Fugen sich öffnen und einzelne Bausteine ausser Zusammenhang geraten und sich übereinanderschieben und durcheinanderrollen, ähnlich wie die einzelnen Körnchen einer bewegten Sandmasse.

Dass es übrigens feste, wenn auch nicht krystallisierte Körper giebt, welche ganz wie Flüssigkeiten, wenn auch unvergleichlich viel schwieriger fließen können, ist jedem bekannt, der einmal die langsamen Veränderungen einer hohl liegenden Siegellackstange oder einer grösseren freistehenden Pechmasse beobachtet hat. Alle schmelzbaren amorphen Körper gehen kontinuierlich aus dem flüssigen in den festen Zustand über und der Punkt, bei welchem der Aggregatzustand wirklich fest wird, d. h. wo sich die ersten Anzeichen beginnender Verschiebungselastizität einstellen, ist so wenig erkennbar, dass wir häufig einen solchen Körper gerade der Fähigkeit des Fließens halber noch flüssig nennen, wo er streng genommen bereits als fest bezeichnet werden müsste.

Da in diesen Fällen schon eine sehr geringe Kraft — das eigene

ON FLOWING CRYSTALS

by
O. Lehmann

(With Plate III and 3 wood-engravings)

Flowing crystals! Is that not a contradiction in terms? Our image of a crystal is of a rigid well-ordered system of molecules. The reader of the title of this article might well pose the following question: 'How does such a system reach a state of motion, which, were it in a fluid, we would recognise as flow?' For flow involves external and internal states of motion, and indeed the very explanation of flow is usually in terms of repeated translations and rotations of swarms of molecules which are both thermally disordered and in rapid motion.

If a crystal really were a rigid molecular aggregate, a flowing crystal would indeed be as unlikely as flowing brickwork. However, if subject to sufficiently strong forces, even brickwork can be set into sliding motion. In a certain sense, the resulting motion corresponds to a stream of fluid mass in which the joints between the individual bricks open. The bricks then run out of control, moving over and rolling around each other in a disorderly manner, rather like single granules in a turbulent mass of sand.

As a matter of fact, there are solid – but nevertheless non-crystalline – bodies which are able to flow like liquids, although with much greater difficulty. This fact is evident to anyone who has ever observed the slow change of an unsupported stick of sealing wax or a larger free-standing mass of pitch. All fusible amorphous bodies transform from the liquid into the solid state continuously. The point at which the state of aggregation really becomes solid (i.e. where the first hints of the onset of displacement elasticity occur) is extremely difficult to recognise. Indeed, because such a material is still able to flow, we would often still regard it as fluid, even though, strictly speaking, it should already be described as solid.

As in the cases mentioned above, a very small force – the body's own weight – is easily enough to produce the phenomenon under discussion. Sometimes one might be tempted to extend the definition of the liquid state, so as to include all those substances which can be made to flow by their own weight alone. However, such a definition would be completely unjustifiable. As an example, consider foundations

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which seem to be completely rigid. But if there is a strong enough loading pressure from the brickwork above them, even they give way and begin to flow.

One must therefore admit that amorphous solid bodies indeed are able to flow. In the final analysis this causes few problems. From a theoretical point of view the disorder which is already present within the molecules of such bodies cannot be enhanced, even by the most vigorous flowing motion. However, there are extremely serious problems associated with the assumption of flow in crystalline bodies and posed by the explanation of such a possibility.

The individual elements of such an array are precisely organised with respect to each other, according to the mathematical rules of crystallography. They maintain any arrangement which they have previously adopted. They do this in the absence of adhesive or connecting joints, notwithstanding vigorous thermal vibrations. Consequently it seems completely impossible for fluid motion to disturb the whole arrangement significantly.

Thus, it used to be generally thought self-evident that a permanent deformation of crystalline bodies, e.g. bending, pressing or kneading, must necessarily be accompanied by extensive repeated destruction of their structural integrity. This would cause a large number of extremely thin cracks, splits and fissures to occur. Their small size would render them invisible or nearly so. They are not sufficiently extensive to destroy the whole structure, nor do they run in such a way to make it possible to pull apart the separated pieces. If one were to cut a glass pane in a zigzag fashion from *a* to *b* (cf. Fig. 1), it would not be possible to separate the two parts, although they are no longer connected to each other. Because of the spring tension of the single fragments, if many such fissures could be generated, the plate would have such a high degree of mobility that it could be easily deformed in different ways either temporarily or permanently.

