

# CHALMERS



## **Development of two methods to evaluate lubricating greases using a rheometer**

**Bachelor of Science Thesis in Chemical Engineering**

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## **Abstract**

Lubricating greases are crucial for both industry and every day applications. Lubrication has become a natural part of all moving equipment and lubricating grease plays an important role in keeping different machinery running without failure.

Even if it is widely known that grease is vital for many devices, deep and specific knowledge of how grease behave in terms of rheological stresses is scarcely found.

The scope of this project was to investigate and develop a standard method of conducting rheological measurements on lubricating greases. Two analytical methods to evaluate apparent viscosity and flow point were developed and evaluated.

It was found that different greases did have measurable differences and that the methods evaluated could assist in comparing different greases to each other as well as in selecting the best grease for specific equipment. At higher temperatures a local minimum for both apparent viscosity and flow point was found. The root of this minimum is probably found in the thickener system of lubricating grease.

The development of a new standard method for measurements of lubricating greases requires further investigation of why grease behave the way it does under rheological stress.

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## **1 Introduction**

Modern society of today has faced a rapid development in the manufacturing sector. A large number of production lines and manufacturing sites are today completely dependent on machinery. This dependency has many advantages but also some disadvantages. One disadvantage is the sensibility when the machinery dysfunctions or experience complete failure. To prevent or at least minimize these scenarios the machinery need to have a solid construction but also a good lubrication. That is why lubrication is of great importance. Lubricating grease is the dominating lubricator for applications where oil would not stay e.g. in roll bearings. It is possible to say that grease is to the machinery what blood is to humans. It is essential for both life time and good performance.

## **2 Background**

Greases are complex compounds and demands great experience and knowledge to manufacture to obtain specific behavior. Today, one of the most important properties of grease is the consistency. Consistency is normally measured and reported with a universal classification system, the so-called NLGI-system[1]. The test is a cone penetration which measures how deep a cone can penetrate the grease in five seconds. This measurement gives the grease a certain NLGI-number. This method is suitable for production control ensuring that each batch has the same consistency.

Problems occur when greases are chosen for new applications or even when old applications need improved lubrication. If the choice is solely based on the NLGI-number the only thing taken into account is the consistency of the grease, usually tested by how deep a cone can penetrate during a certain time interval. This is constraint and the results are somewhat non-scientific which makes it difficult to use the results for anything else than production control[1]. This method does not say anything about the grease structure, properties or its rheological behavior.

To be able to obtain the most favorable lubrication some parameters have to be studied in detail. By looking at greases behavior and properties when exposed to different forces and environments greater understanding could be reached on how to create the most favorable functionality. To improve the selection process of grease, a new universal standard of evaluating grease is needed. This need arise when different greases are to be compared and when deciding which one best suits a certain application. To introduce new universally acceptable standards is huge work. Two possible new standards could be the measurement of apparent viscosity and flow point. These two standards would provide more solid knowledge of grease rheological properties and would also be possible to use as engineering values when designing machinery and other equipment that needs lubrication[2].

### 3 Lubricating Grease

The definition of lubricating grease by American Society for Testing and Materials (ASTM) is “a solid to semi-fluid product of a thickening in a liquid lubricant, other ingredients imparting special properties may be included”[1]. This definition can be somewhat obscure and hard to understand although it correlates to the nature of greases complexity. Lubricating grease consists of a structure where different entanglements and bindings generate a large and complex lattice. This common nature of lubricating greases contributes to big challenges for the research and development of new products. To reach greater understanding of the behavior and properties of lubricating grease the whole grease industry, both producers and most importantly customers, need to improve their way of considering grease. Up until today the production of grease is often a trial and error based procedure where the earlier mentioned consistency has been the most important property[2].

Another important reason to why a new standard needs to be applied is the fact that grease is used in all types of different environments where it is exposed to a number of different types of stresses. One of the most important attribute for grease is to have good mechanical stability. Temperature is probably one of the conditions that affects grease the most. Some products need to withstand hot environments whilst other might need to be mechanically stable at a different temperature range. A good example of this is an excavator. A cold winter morning the temperature can be well below -20°C whilst in the summer it is above +20°C. It is good to keep in mind that it is not only the temperature that affects grease in a certain environment or application. Often other parameters like strain, shear and rotational forces might influence grease giving a synergetic effect. During such circumstances it is of great importance that the grease functions properly. That is why a deep scientific understanding of grease is crucial.

#### 3.1 Grease components

Lubricating grease is usually built on the same basis, often consisting of at least three components:

- Base oil
- Thickener
- Additives

The configuration of grease is generally 85 % base oil 10 % thickener and 5 % other ingredients, often different types of additives.

##### 3.1.1 Base oil

Base oils used to produce lubricating greases are usually *mineral oils* which are the most common but there are also *synthetic oils* and for some special application vegetable oils. Vegetable oils are more often used in the food industry[3]. There are however so called white mineral oils that are competing even in this sector[4].

The solubility is often regarded as the most vital factor when choosing base oil. It is of great importance that the solubility and interaction between the base oil and the thickener is properly balanced. By carefully choosing the right base oil based on solubility will most certainly improve the functionality of the grease. By doing this, greases will have higher yield since less thickener is needed to obtain a specific consistency, retrieving a faster and better mixing during the manufacturing process, this also provides less oil separation and better storage stability[5].

Another key factor when choosing base oil is viscosity and its temperature dependent behavior. Often base oil is regarded as either high- or low- viscous. High viscous base oils are used when dealing with temperatures at and above ambient conditions. Low viscous oils are used because of its eminent low-temperature properties and fairly good heat transfer ability[5]. Other important aspects when choosing base oil is, oxidation stability and evaporation loss.

### 3.1.1.1 Mineral oils

Mineral oils are purified and extracted from crude oil in refineries. Depending on the heritage of the crude oil and separation processed used, mineral oils has a wide content variety in terms of chemical components, this calls for substantial quality control of raw material to maintain a high standard with no or very small deviations[3]. There are three subgroups of mineral oils distinguished by the carbon bond of the mineral oils[6].

- Aromatics (Excellent solubility)

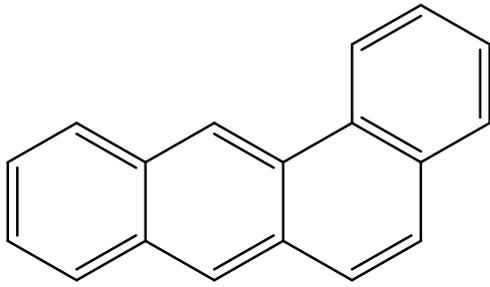


Figure 1. Aromatics structure

- Naphthenics (good low-temperature properties)

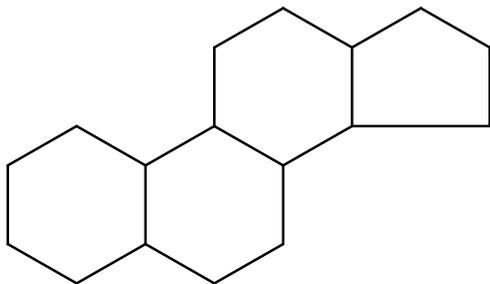


Figure 2. Naphthenic structure

- Paraffins (high viscosity index, low sulfur content and good oxidative stability)

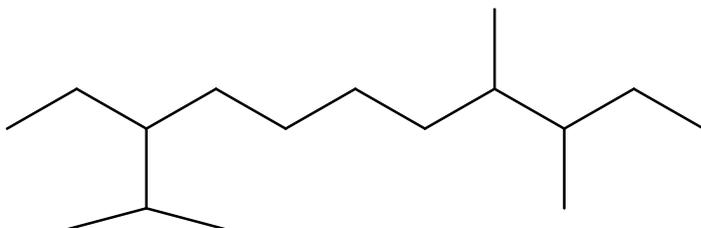


Figure 3. Paraffinic structure

### 3.1.1.2 Synthetic oils

Compared to mineral oils, synthetic oils have one big advantage, it can be customized from different chemical compounds to best suit the grease structure and later for a specific application[5]. In addition to this, synthetic oil has many advantages such as improved stability, less internal friction, and lower toxicity risk.

