

Development of Industrial Rheology.

Nils Gustafsson

Nitec AB, Box 1204, S-181 23 Lidingö

ABSTRACT

Rheological measurements can be separated in two main areas. One is research rheology, which is made mostly in universities and is also predominantly represented in rheological meetings. The other area is industrial rheology which is used for product development and quality control and this paper will discuss the development in this area.

INTRODUCTION

In many products the rheology has a critical influence on the end quality and the goal with industrial rheology is to increase the quality and decrease variations. However, this is not always easy while many products these days have a very complex structure and the possibilities for complex measurements during production are very poor.

In the R & D laboratories today there are very powerful rheometers available and it is relatively easy to design products which, through complex building and rheology can meet demands from the market, which was impossible 15 years ago. A complete rheological control in the production of such products is difficult as the time available for measuring is very short and many products need a long measuring time for detecting all properties. The state of art and today's opportunities for such measurements will be treated by discussing different instrumentation.

1. HISTORY

The history of industrial rheology is very much related to persons who developed instruments making reliable measurements possible for increasing quality of non Newtonian materials. During the first decades of this century viscosity was measured with different kinds of flow out devices - cups and capillaries like Marsh Fummel - made of different materials and ending up with more sophisticated units like Ostwald and Ubbelohde capillary viscometers which are still in use.

1.2. HÖPPLER

Höppler was working as a chemical engineer at Gebr. HAAKE in Berlin, Germany, at that time a chemical company producing starch products. The viscosity of these products was fluctuating very much between batches and he had to find a way of reproduceble measurement to control the viscosity. He developed the falling ball viscometer - actually the first controlled stress instrument - in 1930 and this is still used for production control in many industries.

1.1 BROOKFIELD

An important year is 1934, when the first Brookfield viscometer was built. The father of the Br. family was running a glue factory and there were many problems with keeping viscosity under control. His sons, who were involved in the business, started to develop something to measure the viscosity with and ended up with the first controlled shear viscometer - at that time

called a "Torque Meter". This unit is still today very much the same and is used for many purposes in the industry.

1.3. CONTROLLING SPEED/MEASURING STRESS

Important names in history of industrial rheology are Epprecht with the Rheomat, Brabender with the Visco-Corder and HAAKE with the Rotovisco. A great number of instrument for specialized measuring purposes have been built, which is not possible to cover in this abstract. With the technique of today with non friction air bearings and advanced driving and detecting systems these kind of instruments have become powerful tools in industrial rheology and are commonly named as Controlled Strain Rheometers.

1.4. CONTROLLING STRESS/MEASURING SPEED

Historic instruments are the "Falling Ball" and the Laray "Falling Ring" (used almost only for printing ink and paint). A relative new instrument is the dynamic Controlled Stress Rheometer. It was developed by the englishman Jack Deer and the first demonstration was made in 1968 after a decade of R & D. At that time, the driving was by compressed air. It gave a new dimension to rheological measurement and is today followed by a number of rheometers in different designs. Due to the simple driving technique by "drag cup" motors, the units are relative cheap in producing and therefore very useful and powerful tool for industrial rheology.

2. INSTRUMENTATION

The instrumentation used today for industrial rheology has to be separated in different parts as there are a great number of devices used. The ideal for a producer is of course to measure continous on-line, but from practical reasons there are limits for this – both from the processes and from the complex rheology of the product.

2.1. OFF-LINE

Off-line measurements can always be made in reliable and reproduceble way and will cover all parts of the rheology of the product. It is, however, time and work consuming and demands often high knowledge in rheology from the operator to evaluate. Because of this QC is still often made with rather simple devices.

2.1.1. CAPILLARY

Under atmospheric pressure used in oil and pharmaceutical industry for Newtonian materials, in late versions even oscillating for measuring elasticity. For high shear rates up to 10^6 s^{-1} for simulating coating process. Simple flow through cups are still much used for paint.

Under high pressure for high viscous materials like polymer melts and rubber. Can operate in controlled shear or controlled stress mode. With modern software elasticity can be calculated. Die swell can be measured and calculated. A special kind of QC is the Melt Index for polymer melts, where the melt is pressed through a capillary by constant stress. The weight of the outflow over time is given as measured value and is a quality standard for polymers.

2.1.2. CONTROLLED SHEAR

Are used as QC for all kind of materials measuring simple viscosity at fixed rate and rate flow curves. Dynamic elasticity and structure recovery used mostly only in R&D.

2.1.3. CONTROLLED STRESS

Are used as QC for all kind of materials measuring simple viscosity at fixed stress and stress flow curves. Creep test, dynamic elasticity and structure recovery used mostly in R&D.

2.2. ON-LINE

Continous on-line measurement can be used as controlling of a process (keeping

viscosity constant or break up at a certain viscosity). Installation can be made in tube as by-pass or in main stream and directly in a container. Process conditions are often a limiting factor for this kind of installations. However, for some applications it is working without any problems.

2.2.1. CAPILLARY

A stream of material is forced through a capillary and the viscosity is measured as a function of pressure drop in the capillary. Due to the risk of disturbing the flow, the material has to be free from particles bigger than 1/10 of the capillary. Low pressure units are used for oils and low viscosity fluids. High pressure units are used for polymer melts. Latest versions have conical capillaries, giving opportunities for two viscosities or melt indexes simultaneously and even dynamic oscillation is now possible. If flow rate can be changed, flow curves can be obtained.

2.2.2. CONTROLLED SHEAR

Is working like off-line units but built for process conditions. Can work in tubes over wide pressure, temperature and viscosity ranges or as immersion units under atm. pressure. As a condition for good measurement is laminary stream and installation almost always has to be made as a by-pass where these conditions can be hold. Much used for painting processes keeping a constant viscosity and low viscosity materials processes.

2.2.3. VIBRATION/DEFORMATION

Different viscosities give different damping effects on a vibrating or a moving rod as a sensor. This effect is used for continous viscosity measuring units of different kinds. The moving of the rod can be very different and latest versions of these units are working with a very small amplitude at a very high frequency. This gives such a small deformation at the material so even structure recovery can be detected if the material is resting around the

sensor. The technique is new but very promising for suitable installations.

3. THE FUTURE

It is obvious that still more complex rheometers with multi-functions will be developed. Additional systems as IR-analyze, thermo-analyze, MWD-analyze and others will be integrated and used together with industrial rheology for total characterization of the material. It is a strong movement to measure as close to the source as possible and therefor there is a demand on the market for more useful on-line rheometers.

4. CONCLUSION

Industrial rheology is mostly from practical reasons different from the universities, where education in rheology is still very much related to basic material research. Development of instrumentation is accelerating and the market situation is pressing suppliers of rheometers to get technical specifications far beyond practical relations to industrial applications and use.

Even if powerful rheometers are purchased, the use of all facilities can still be very poor. To partly overcome this special softwares are developed, where complex measurements can be made and evaluated automatically. Development of on-line rheometers for complex measurements is in progress and will have a more profound influence for industrial rheology in the future.