Examples of deformed crystals are known for all forgeable metals, e.g. lead, tin, zinc, silver, gold, platinum, iron, copper, etc. It is known that all these metals become increasingly fragile on continued forging. This is explicable if forging increases of the number of fissures. In the same way, the perturbation caused by the cleavage process may result in an initial increase in the hardness; a starting fissure will not be as easily transmitted in the aggregate as would be the case in

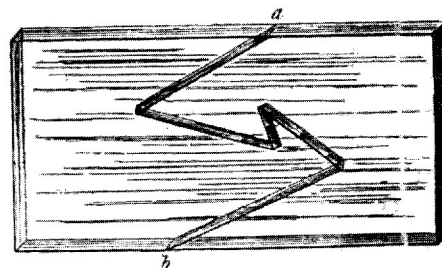


Fig. 1

the homogeneous crystal. For the same reason cane-work and sponge-like cellulose withstand the penetration of projectiles much more than the same quantity of material in a compact form, and a wire rope breaks less easily than a massive iron rod of the same weight and length.

Metals recover their initial toughness on annealing. This may be explained by the fact that at high temperatures they are changed into another enantiotropic modification. On cooling the initial state is then re-established. The fissures are closed in the course of volume changes in larger-sized unbroken crystals.

To a large extent metallic substances are deformable. Following his most interesting experiments with embossing, Tresca talks specifically about the flow of solid metals. By contrast, brittle bodies, e.g. rock crystal, can be pulverised by very careful application of pressure as soon as the elasticity limit is exceeded. Spring's results demonstrate that these observations may be attributed to the fact that fissures in metallic bodies can easily be closed under sufficient pressure by welding together their edges. However, in the case of quartz and similar hard materials, this welding capacity is very low, probably because, as a result of the rigidity of the material, the crack edges are not able to come sufficiently close to each other.

By making the above proposal, we are using a circular argument, assuming something which still has to be proved. Put another way, we suppose that all crystals, at least to a limited extent, are endowed with the capability of permanent deformation without forming cracks.

Reusch has already proposed the mobility of crystal particles in order to explain his observations concerning the deformation of rock salt. He believed this to be related to the ability of crystal particles to slide along dodecahedral faces, by which we mean those faces which truncate the cubic edges. Fig. 2 is an attempt to demonstrate the sliding of rock salt particles for a crystal pressed

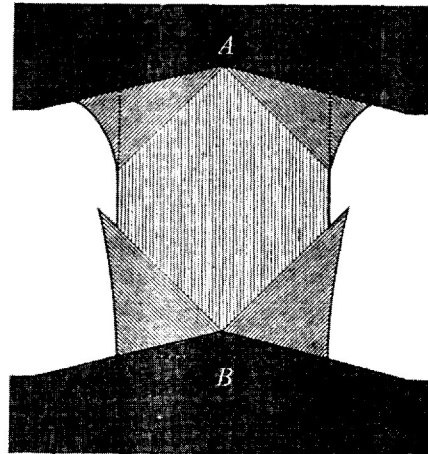


Fig. 2

between a hollow pyramidal upper punch *A* and an analogous lower punch *B*, whose initial shape is given by the dotted lines. It is assumed that the sliding only occurs for layers which are directly in contact with the punches and takes place only along the hatched lines.

Suppose such pressure were applied to rock salt and the expected sliding were indeed to take place only along the dodecahedral faces. In that case, the cleavage would have to remain undisturbed after the deformation, and the cleavage direction in the deformed particles should still be parallel to the initial cubic faces. However, Reusch himself had already observed experimentally a disturbance of the ability to cleave. Later observations, e.g. in the case of galena which can be cleaved in a similar manner, have also confirmed that the cleavage planes can be deformed to a greater or lesser extent.