Synthetic oils are therefore a more appropriate choice when certain applications demand a wide range of operating conditions or chemical resistance. Other key factors that make synthetic oil superior to mineral oil are that it has compatibility with rubber and plastic along with superior electrical properties[5]. There is yet another important aspect that should not be neglected when choosing base oil. The environmental impact is much lower for synthetic oil than for mineral oils[3]. Although the cost of producing synthetic oil is higher than for mineral oil the previously mentioned advantages are of greater importance.

### 3.1.2 Thickeners

Lubricating grease has very complex structures and can have a large number of components depending on the application[2]. Nevertheless the most important component in lubricating grease is the thickener. Today there is quite a large supply of different thickeners, most common are metal soaps but there are also non-soap thickeners like clay, silica gels and polymers. The primary objective of the thickener is to supply a solid structure to keep its consistency during non-operating conditions but it also provides mechanical stability to the grease both over time and under the influence of different forces e.g. shear stress[5]. The most common question grease manufacturers face from customers is to produce multipurpose greases. To create this multipurpose grease can be regarded as the most challenging aspect within lubricating grease industry of today and requires extensive scientific work.

#### 3.1.2.1 Metal soap thickeners

“Soap is by definition the reaction product between a fatty acid and alkali. Metal soaps are commonly referred to as a sponge containing oil”[1]. Figure 4 illustrates the sponge structure; here

the base oil has been extracted from the grease which enables a better view of the thickener system.

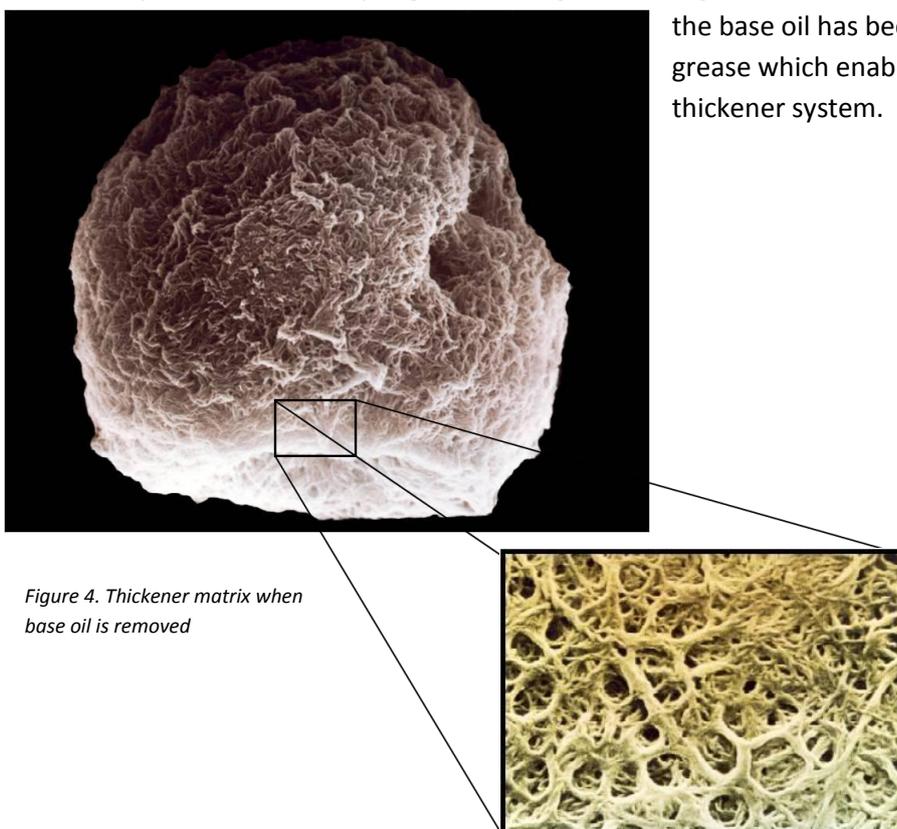


Figure 4. Thickener matrix when base oil is removed

Metal soaps are the most common and widespread thickeners in lubricating greases. It has its origin way back when modern grease started to be produced, during the industrial revolution. At that time calcium, sodium and aluminum soaps were the most frequently used thickener but when more challenging application areas were developed there was a need for higher standards of lubricating greases[2]. Lithium soaps became a commodity during World War I and II, when the aircraft industry started to develop rapidly and were in need of grease with good thermal stability, shear stability and water resistance.

### **Fatty acids**

Fatty acids contain carbon chains with one carboxylic group. The chain can have different lengths but normally contains between 2-18 carbons. The carboxylic group makes the shorter chains water soluble, while the longer chains are hydrophobic, carboxylic group or not. One commonly used fatty acid in grease manufacturing is 12-hydroxy stearic acid. The hydroxylic group is situated on carbon 12 (starting from the Carboxylic carbon). Stearic acid is a saturated fatty acid containing 18 carbons. This length is categorized as long chained (>12 carbons), this chain length makes it insoluble in water.

#### **3.1.2.2 Lithium soaps**

Lithium soaps used to be based on beef tallow but are today solely based on castor oil derivate, 12-hydroxy stearic acid seen in figure 5.

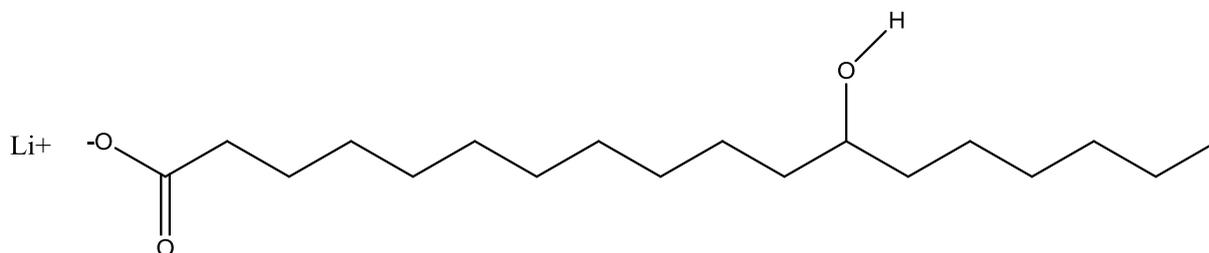


Figure 5. 12-hydroxy stearic acid

Along with previous mentioned advantages, lithium soap possesses excellent compatibility with a wide range of additives. This quality makes lithium soap favorable to customize for special applications and environments. The only disadvantage with lithium is its low temperature properties where the pumpability at temperatures below -10°C can be very limited.

### 3.1.2.3 Lithium-complex

Lithium-complex soaps are usually, compared to lithium soaps, based on 12-hydroxy stearic acid but with a dibasic acid however there are also other formulations. This formulation forms a structure within the network structure forming a lattice with longer carbon chains and more intermolecular bindings. A schematic structure can be seen in figure 6.

