CIOS A 32

I.G. FARBENINDUSTRIE AKTIENGESELLSCHAFT LUDWIGSHAFEN-ON-RHINE

Rep. No. 487

INVESTIGATION OF LUBRICANTS IN THE I.G. - KÄLTESCHRANK

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(Paper read on the occasion of the Discussion about matters relating to lubrication and lubricating oils, DVL. December 1941.).

Summary:

The I.G. - Kälteschrank (Refrigerator) developed at the Technical Experimental Station of Oppau has been found to possess many uses for investigating lubricants. With it the delivery and pressure of a pump subjected to the effect of cold can be ascertained and the influence of the lowering of the setting point can be appreciated. In conjunction with an indicator, actuated by the torque of a bearing, the whole process on the breaking away of a film of oil may be observed. Further research deals with the break-away force of oils and its dependence on extrapolated viscosity. The ratios between the break-away force and Walther's directional factor m are also considered.

In the investigation of lubricating greases in the I.G. - Kälteschrank it was found that the breakaway force increases very much when the rate of shear is reduced. It has been found furthermore that apparatus with a fixed torque, i.e. with variable rate of shear, are unsuitable for investigating greases. In proof of this, experiments with greases were carried out in the Schwaiger viscometer, and these clearly demonstrate the error. The latter is avoided by testing in the bearing ring of the I.G. -Kälteschrank (refrigerator), the shear speed remaining constant.

The behaviour of lubricants in the cold may be briefly summed up by saying that on cooling down, the viscosity increases. There are oils which gradually solidify and become glassy without separation taking place, and others which separate out paraffin crystals at a given temperature. With the former type of oil, the usual test methods generally determine a vague freezing point that is generally fairly low; this is however not really a specific point in the behaviour to cold, but merely the result of an approximate viscosity measurement. Oils of this type have a steep viscosity curve and break down in use far above this apparent setting point. The lack of a real freezing point is due to the fact that the predominantly naphtenic-aromatic oils possess a good aptitude for dissolving paraffin, or else paraffin is not present at all.

In the case of oils of the second type, on cooling paraffin crystals separate out and according to the homogeneity of the material, more or less sharp pour points are obtained. The crystals form a structure which contains the residual oil in the fluid state. The behaviour of such oils is often quite good in spite of their high pour point, as thanks to their paraffin character they have a very flat viscosity curve. The residual oil is therefore fluid at low temperatures and only offers slight resistance, e.g. on starting up an engine. The strength of the crystalline structure is generally too slight to increase this. The observations in determining the pour point are also deceptive as regards the quantity of solidification actually present.

It goes without saying that separations above the winter temperatures are not desired, as they impede the delivery of the oil. By the addition of pour-point depressants the commencement of crystallisation can be affected to a slight extent and the size of the crystals to a large extent, so that such oils only solidify at considerably lower temperatures (Fig. 1). Oils with definite setting points are not of course affected by such additives or are only affected to a slight extent.

The behaviour of lubricating oils in the cold has previously been investigated by the U-tube process of the German Railways or by the analogous process described by Vogel. By this means the so-called flow limit was determined, i.e. the stability of the crystalline structure, and then the flow properties of the crystal fluid were examined, which was determined by the quantity and nature of the crystals and also by the viscosity of the residual oil that could still be called a true fluid.

These methods were unsatisfactory, chiefly because the interpretation of the results meets with difficulties in practice. The Technical Experimental Station therefore commenced experiments in 1930 which were connected with known practical results. Tests were carried out on engines in the refrigeration chamber and the particularly characteristic conditions of these engines were simulated in small apparatus.

Experiments on engines are extremely tedious and although they possess the advantage of reproducing actual practical conditions, they also have the drawback that the effects of running, e.g. oil dilution can scarcely be overcome. In more accurate experiments to be carried out on apparatus it must not be forgotten that the effects in practice on the lubricants are generally left out of account.