From this one may conclude that real flow, without crack formation and without parallel displacement along defined gliding planes, is possible even for homogenous crystals. One may wonder how a such deformed crystal will behave, and especially how further growth will occur when we insert it in an appropriate mother liquor. Will it remain with the bent faces of the artificially imprinted form or will it gradually adopt the normal form? Perhaps it might even undergo a transition to a structureless amorphous state, which is considerably growth-retarded, since a principal difference between crystalline and amorphous bodies is the fact that the former can grow but the latter cannot, even under ideal conditions.

It has been shown by experiment that it is the first case which occurs. The crystals proceed to grow with bent faces, but not quite in the usual way, though apparently with a growth rate equal to that of normal faces. A disturbance becomes evident by the occurrence of internal tension, in such a way that each newly deposited layer can be taken as a thin flexible bent lamella which is attached to the surface but retains its own tension. Thus, by summing the effects, the resultant tends to cancel out the existing deformation. A bent crystal, for instance, shows a tendency to extend rectilinearly on growing. Not infrequently, especially in the case of very thin needles and lamellae, this process will be taking place, when, quite suddenly, the bent needle straightens up all at one go. Sometimes this occurs with such vehemence that the whole crystal bursts into two or more parts, in a certain sense nearly exploding.

Observations of this behaviour are known in abundance for microscopic crystals. It happens when the crystals are still extremely thin, hair-like or lamella-like (as in the case of so-called trichites), and which have been bent not by external forces but by internal forces (probably by action of surface tension). This behaviour can also easily be verified using artificially bent needles, e.g. those of caffeine, in which, however, the initial bend is only elastic and therefore temporary.

To date I have not been able to create a permanent deformation in larger crystals without destroying them completely. This lack of success is probably because we do not have a suitable apparatus which can perform the deformation both sufficiently slowly and without creating any shock disturbance. In order to exert pressure one could use, for instance, a rising gasometer bell jar, the rise in

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which will be caused by water which slowly seeps into the container. An alternative simpler method would use a bucket, into which water is poured very slowly, drop by drop, causing the weight of the bucket to increase progressively. This could work if the observation site were free from vibrations caused by passing carriages, etc.

Deformation tests of crystals involving either squeezing in a screw press (the so-called compression strength tester), or in a lever press (punch press), or by forging on an anvil, always resulted in aggregates. The initially homogenous crystals were smashed to pieces. However, the debris did not form a loose accumulation. Rather, by deforming fragments in such a way that their extremities nestled closely to the extremities of the surrounding fragments and welded together, the result was a semi-transparent, solid, connected mass. In Fig. 3 we see a microscope view of what happens when a crystal forged by this method to a thin lamella, e.g. salmiak, grows further. The small particles in contact with the solution can be observed growing further at their outer borders, in a skeleton-like fashion.

The single fragments are rather large when the deformation is weak, but the bending is too insignificant to expect any disturbance of the growth. In the case of very strong deformations the fragments are correspondingly smaller and the influence of the bending cannot be observed.

I have not so far found an approximation to the amorphous state which might have been expected from the usual ideas concerning the molecular constitution. Such an approximation might imply that a normal crystal, growing simultaneously in the same solution would gradually consume the whole material. This corresponds to the fact that amorphous bodies are without exception consumed by crystals which are brought close to them, provided they possess the property to dissolve, but they do not themselves grow in the solution. Thus the decreased content of the solution brought about by the elimination of the crystals can be increased again only by dissolving the amorphous bodies.

Crystals of the regular modification of silver iodide exhibit only a waxy consistency and can be spread with a dissecting needle on the object slide of a

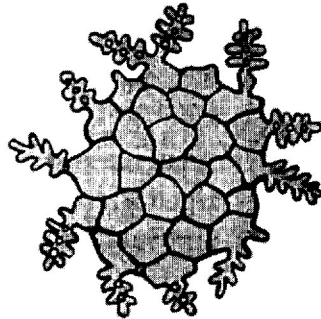


Fig. 3

microscope like hot sealing wax. Yet while they are growing, they very closely resemble thinly forged salmiak crystals between hammer and anvil. The same applies to deformed crystals of tin and lead which have been dipped as cathodes into appropriate solutions during microscopic electrolysis.