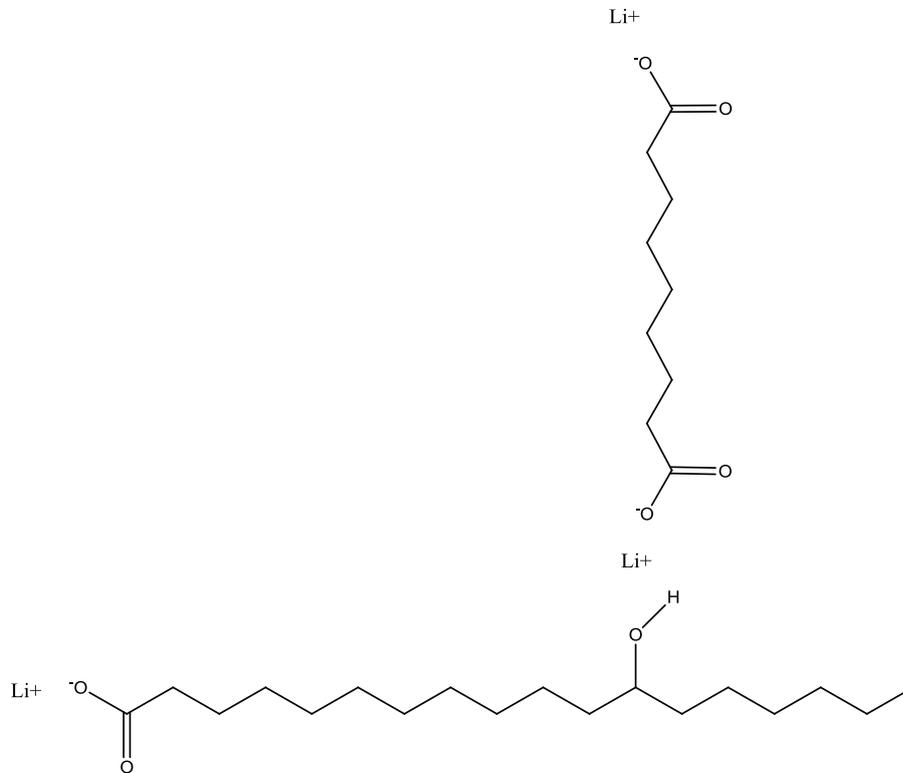


Figure 6. Cross linking structure in Lithium complex

Because of the rigid structure (see figure 6) lithium-complex soaps succeeds lithium soaps both in terms of thermal stability and in shear stability. Certain lubricating grease formulations using lithium-complex can serve during operating conditions up to 200°C. During such extreme conditions re-lubrication is essential. Because of the rigid fiber structure of the matrix the lithium complex also has a great capability of retaining the lubricating base oil which makes it excellent for application that requires high shear stability and load carrying capacity e.g. in steel and paper industry but also in roll bearings in cars and trucks. Another important factor is that it has great pumpability which is a great quality when producing grease but also when used in centralized lubricating systems. All these excellent properties that lithium-complex possesses makes it a multi-purpose grease with excellent development possibilities[5].

### 3.1.2.4 Non-soap thickeners

Metal based soaps are used in the majority of all greases but there are some non-soap thickeners, inorganic solids such as polymer, clay and silica gel just to name a few. These special thickeners usually have a very narrow spectrum of usage areas. They are very limited in terms of performance but have found few application areas where they are able to function properly[5].

Polymer thickened greases has good overall properties and moderate thermal stability. It can be used in a wide range of applications and has long re-lubrication intervals. This property is an advantage especially when used in windmills. Clay has somewhat good thermal stability and can

therefore serve as a decent thickener at temperatures up to 200°C. It is quite useful at high temperatures since it will neither melt nor drop.

Silica gels are today rare although when developed they were intended for low temperature applications in small mechanical devices. However since this kind of thickener experienced oil bleed and were very unstable it was not fulfilling its intentions and was therefore replaced by more suitable thickeners. Today silica gels are found in very few applications although it fulfills an important role in environments where metals e.g. aluminum are present. Here silica serves as an inhibitor of alkalinity.

### **3.1.3 Additives**

Lubricating greases of today are, as mentioned before, based on scientific grounds and almost all greases contains some form of additive. Additives contribute with the final touch to the grease in terms of optimal performance[7]. The most important features of additives are solubility (to comply with the base oil) and insolubility (to enable use of solid lubricants). These two properties are most crucial depending on the wanted effect. Some lubricating greases demands active additives on the metal surface to compete with polar molecules in the soap structure[7].

The most common additives serve as:

- Corrosion inhibitors
- Antioxidants
- Extreme pressures and anti-wear agents
- Tackifiers

## **3.2 Extreme environments**

Lubricating greases have to withstand and properly function in extreme environments. One example of when lubricating grease is severely challenged is in its marine applications. At sea there are a number of features that need to be fulfilled. These include mechanical stability in a wide temperature range, good adhesion to the lubricated surface and good cohesion of the grease to withstand water spray-off but also to inhibit corrosion from saltwater. Another extreme environment is faced when drilling tunnels. When drilling tunnels extreme loads are placed on the machinery which requires a lubricant with very high pressure resistance. In addition the drilling normally takes place under ground in a cold and often moist environment. This calls for good low temperature properties at start up but later also good high temperatures after many hours of drilling. The moist or even wet environment requires grease that will neither be affected by water nor spray off.

## 4 Rheology

The word rheology comes from the Greek rheos which means “to flow”. It is usually defined as the practical and basic knowledge of materials deformation and flow behavior.

Rheology measures and enables study of the relation between force deformation and time.

Practically rheology is called rheometry which is a controlled way of deforming materials to study the materials behavior when subjected to certain deformation energy. The materials will respond to this in one of three different ways[8].

- **Viscous materials**
  - Loss of all deformation energy, this will make the sample flow. As all deformation energy is lost the sample will not regain its original shape when the stress is relieved (i.e. irreversibly deformed). A good example of a viscous material is water.
- **Elastic materials**
  - All deformation energy is stored which means that no flow occurs, independent of the applied force. Since the energy applied to the sample is stored within its structure this energy will be used to regain its previous form when the applied stress is withdrawn, commonly known as the rubber-band effect.
- **Viscoelastic materials**
  - Some deformation energy is stored within the sample and when the applied stress is withdrawn the sample will partly regain its previous form. An everyday example of a viscoelastic material is toothpaste.

Viscosity  $\eta$  [Pa s] is the ability to resist flow, in other words the internal friction of the sample. High resistance is equal to high viscosity and low resistance to low viscosity. Viscosity gives information about material properties when pumped, swallowed, stirred etc[9].

Elasticity  $G$  [Pa] is the degree of structure within the sample. Higher value indicates more and/or stronger internal bonds and/or cross linking. Elasticity gives indications about material storage ability, stickiness, lubrication and handling properties[9].

Strain [%] is the relative deformation of the material being sheared, in other words how much is the material deformed applying a given force.

Shear stress  $\sigma$  [Pa] is the force per unit area that causes the sample to flow, in other words how big is the force per area unit at the very moment when the sample undergoes a phase change and starts to flow.

Shear rate  $\dot{\gamma}$  [ $s^{-1}$ ] is the deformation per unit time, it gives information about how quick the deformation is and relates deformation to the time elapsed.

## 4.1 Rheological measurements

To conduct rheological measurements there are three main devices to use[9].

**Rotational viscometer** - only measures viscosity

**Rheometer controlled rate CR** - Can apply controlled shear rate or controlled strain. Measures the corresponding shear stress in the sample tested.

**Rheometer controlled stress CS** - can only perform controlled shear stress and measure the corresponding shear rate or strain in the sample. One disadvantage is that the strain is not always controlled, which makes it difficult to interpret data.

The rheological measurement device used to retrieve the data in this report is a rheometer with controlled rate (Anton Paar Physica MCR 301) in this report mentioned as the MCR (Modular Compact Rheometer), illustrated in figure 7a,b and c below.



Figure 7a The Physica MCR



Figure 7b Probe lifted to apply grease



Figure 7c Probe in measuring position

The MCR can perform measurements with both rotational and oscillating movements providing improved data for both comparisons and conclusions. Both rotating and oscillating movements can be performed with a probe giving either plate-plate or cone-plate measurements. The advantage of using the cone probe is that the shear will be equal throughout the sample, this is not the case when using the plate but the difference is negligible. The major reason to use a plate probe is that it is advantageous for samples with sensitive structures[9]. Even a small penetration into the sample may affect its rheological behavior, this could give unreliable results.