Our experiments are already familiar through the Reports of the Technical Experimental Station. A collated publication is still lacking, apart from the treatise in the "Ringbuch of Aeronautics", - "Die Chemische Technologie der Schmierstoffe", in which Dr. Zorn also reports on our experiments. It would therefore be of some interest to furnish a survey of development up to date and the experience of recent years.

We initiated our experiments in 1930 with (and as far as we know we were the first in the field with this) the behaviour of a film of oil between piston and cylinder and after preliminary experiments on a Hanomag cylinder, evolved the apparatus shown in Fig. 2.

A cylinder is provided here which is surrounded with a cooling jacket, through which toluol cooled with CO_2 snow can be passed. The piston, which is coated with oil, is held poised by a balance arm. After cooling and at the end of the requisite period of waiting, the piston was set in action by compressed air and the pressure necessary to do this was used as a measure for the resistance.

Another apparatus dating back to 1931 is shown in Fig. 3; here two dishes moistened with oil, the outer one with a cooling jacket, are rotated in opposite directions, the torque being produced by weights on a cable drum! Apparatus of this type have already been mentioned in technical literature, at any rate as regards fluids.

All those apparatus failed because their construction was greatly affected and prejudiced by the cooling jacket. In spite of all possible care, it was unavoidable that the moisture of the atmosphere had a detrimental effect on the measurements by the formation of ice.

In 1932 we therefore adopted the principle of using refrigerators, in which the most varied machine components could be examined. Fig. 4 shows a section of such an apparatus. Characteristic of this type of construction, which was evolved by ourselves, is the cooling by solid carbon dioxide, which is kept in a separate compartment. By means of an adjustable grid this source of cold can be brought in contact with theexperimental chamber proper. By this arrangement not only can relatively very low temperatures be generated, but work can proceed comparatively quickly, as opposed to devices with refrigerating machines. The object being experimented with and the measuring instruments are visible through a window. The coarse adjustment of the temperature is effected by adjusting the grid, or else a finer adjustment can be made by electric heating, which in the new model is automatically controlled by a thermometer.

Refrigerators of this type have stood the test of time and are still used. From the large number of experiments conducted with lubricants in the cold state, a few examples will be picked out:

For instance, we occupied ourselves with the question as to how an oil pump behaves in the cold and built such a pump in the refrigerator (Fig. 5). By means of this apparatus the temperature could be determined at which the oil no longer flowed to the pump and furthermore the output and pressure could be observed as a function of temperature. The influence of the setting point is unmistakeable (Fig. 6), as at a somewhat lower temperature the oil no longer flows to the pump and its pressure therefore drops. The effect of an agent that lowers the setting point is thus clearly demonstrable. Investigation into the pump delivery output gave a similar picture.

In order to simulate conditions in a geared mechanism, the device shown in Fig. 7 was built, in which a rotor provided with blades rotated in a housing with internal fluting, and thus kneaded the solidified oil. The requisite torque was recorded by an indicator.

Experiments showed that in the case of solidified oil, the break-away took the form that the blade wheel filled with oil rotated freely as a disc within a corresponding cavity of the housing. As will be seen from the diagram traced on the drum, a very high torque first of all occurs, which speedily falls to a very low figure, and one that cannot be ascertained with exactitude. With oils having pour point depressants, on the other hand, a uniformly low torque is present, which is produced by a crystalline fluid that is obviously still viscous. The recordings shown are made by different springs. That actual maxima of the torque that occur are in the ratio of 12 : 1. All these experiments were highly valuable for assessing pour point depressants and their effect on various oils, particularly at a time when considerably less was known about the behaviour of lubricants in the cold than is known to-day.

The example of this kneading device shows that the crystalline structure may very well possess a strength that exerts a practical influence. It was first of all a surprise that in examining a bearing and a journal, no effect of the setting point was found. The device used has already been reproduced diagrammatically in Fig. 4. It may also be seen in Figs. 8 and 9, which at the same time show the method adopted in constructing the I.G.-Külteschrank (refrigerator) since 1938.