In the light of all these observations, it has not seemed possible to discover a substance whose crystals could be regarded as in a state of flow from direct observations, yet did not disintegrate and reform, but rather maintained their internal correlation under constant deformation in the same manner as do amorphous and liquid bodies. However, it seems that as a result of a recent discovery by Mr F. Reinitzer in Prague, such a substance, weakly fluid but crystalline, has indeed been detected. The nature of these crystals has not yet been fully understood, and perhaps optical illusions may be involved. Nevertheless, I have no hesitation in reporting the observations here, since so far it has proved impossible to construct an explanation of the phenomenon in terms of extremely soft crystals of a syrupy or gum-like type.

The substance in question is cholesteryl benzoate¹. In a letter in March of last year, Mr Reinitzer, to whom I owe the substance under investigation, told me the following about the contradictory behaviour of the substance which he observed:

'If one may so express oneself, the substance exhibits two melting points. It first melts at 145.5°C, forming a turbid but unambiguously fluid liquid. This suddenly becomes totally clear, but not until 178.5°C. On cooling, first violet and blue colours appear, which quickly vanish, leaving the bulk turbid like milk, but fluid. On further cooling the violet and blue colours reappear, but very soon the substance solidifies forming a white crystalline mass.

When the phenomenon is observed under the microscope, the following sequence is easily detected. Eventually on cooling large star-like radial aggregates consisting of needles appear, these being the cause of the cloudiness. When the solid substance melts into a cloudy liquid, the cloudiness is not caused by crystals, but by a liquid which forms oily streaks in the melted mass and which appears bright under crossed nicols.'

These observations indeed contain many contradictions. For, on the one hand a liquid cannot melt on increasing temperature and also at the same time exhibit polarisation colours between crossed nicols. On the other hand a crystalline substance cannot be completely liquid. That a pulpy mass of crystals and liquid was not present follows from the high degree of purity of the substance under investigation; the substance came for use in the form of totally clear and well-defined crystals. In addition, at the temperatures concerned there was no possibility of chemical decomposition, and furthermore through direct visual observation in a microscope

¹ Both the acetate and the benzoate of hydrocarotene also exhibit the same behaviour; see also F. Reinitzer, *Sitzungsber. d. Wien. Ak.* **94**, (2) 719 and **97**, (1) 167, 1888. O. Lehmann, *Molekularphysik* **2**, 592, Anhang.

it would have been very easy to recognise clearly the edges of crystals in the liquid, especially because of the strong influence of the former on polarised light.

Reinitzer's further investigations led to still more serious complications. From them one is led to the conviction that when the material is cooled below 178.5 °C, the modification which first develops is a uniform, solid and physically isomeric modification consisting of spherocrystals. When the system is further cooled it partially reliquifies, since the spherocrystals 'melt (during cooling!) beginning at the periphery, and swim as solid bodies in a liquid'.

Despite all these contradictions, in my own investigations I have really been able to confirm Reinitzer's results. The impossible here really seems to become possible, but as to an explanation I was at first totally helpless.

If the substance were crystalline – as I deduced from my own observations in another context – it must be possible to prevent the crystals getting into close contact with each other as they grow by adding a small amount of a solvent. Gaps should remain filled with liquid, which would allow the edges of the single crystals to be clearly seen, and so probably to identify their polyhedral shape or at least their ability to grow. However, my attempts in this direction failed because of a lack of an appropriate solvent, until Mr Reinitzer replied to my enquiry that he had succeeded in finding such a substance which formed by itself if heated over a longer period.

And in fact I too have been able to obtain this substance by heating the sample without a cover glass over a longer period. The images which now appear allow little doubt that the mysterious modification in the temperature span of 145–178.5 °C is really a solid, crystalline, totally homogeneous enantiotropic modification.

I have not been able to recognise any polyhedral edges in the bodies separated by liquid, nor a clearly observable ability to grow. But this may be caused by the fact that the state of aggregation of these crystals, as already supposed by Mr Reinitzer, is extremely similar to the liquid state. Thus the ability to produce sharply defined forms which are totally distinct from the real liquid state, is only extremely small. Nevertheless, the other properties of the small bodies clearly point in the direction that they are crystals. Only this hypothesis can reconcile all the observed apparent contradictions, and make the total behaviour of this substance consistent with that of other similarly behaved substances.

The behaviour of the substance, contaminated in the way indicated, under the microscope between crossed nicols is now as follows.