## 4.2 Thixotropy

Thixotropic fluids display lower viscosity over time at constant strain. This behavior can be studied by so called loop tests where a sample is subjected to low shear rates increasing to high shear rates and then back to the starting point. As seen in figure 8 a non thixotropic liquid will go back on the same path while a thixotropic liquid will make its way home by a new path. It is possible to compare how thixotropic a liquid is by comparing the area in between those two paths. The liquid with the largest area is the most thixotropic[9].

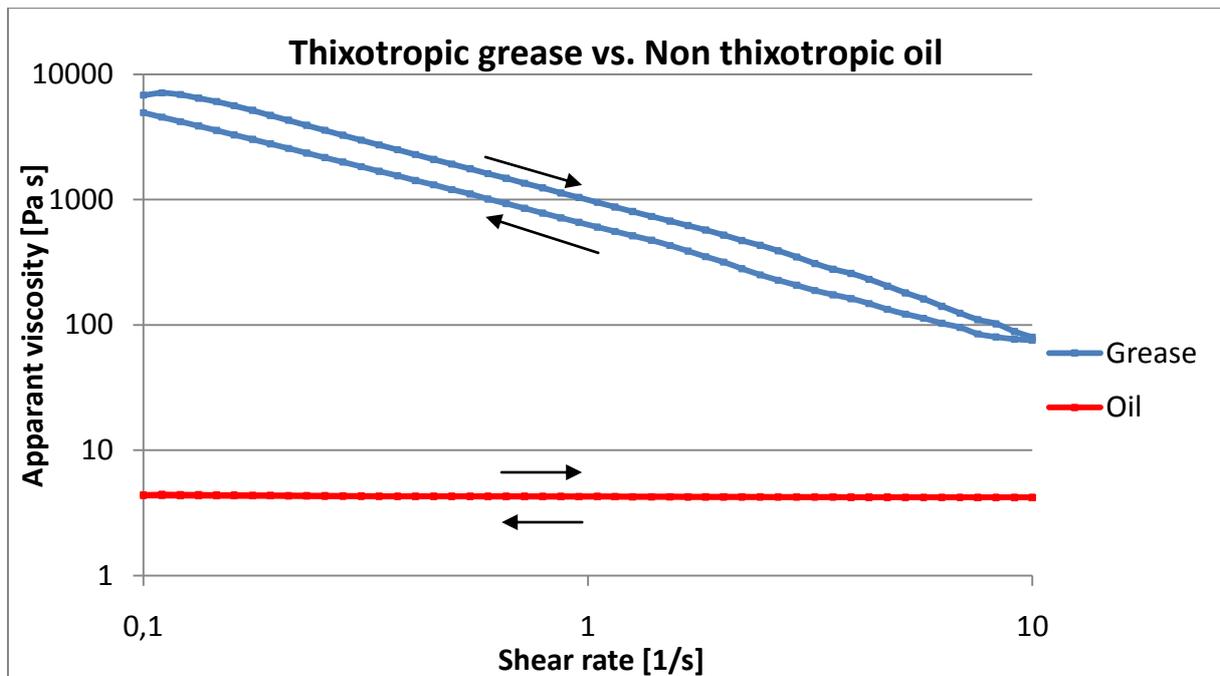


Figure 8. Loop test comparing thixotropic grease and a non thixotropic oil.

## 4.3 Shear thinning (pseudo plastic)

Shear thinning fluids display lower viscosity with increasing strain, in other words their viscosity is reduced when the stirring is enhanced. This property is common among lubricating greases and provides a problem when equipment is stopped. This problem occurs since the amount of energy required forcing the grease to start moving is much larger than the energy needed to keep it moving with continuous flow.

#### 4.4 Apparent viscosity

Grease is not a fluid and can thus not per definition have a viscosity but it inherits some properties normally seen in liquids[10]. Thus the term apparent viscosity is normally used. Apparent viscosity could easily be measured with a rheometer by implementing a logarithmically increasing rotational force to the grease at constant temperature. In figure 9 a typical low temperature response from a rotational test is seen.

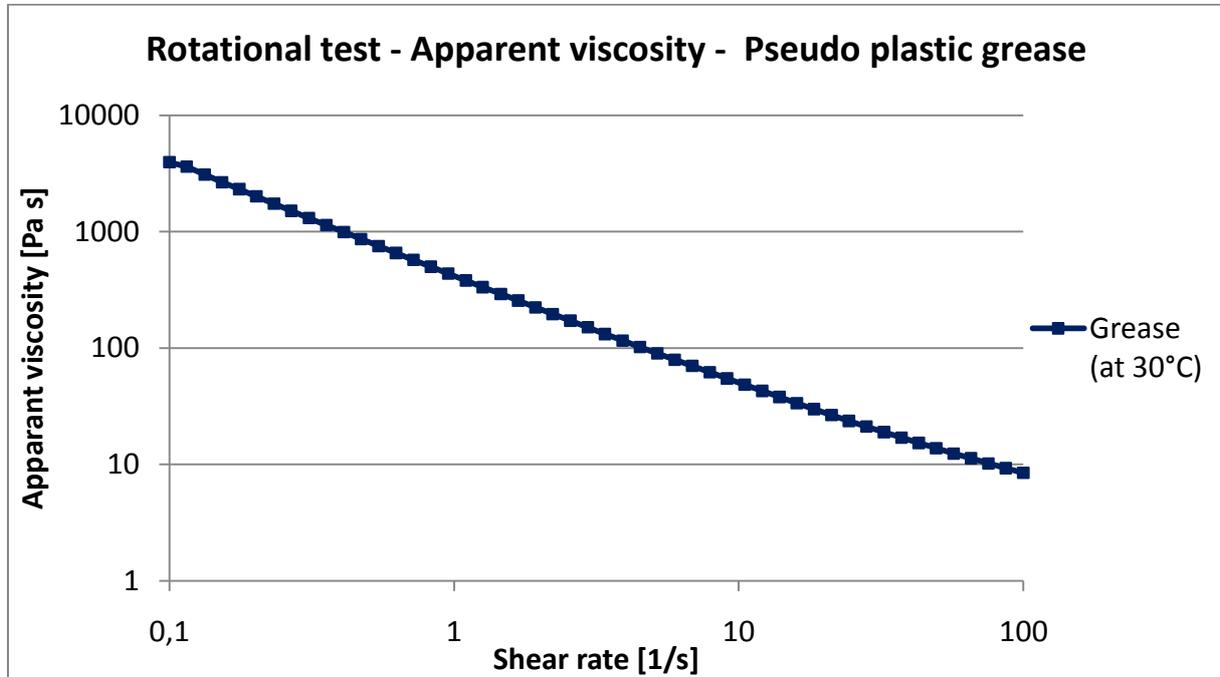


Figure 9. Typical response from a pseudo plastic grease exposed to rotational forces in a rheometer.

## 4.5 Flow point

Flow point is the point where the gel structure (storage modulus  $|G'|$ ) is equal to the liquid (loss modulus  $|G''|$ ) [10].  $G'$  is a value of the measured deformation energy stored by the sample and  $G''$  the deformation energy used by the sample. This behavior is due to grease viscoelastic properties. All viscoelastic greases have storage modulus above loss modulus when flow point/strain is plotted see figure 10.