The bearing ring, in much the same way as shown in the case of the kneading device, is connected with an indicator which traces the torque on a drum. As in starting, the journal is suddenly set in motion after being stationary. For this, an electric motor with gear and coupling is provided. According to the type of motor, the speed of the shaft is about 60 r.p.m. The adjustment between the journal and the bearing ring corresponds to light running. By used different journals and widths of bearing (Fig. 10), various measuring ranges can be set. The spring can also be changed. This device represents definite progress compared with the earlier apparatus. Formerly, by employing suspended weights exerting a determined pressure constant forces were produced and the sliding speeds thus obtained were used as a measure for the resistance. In the new device, on the other hand, sliding speeds of a definite magnitude were produced and the resultant resistance forces were measured. According to Newton both methods can be used with true fluids and may also be applied to determining the viscosity. In the present instance of solidified oil, however, we are no longer concerned with fluids. It was observed that even in apparently amorphous oils i.e. oil solidifying without crystalline separation, there are traces of a certain structure at low temperatures. In these circumstances it was not possible for practical reasons to determine the sliding speed as a function of the forces in play. At low temperatures very often no movement occured, and the use of forces of different magnitudes produced no comparable results. Newton's axiom is not therefore applicable and the effect of the shear speed must be eliminated by the fact that the process takes place at a definite shear speed and the requisite forces can be measured.

We have always attached importance to observing the whole process on the break-away of a film of lubricant, not just contenting ourselves with measuring the resistance offered by a broken film. Fig. 11 shows that the torque which occurs at the beginning should be scrutinised carefully, as in the different oils it follows a characteristic course.

It must be confessed now that the first starting resistance of the oils film with ordinary oil is of no practical significance, as the resistance of the masses to be accelerated in an engine is very much greater. For a thorough research into the oils, the recodning of the break-away nevertheless is a valuable aid. It has been shown that the crystalline structure may very well give a measurable resistance, as is particularly the case with olive oil. The size of the crystals forming in the ring clearance is clearly restricted by the space available, so that the effect of pour point depressants is not demonstrable.

The peripheral force corresponding to the maximum torque, in relation to the surface of the film, was formerly called the adherence and used for assessing the oils. Since however the term "adherence" has been earmarked for a wetting process, in accordance with the new nomenclature, in future the expression "break-away force" will be used instead here. It must be borne in mind that this figure does not include the shear strength, so that the values indicated are only valid for a determined diameter of journal. If the assessment is not to include the strength of the crystalline structure, the resistance exerted by the broken oil-film may be used, and the height of the diagram can be measured after a definite interval from the commencement of rotation.

Experiments with lubricating oils in the cold waste a good deal of time as the oil-film takes a certain time to form a structure. (Fig. 12.). Very homogeneous oils very quickly reach a final state. In mineral oils a sharp rise in adherence is clearly demonstrable at first and amore gradual rise afterwards.

Particularly typical is the behaviour of a vegetable oil (Olive oil), whose oil film has not become stable after as much as 8 hours. This phenomenon is well known, and Erk reports, to take an example, that castor oil at -20°C does not assume a waxy form for days, which is then only transformed at + 8°.

The speed of growth of the crystals, which also depends on the viscosity of the residual oil, must therefore be examined over a corresponding period. Furthermore, after each experiment the apparatus must be warmed up again and furnished with fresh oil, as a crystalline skeleton that has once been destroyed grows up in a different way from a completely new one, where to beginwith no crystal nuclei are present. That the speed of cooling of such multiple mixtures must be watched is a fact that is as well known to the lubricant expert as it is to the metallurgist, in regard to the crystalline structure.

The examples given in Fig. 13 are representative of the many experiments that have been made on the adherence of oils. The effect of a low viscosity with the same viscosity index is to be found in Oil 1 as compared with Oil 2. Oil 3 has a slightly higher viscosity than 2, but has higher adherence owing to its steeper viscosity curve.