The substance is heated until a clear melt is formed, and then cooled down slowly. Small blue-white spots appear over all the whole liquid. These grow in number and finally cover the whole space, which now appears as a cloudy-white mass. On further lowering the temperature, plates of common crystals are generated here and there. These quickly grow and eventually consume the cloudy mass; conversely they disperse into it on heating.

When the temperature is held almost constant close to 178 °C over a longer period, the plastic white mass adopts a coarser structure. Some of these coarse grains show a black cross similar to spherocrystals, especially for samples which

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167, 1888. O. Lehmann, *Molekularphysik*

are not covered with a cover slip. Probably these are not multiply radial spherocrystals in a real sense, but only more or less complicated star-like aggregates.

The most striking change of the mass occurs if it is moved by pressing on the cover slip by means of a dissecting needle. Along the lines of flow the bright spots and spherocrystals run together, forming striations which become broader and broader on continued motion, and which finally may cover the whole visual field. However, they only appear bright if their longitudinal and transverse directions are inclined with respect to the principal directions of the nicols; otherwise they appear dark, just like crystals whose vibration directions are identical with those of the crossed nicols.

If one heats up the initial state or the striated state by only a small amount it becomes paler; conversely on cooling it becomes brighter. Depending on the thickness of the sample, instead of appearing whitish it can also exhibit various polarisation colours, like an aggregate of crystal lamellae of different thicknesses.

On heating more strongly, black dots occur here and there in the mass (cf. Plate III, Fig. 4). These are circular and become larger and larger, i.e. holes develop, filled by molten mass, so that the bright mass now forms a network.