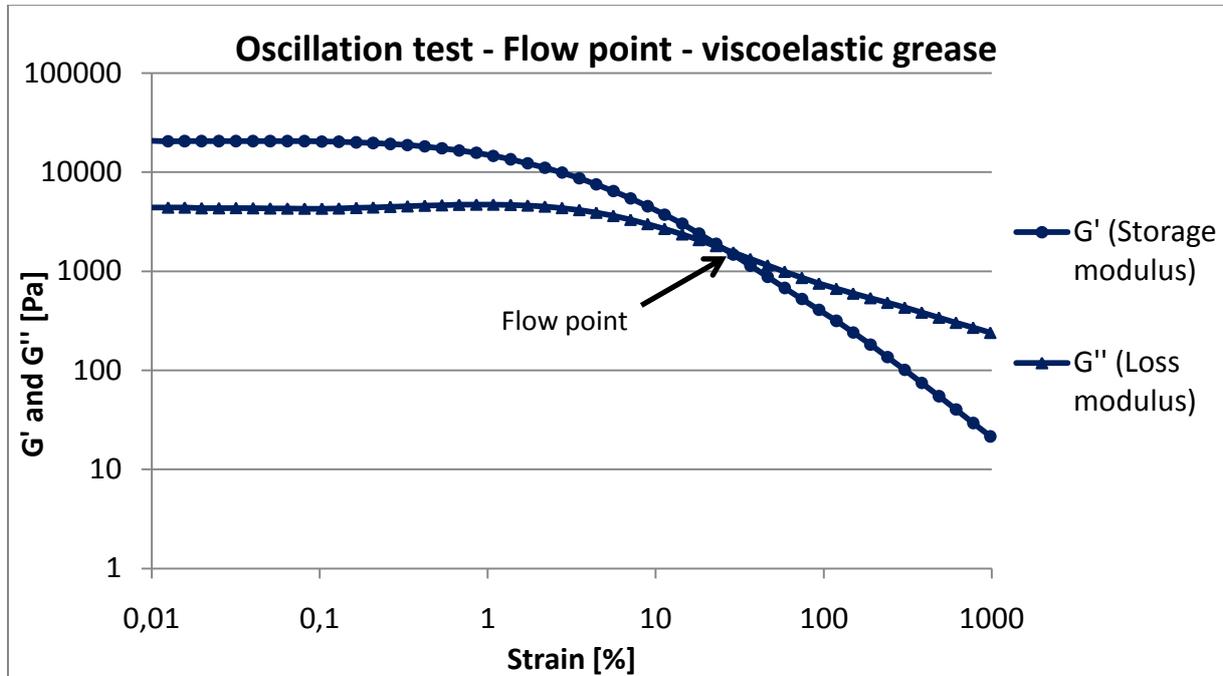


Figure 10. Typical response from a viscoelastic grease subjected to oscillating forces. Note that storage modulus always appear above loss modulus before they intercept at flow point.

Exposing grease to a logarithmically increasing oscillating force the grease starts to shear. To start with the material exhibiting a gel like behavior  $G' > G''$  but as more shear stress is applied the internal structure is broken causing  $G'' > G'$  and the material shows a liquid behavior [10].

This change to liquid could be studied by measuring complex modulus  $|G^*|$ . This is the combined effect of storage modulus  $G'$  and loss modulus  $G''$ .

## 5 The rheological study

To conduct a rheological study an analytical method was chosen and simple base greases produced. Cooking base grease without additives was made to simplify the evaluation of the data perceived.

### 5.1 Analytical methods

The analytical methods used while measuring the rheological behavior of greases was as mentioned before, apparent viscosity and flow point. These two methods were based on the same premises. After applying the grease to the bottom plate of the rheometer both measuring programs used a relaxation period of five minutes, ensuring that no internal movements within the samples structure occurred and to make sure that the grease was correctly and homogenously temperate. The temperature interval used was  $-20^{\circ}\text{C}$  to  $100^{\circ}\text{C}$  with an interval of 10 degrees between each data series. Both programs had 50 measuring points with five second intervals. These measuring points were collected and plotted in a diagram. The last step was to heat the plate to  $25^{\circ}\text{C}$ , this to ensure good and safe removal of grease from the plate after each run. No data was recorded at that time.

- a) **Rotational - Apparent viscosity (shear rate ramp)** was measured, in the unit  $\text{Pa}\cdot\text{s}$ , by applying a logarithmically increasing rotational force, shear rate. The interval was 0.1 to  $100\text{s}^{-1}$ .
- b) **Oscillation - Flow point (strain ramp)** was measured by applying a logarithmically increasing oscillation force, strain. The interval was 0.01 to 1000%.

During the course of the project it was decided to conduct additional analyses of flow point to reach deeper understanding of the rheological behavior and properties of the greases.

- c) **Oscillation – Flow point (temperature ramp)** was measured by applying a constant increasing temperature, starting at  $-20$  and finishing at  $100^{\circ}\text{C}$ . The program was designed with an increase of  $3^{\circ}\text{C}/\text{min}$ , rendering in 480 measuring points. The strain was kept constant during the experiments on 0.3, 1.0 or 10.0%.

## 5.2 Grease cooking

The general configuration of grease is as earlier mentioned 85% base oil 10% thickener and 5% other ingredients, often different types of additives.

Based on this formulation four simple greases were produced to retrieve fundamental understanding on how each component affect the rheological behavior of grease. Lithium-based greases represent the largest share of the world market therefore this category was chosen as the starting point for development of a new standard method.

Four different base greases were produced:

- Pure lithium
- Lithium-complex
- Pure lithium + polymer (tackifier)
- Lithium-complex + polymer (tackifier)

The lithium grease named Lab 3819 was produced from:

- Heavy naphthenic distillate (base oil) 110mPa s at 40°C
- 12-hydroxy stearic acid -C18 (fatty acid)
- Lithium hydroxide monohydrate (counter ion to fatty acid)

The base oil was heated during stirring to about 90°C where the fatty acid was added whilst the temperature was kept constant until all fatty acid was melted and homogenized in the mixture. Then the lithium-slurry was added. After the saponification reaction had occurred the temperature was again increased to approximately 110°C to evaporate all the added water. The final step of the saponification was to increase the heat to 195°C at which the soap melted. When this point was reached the cooling process began by adding base oil and reducing the temperature until the grease was sufficiently cooled. The amount of added base oil and the cooling rate affects the consistency of the product. Therefore it is essential to keep track of added amounts to enable further addition of oil after the cooling process. This is done to reach the desired consistency before the soap content is calculated. This final step rendered a simple lithium grease and the consistency was checked by cone-penetration. The target NGLI-number was 2.0 but a portion of the produced grease was taken aside at NLGI 2.5 to later be mixed with polymer solution or other additives and thus reach the desired NGLI-number 2.0.

The lithium complex grease named Lab3820 was produced from:

- Heavy naphthenic distillate (base oil)
- 12-hydroxy stearic acid -C18 (fatty acid)
- Azelaic acid -C9 (dibasic acid)
- Lithium hydroxide monohydrate (counter ion to fatty acid)

The production of a complex soap is essentially the same as for a simple soap. The difference between the lithium and the lithium-complex grease is that for the complex two different fatty acids are used which requires two saponification steps. Just as with Lab 3819 a small amount of the

produced grease was taken aside to enable further production of grease with different additives. This grease was later used to produce Lab 3831.

Lithium grease with polymer named Lab 3830 was produced by adding a polymer solution containing high molecular Poly-iso-butylene (700000mPa s at 150°C) solved in mineral oil to Lab 3819 Sample 1 with NLGI-number 2.5. The amount of polymer solution was dependant on viscosity and to compare as equal samples as possible the targeted NLGI was the same for all greases (NLGI 2.0).

Lithium-complex grease with polymer named Lab 3831 was produced with the same technique as Lab 3830. The only difference was the grease to start with. In Lab 3831 the grease Lab 3820 sample 1 was diluted with polymer solution until the desired NLGI (2.0) was reached.

The cooking process was constantly monitored and the temperature and consistency was closely examined. A schematic temperature profile can be seen in figure 11.