A completely different picture is afforded by olive oil, which at low temperatures and in spite of flat, i.e. good, viscosity curves, produces very high values, as the low temperatures cause its triglycerides to crystallise out more quickly than is the case at higher temperatures, and so the insufficient time of waiting of 2 hours is partly compensated for. The dependence on temperature as shown is therefore overcome by the speed of growth of the crystals.

With the other oils discussed, this is not the case as here the waiting period was adequate. Since olive oil contains about 30% glyceride and in the present instance had a setting point of - 13°C, the structural strength finally attained was great. Olive oil is therefore a special case showing that an oil with a very flat viscosity curve may be unsuitable for the conditions prevailing for starting up from cold, owing to the high degree of separation.

If 5% olive oil is mixed with Oil 1, the flat viscosity curve of the vegetable oil will have a favourable effect. The separations cannot exert any influence owing to their insignificance; possibly the triglycerides also remain in solution.

Fig. 14 accordingly shows that in the usual engine oils the separations characterised by the setting point have, in small quantities, no effect on the starting resistance. Of the oils "D" with 2.20°E_{100} or "B" with 2.15°, Oil "B", which is less favourable in starting resistance, has the lower setting point. Here the viscosity curve, which for this oil gives a VI = 72, is decisive. The thinnest oil "A" has a setting point of -24°. Owing to its obviously naphthenic-aromatic character, it has a steep viscosity curve, and therefore a high starting resistance, and it is safe to say that its setting point is an apparent one.

The temperatures at which starting is possible with oils - ignoring the dilution of the oil by fuel residue, may be estimated from the curves, if 2 Kg/cm adherence is assumed to be approximately the limit with a shear speed of 19 cm/sec, a limit with which most starters can cope.

The adherence is in the first place determined by the extrapolated viscosity and the behaviour of similar oils may accordingly be predicted with some degree of certainty, as will be seen from Fig. 15. Schwaiger also shows in his work a very considerable agreement between the " cold-viscosity" obtained by himself and the extrapolated viscosity.

At the other end of the scale, however, is olive oil, whose starting resistance is completely determined by the separation; viscosity only increases very slowly in the cold. For various types of commercial oil, Fig. 16 presents a contrast; here it will be seen that oils with the same extrapolated viscosity may have very different adherences. As a rule of thumb it may be assumed that thick oils behave more favourable and oils with a flat viscosity curve behave worse than is indicated by the extrapolated viscosity.

If for oils of the same viscosity at 100° the adherence at, for example - 10°, is plotted against Walther's factor, the characteristic ratios given in Fig. 17 will be obtained. A very similar result is obtained if instead of the direction constants the viscosity index or the pole height is used (Fig. 18). First of all it will be found that the starting resistance naturally diminishes with increasing flattening of the viscosity curve. This is particularly marked in the case of the thick oils, while with thin oils an improvement marked in the viscosity curve does not have such a powerful effect. The consequence of this circumstance is that the curves appear to intersect at a point. According to this ratio, which was discovered by us in 1934, it might be expected that with a flatness of the viscosity curve (which has not, indeed, been achieved), the oils as regards their starting behaviour, are quite independent of the viscosity.

As a rule attempts are made to ascertain the properties of lubricating grease by using pressure viscosimetres. It is difficult by this means to achieve a relationship with actual conditionespecially as (and this will be shown later on) the shear speed plays a considerable part in the assessment. We therefore quickly included grease in our experiments. The good results obtained by us in this connection were responsible for the DVL taking over an I.G. -Kälteschrank (refrigerator) from us in 1938.

Whereas according to the definition of the viscosity constants, the force to be used bears a relation to the speed produced, other laws apply to non-fluids. If, for instance, in the I.G. - Kälteschrank (refrigerator) grease was placed between bearing and journals, according to experiments conducted by Dipl. Ing. Halder, the adherence rises sharply if the shear speed is reduced for example to one third (Fig. 19). The direct-measured values of the torque naturally drop in accordance with the low speed and were converted to the normal speed of 60 r.p.m. Both experiments show that the greases react differently to a variation in the shear speed. From this it may be concluded that greases should not be examined in apparatus working with a fixed torque i.e. with a variable shear speed, and that for every quality figure the shear speed used must be indicated.