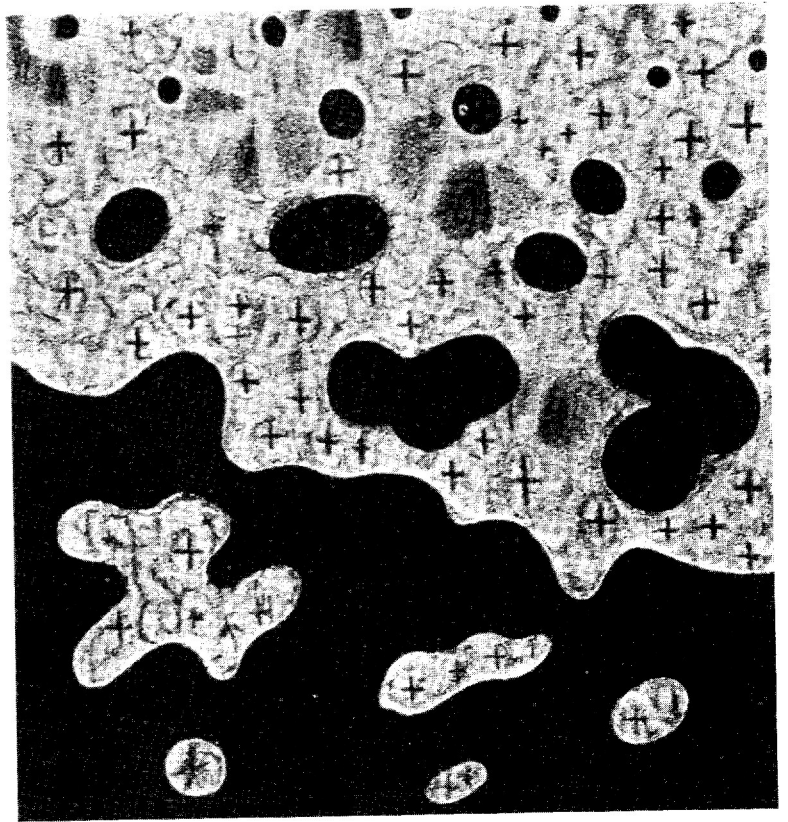


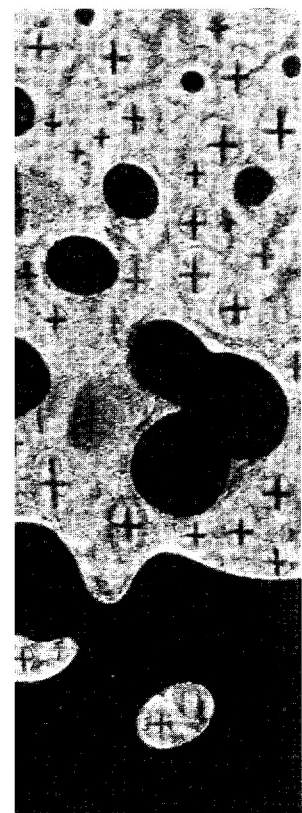
Fig. 4

are not multiply radial spheroplicated star-like aggregates. As moved by pressing on the cover glasses of flow the bright spots and which become broader and broader the whole visual field. However, in reverse directions are inclined with otherwise they appear dark, just like with those of the crossed nicols. In state by only a small amount it becomes brighter. Depending on the position it can also exhibit various lamellae of different thicknesses. Here and there in the mass (cf. the larger and larger, i.e. holes) the bright mass now forms a network.

Soon the network also melts as the connecting threads form an aggregate of bright spots, which would itself also eventually melt on further heating.

These isolated bright conglomerates are now most suitable for investigation. They possess razor-sharp edges and exhibit no observable surface tension effect; the result is that they can be viewed as melted crystal aggregates, and this conclusion is consistent with their polarisation properties. A closely analogous behaviour also appears during the melting of silver iodide; in this case likewise at first circular holes are formed (obviously caused by contact movement¹ in the developing melt), and finally very similar uneven rounded residues emerge while the holes are growing. However, a significant difference becomes obvious on cooling. Fragments of silver iodide rapidly grow like other crystals and enlarge to clearly observable and often multiply branched skeletons, albeit with much rounded edges and faces. The residues of our mysterious substance, however, exhibit no clear ability to grow, unless the small changes of polarisation colours are to be interpreted in that light. Rather, a dense precipitate of the aforementioned bright spots suddenly forms, which extends more and more and gradually coalesces with the larger residue (with additional movement). In this way many large isolated pieces of this curious material can be produced. The fact that there are really distinct pieces and not membranous or lamella-like species is easy to recognise in the vicinity of air bubbles. Here, as is known, a vigorous convection occurs which circulates the liquid around the air bubble from below and away from above, so that particles contained in the circulating liquid continuously roll, rotating about their axes, and so can be viewed from all sides. It will be obvious that under these circumstances they possess a certain stiffness, insofar as they generally keep their form during the movement. This stiffness, however, is extremely small, for if the whole mass is caused to flow even by a slight pressure on the slide, then the polarising particles will be totally distorted corresponding to the flow lines of the liquid. Thus the flow lines are diverted by an obstacle, even an extremely small one such as the surface of an air bubble. The mysterious crystals flow together with the fluid as if they were a part of it, except that they are endowed with a polarisation capacity. This extremely large plasticity, i.e. the near total lack of displacement elasticity, which even in the case of regular silver iodide is still clearly obvious, makes it easier to understand why the growth tendency of the crystals is so extremely small. As regards the behaviour of the substance which is not contaminated by an added oxidation product, the only difference to be seen is that it is not possible to isolate single parts of the plastic mass. The first violet colour appearing preceding the development of cloudiness is caused by the appearance of the first plastic crystals. As soon as this vanishes, the whole mass of this modification solidifies; to be more exact, it crystallises, for there is no sign of rigidity. When the mass is observed under the microscope but with a superimposed cover slip, a flow soon takes place and this is caused by the familiar crystals

¹ c.f. O. Lehmann, *Molekularphysik* 1, 271 and 493 ff.