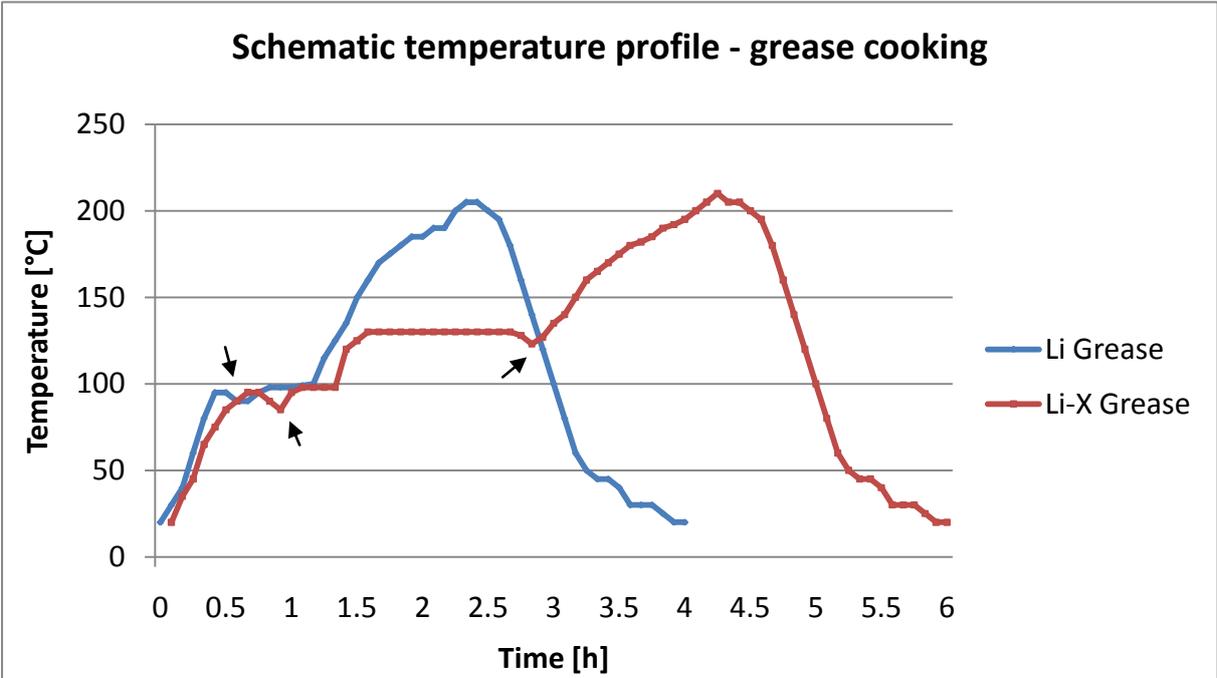


Figure 11 shows an example of how the temperature profile for grease cooking might look.

The arrows in picture 11 points to where the small temperature drops that indicate that the saponification step has started.

### 5.3 Nomenclature

The nomenclature used for the greases in this report is presented in Table 1.

Table 1. Constitution of greases mentioned in the report.

Name of sample	12-HSA	Azeleic acid	Polymer	Oil content [%]	Soap content [%]	NLGI	BOV [mPa*s]
Axcellence 152 EP	X	X	X	87	10.3	2	210
Axcellence 1525 EP	X	X	X	86	11.3	2.5	210
Li-X grease II	X	X	X	N/A	12	2	188
Lab 3819	X	-	-	86.2	13.8	2	110
Lab 3820	X	X	-	79.5	20.5	2	110
Lab 3830	X	-	X	89.6	10.4	2	573
Lab 3831	X	X	X	86.7	19.3	2	191
Lab 3687 A	-	-	X	85	15*	2	48
Lab 3693 A	-	-	X	86	14*	2	48

12-HSA= 12-hydroxy stearic acid

NLGI= national lubricating grease institute

BOV= base oil viscosity measured at 40°C

Soap content is calculated according to equation 1

Soap content = \_\_\_\_\_

Equation 1: calculating soap content

Since the only difference between Lab 3819 and Lab 3830 is that the sample is differently diluted to reach the desired consistency (the same goes for Lab 3820 and its counterpart Lab 3831). It was very interesting to see that the greases diluted with polymer solution needed less dilution to retrieve the same consistency. This in turn gave the polymer diluted samples higher soap content.

The nomenclature used for the polymeric oils in this report is presented in Table 2.

Table 2. Polymeric oils mentioned in the report. Viscosity is measured at 40°C

Component	Viscosity [mPa*s]	Content
Tack 09	5600	PIB/MO
Base 04	1350	PAO
Spectrasyn	10000	PAO

PIB= Poly-iso-butanol

PAO= Poly-alpha-olefin (high viscous synthetic oil)

MO= mineral oil (high viscous mineral oil)

## 5.4 Developing a standard method

To be able to compare different lubricating greases it is essential to designate a standard procedure.

Rheological measurements are common in many other trades. They are occasionally carried out within the lubricating grease society but could hardly be said to be standard procedures. When carried out, two of the most common ways to look at rheological behaviour of lubricating grease are rotational and oscillating tests. These methods are standard methods in other industries and many scientists within the lubricating business do use them occasionally for measurements on grease. Since these methods are at least somewhat known and acknowledged it was considered both favourable and reasonable to develop a standard method out of these.

There are today some common measurements for lubricating greases. As mentioned before the most common test for lubrication greases is the "Cone penetration" ASTM D217. This is as already argued not so scientific but gives information about consistency.

A more scientific approach in this area of interest would be to evaluate greases apparent viscosities. Apparent viscosity is measured with a rotational program on a rheometer and gives information about the sample's structure and viscosity. Such information provides necessary data that can be evaluated and used as engineering values when choosing the appropriate flow properties for a given application. By making this a standard procedure and labelling sold greases with the derived information the lubricating grease business would take a huge step towards a more scientific approach. This approach would naturally also render improved lubrication of equipment and greater knowledge for both producers and customers.

Apparent viscosity is a very powerful tool to understand and compare lubricating greases. Nevertheless it should not stand on its own. A logical way to complement the information obtained by the rotational test is to perform an oscillating test. When subduing a sample of lubricating grease to oscillating forces it is possible to gain information about the inherent flexibility in the material tested and to draw conclusions about pumpability. Pumpability is of great concern since many applications use centrally lubricated systems. Choosing the wrong lubricant for such a system may cause clogging and severe damage to the equipment.

## 6 Results

The results are presented in three main sections, divided by the different analytical methods presented earlier (i.e. apparent viscosity, flow point and temperature ramp).

### 6.1 Apparent viscosity

Looking at apparent viscosity the expected behaviour of grease was the same as for other fluid materials namely decreasing viscosity at increasing temperatures due to greater internal movements. This was found to be true even if greases could not really be stated as being pure fluids (the fluid is dispersed within a thickener lattice). This expected behaviour only applies to low temperatures; at higher temperatures a local minimum in the apparent viscosity was found. This local minimum was found in all tested lubricating greases. This local minimum could be described as a plateau in an otherwise smooth and constant slope. This plateau is best seen when plotting viscosity towards shear rate, figure12.