In order to prove this, experiments were carried out with an apparatus of this type. The viscosimeter developed by Schwaiger was used (Fig. 20). This is a dish or cylinder viscosity ., built into a refrigerator. A journal of 80 mm diameter is provided, surrounded by a sleeve in a loose seating. The journal is rotated by a cable drum, set in motion by a suspended weight. The shear speed therefore adjusts itself to the resistance. The values of the apparatus intended for testing lubricating oils in the cold were given in centipoises, although with cooled oils there is never any question of true fluids.

The resistance of the broken oil-film is measured. From the lowest attainable temperature of - 15° onwards, measurements were taken with rising temperature, without heating being applied. With current oils the apparatus gives very practical values, although it does not permit of any deep insight into their behaviour and only works correctly if the material being examined approximates to a fluid. Such is generally the case at a temperature of - 15° as the setting points of these oils, which have pour point depressants mixed with them, are lower. Fig. 21 shows that there is a certain ratio between the values of Schwaiger's apparatus and the I.G. apparatus. If the setting point lies close to the temperature at which the investigation was carried out, the influence of the shear speed will make itself felt by the fact that the apparatus indicates too high "Viscosities". This is perfectly understandable, for non-fluids have at low temperatures a certain stability, also called the flow limit, which finally is as great as the force introduced, so that no movement is possible and consequently the "Viscosity" is infinite. This is however incorrect, as the resistance is far from being infinitely great.

To avoid these difficulties by applying greater forces is not of course possible, as this does not mean a mere alteration of the measuring range. Indeed, the sliding speeds are altered simultaneously so that the measurements would be taken under quite different circumstances, and the magnitude would have to be altered whose effect on the measurements is precisely the cause of the present difficulty.

While the effect of the shear speed is demonstrable with oils under the said conditions, its effect in such a cylinder apparatus is shown particularly with greases having a soap structure filled with thin oil.

Different shear speeds are produced very simply by suspending different weights. Cases will occur where a trebled weight, i.e. torque will produce not three times but a hundred times the drum speed. Accordingly, with rising torque, a drop in "viscosity" will occur, as shown by the next figure, No. 22. It is interesting to note that the greases investigated alter their order of evaluation. Whereas Grease No. 1 is only slightly affected, Grease No. 2 passes from the first to the third place. Grease 1 is therefore less affected by the shear speed and bears more resemblance to a pure fluid than Grease 2.

I should like to mention expressly that Schwaiger's apparatus is intended for measuring oils and not greases. If nevertheless greases have been examined here, this was merely done in orderto demonstrate by means of a specially pronounced case, that cylinder apparatus yield useless values in all cases where the shear speed plays a part. High "viscosities" are given too high, because a low shear speed is obtained, and with low resistances the reverse is the case. As a result of this circumstance, a distortion of the "Viscosity curve" takes place, as the shear speed is less at low temperatures than at high. It is not however possible to correct the curves for flatness because, as shown above, the "viscosities" of the individual materials are affected very differently by the shear speed. Thus the "viscosity curves" are distorted in a manner which varies from material to material and is quite beyond control.

The peculiarity that greases are affected in different ways by altering the shear speed, may be sought in their more or less great similarity to true fluids. It is necessary to carry out a thorough investigation in this direction at different, but in each case fixed, shear speeds.

In examining greases, as excellent examples of non-fluids, it will be found that the path trodden by the Technical Experimental Station, (who were the first in the field) of measuring lubricants in the cold state at a fixed shear speed, was the right one.

Not only the resistance between bearing and journal is of importance; the brittleness of the solidified grease must be reckoned with, for the grease must always remain so pliable that it does not break out of the bearing. An interesting question arises also through the peculiar effect of water-content on the behaviour of greases in the cold. We hope to be able to contribute to the clarification of this and other questions within the scope of our research on the behaviour of lubricants in the cold.