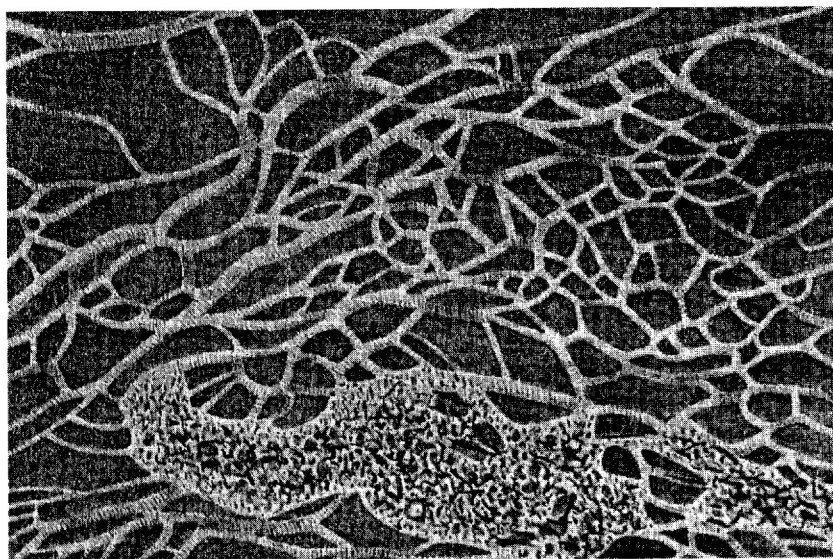


Fig. 5

developing sometimes here and sometimes there, and by the related volume change. This motion is the origin of the fact that the plastic crystals now seem to melt again. But actually they are only deformed and form an apparently homogeneous laminate. This strongly rotates the plane of polarisation of light, similar to a stack of mica lamella (more precisely: generating elliptically polarised light). They therefore appear intensely light-blue, or in different circumstances, can generate other different interference colours. Here and there, where the position of the crystals remained unchanged, they appeared bright, but run through by bright stripes (cf. Table III, Fig. 5). Because the stripes are in sharp contrast to the light-blue background, they look like bizarre 'oily streaks', generally pointing along the direction of the prevailing flow.

If the present interpretation of the observations is to be believed, a unique phenomenon is reported here for the first time. A crystalline and strongly birefringent substance has been observed which possesses such low physical strength that it cannot resist the effect of its own weight. As soon as it is not constrained by a liquid of equal density, it flows like syrup or gum; in this respect it would resemble the swimming oil ball of Plateau¹ were it to be removed from the influence of gravity.

Karlsruhe, 30 August 1889

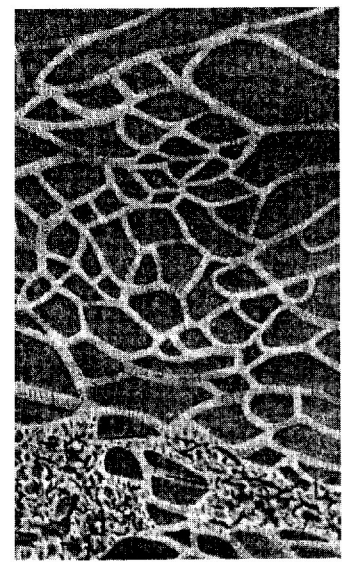
¹Joseph Antoine Ferdinand Plateau (1801–83) was a blind professor of physics in Ghent (Belgium). He is best known for his contribution to the molecular forces and elastic theory in liquids and thin liquid and soap films, leading to the so-called Plateau problem. This is the variational problem of minimising the surface area of a system with given volume (*ed.*).

Otto Lehmann was born in Constanz, Germany, in 1855. He was the son of a school teacher with an amateur interest in microscopy. After a peripatetic childhood he entered Strassburg (present-day Strasbourg in France, but at that time, like the rest of the disputed provinces of Alsace and Lorraine, in Germany) University in 1872, and obtained a doctorate in physical chemistry from that university in 1876. After academic posts in Mülhausen (present-day Mulhouse in France), Aachen and Dresden, he obtained in 1889 a full professorship at the University of Karlsruhe. Here he spent the rest of his life.



Lehmann was the author of an important physics textbook and an expert in polarising microscopy. Most of his career after 1889 was spent in studying liquid crystals, propagating their importance, and propagating *his* importance in their investigation. If not quite responsible for coining the term, he is responsible for advocating its wide use, even though it is now known to be a misnomer. He organised the first liquid crystal summer school in 1912, and together with Ernst Haeckel propounded the idea that liquid crystals were responsible for many of the processes of life. He was nominated, unsuccessfully, for the Nobel prize several times beginning in 1913. He died in 1922.

In the facsimile of the original article, we call the reader's attention to the reference to Plate III (Figs. 4 and 5) (which was detached from the article at the end of the journal volume), and to the woodcuttings; these were the figures which were clearly prepared specially.



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