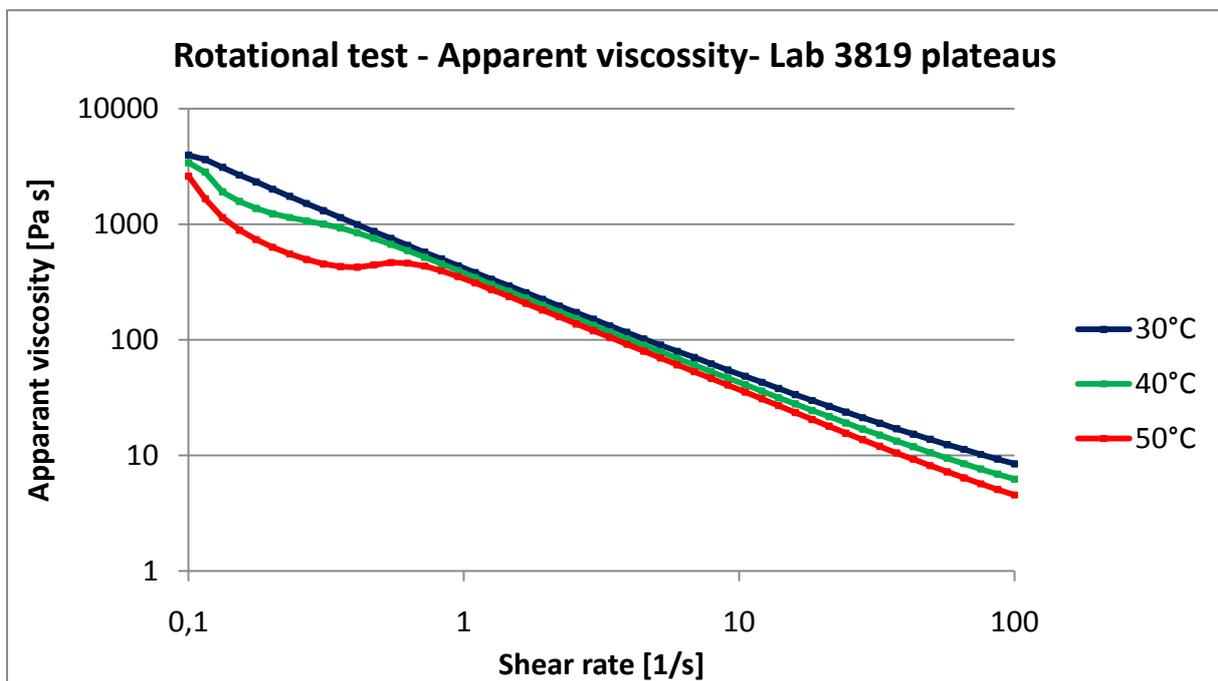


Figure 12 Plateaus are formed at higher temperatures. Here seen in simple lithium grease (lab 3819).

Figure 12 illustrates how the plateau builds up at higher temperatures. At 30°C no plateau exists but at 40°C the plateau is clearly seen. Increasing temperature gives more evident plateaus that are either prolonged or shifted towards higher shear rates (for full temperature series see appendix figure 29).

Since the apparent viscosity is measured in the region where this phenomenon occurs the values are often affected by this behaviour (see high temperatures in appendix figure 29, 30, 31,32, 33, 34) This behaviour has been found in all greases tested. To find out what caused this behaviour a number of additional tests were performed.

A possible explanation of this phenomenon could be that the thickener is insufficiently dispersed during production. To see if this was the case some greases were exposed to enhanced milling both during and after production. As seen in figure 13 this extra milling did not reduce the observed plateau.

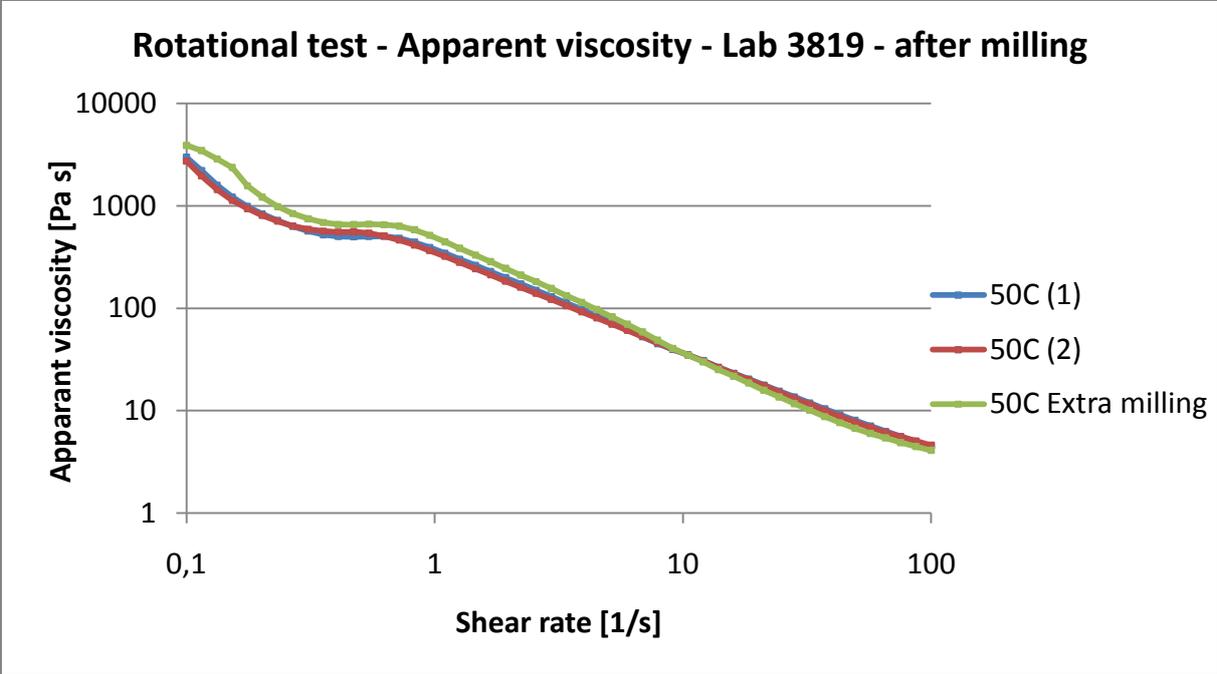


Figure13. Extra milling (green) gave a stiffer material but did not cure the plateaus. The reproducibility of the plateau is very good (red and blue).

The extra milling makes a small change in apparent viscosity but it did not reduce the plateau. In figure 13 it is also possible to see that the plateau is reproducible rather than accidental. Not only does it appear in all different greases tested but as seen in figure 13 (red and blue graphs) it is also very reproducible.

Another possible cause of this obstacle could be air in the sample. One sample was therefore subdued to extensive deaeration but figure 14 indicates that the deaerated sample possess the same plateau.

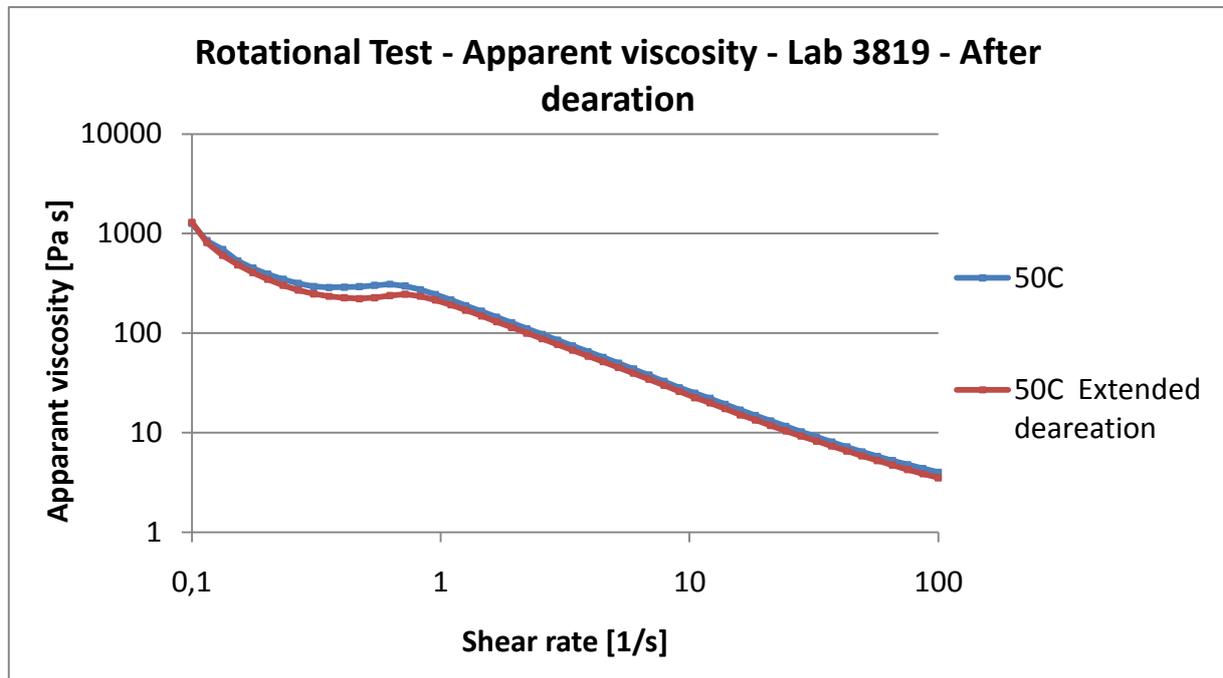


Figure 14. The deaerated sample shows similar but not identical behaviour. Even if there is a small change this change is not a reduction of the plateau.

To find out if this plateau appeared only in a certain thickener, greases with different types of thickeners were tested (metal soaps with and without polymer (tackifier), polymer (thickening agent) with and without elastomer and greases with particles as thickener) all of these greases showed similar plateaus even if they occurred at somewhat different temperatures. The first plateau normally occurred somewhere between 40 and 70°C and it got more significant or extended at higher temperatures.

Sometimes grease manufactured in full scale in the factory inhibits different properties than grease made in the small pots in the laboratory. To make sure that this was not the cause of the plateaus, tests were performed on a number of greases from both laboratory and full scale production. All of them formed resembling plateaus for similar greases.

To find out whether this behaviour could derive from entanglements a high molecular PIB (same as in the tested greases) dissolved in oil was tested and found to decreasing viscosity with increasing shear rate (see figure 15). This behaviour was fundamentally different from the one displayed in grease and occurred in the opposite temperature region.

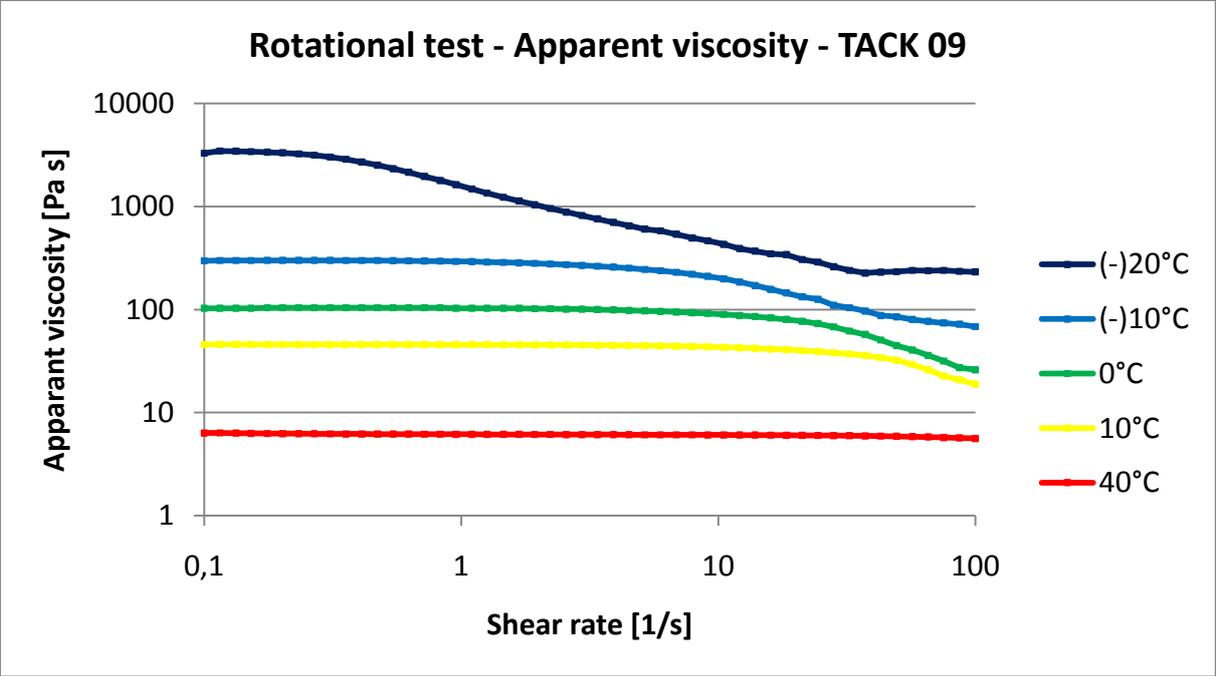


Figure 15. The polymer solution (TACK 09) exhibited a behaviour fundamentally different from the one seen in grease.

As opposed to the tested greases the polymer solution exhibited decreasing viscosity with decreasing temperature. Another difference is that these plateaus appear at much higher shear rates than for grease. The pour point of the oil in which the PIB was solved is approximately -12°C. This relatively high pour point explains the behaviour at low temperatures. Between -10°C and 10°C the temperature is above pour point but the structure is still brittle causing the otherwise straight lines to drop as the structure is broken down at higher shear rate. At higher temperatures where the plateaus were found for grease no plateaus are found for the sample TACK 09.

Since the polymer was dissolved in oil the same test was performed on pure oil with high viscosity, to enable application on test device, this oil did not display any plateaus, neither at high nor low temperatures.

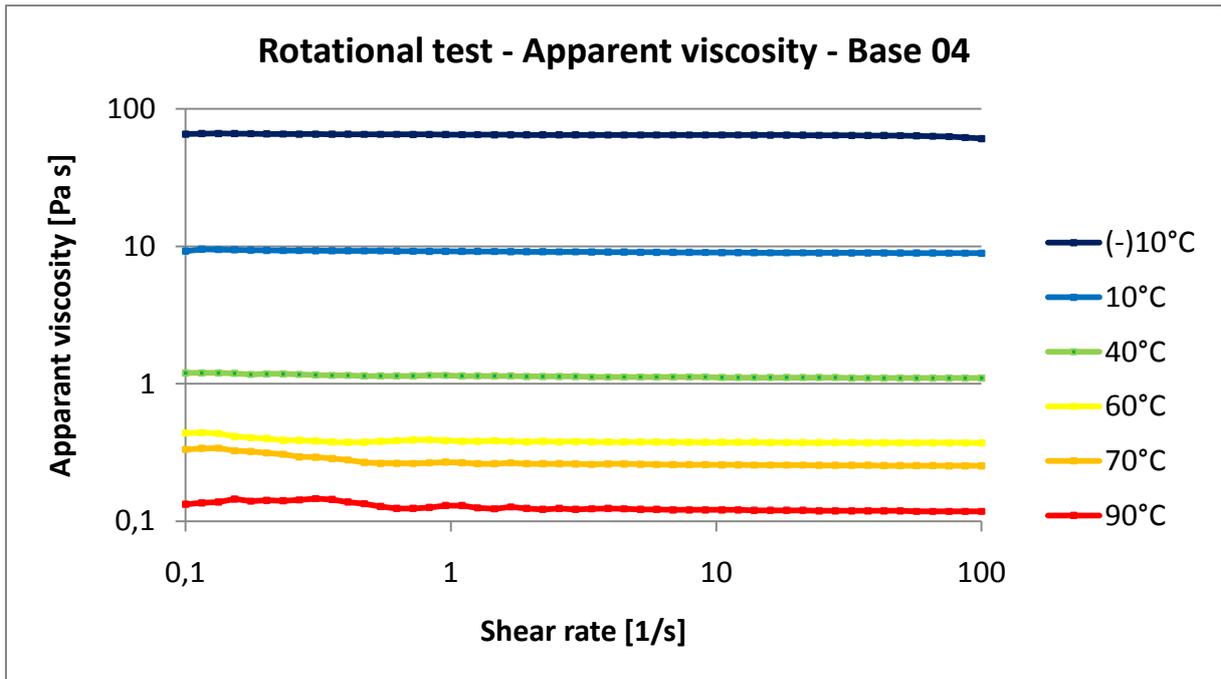


Figure 16. The lack of plateaus in Base 04 is obvious.

As seen in figure 16 neither temperature nor shear rate caused formation of plateaus in test sample base 04, a Poly-Alpha-Olefin(1350mPa s at 40°C). The fluctuation of the high temperature curves do not form plateaus nor decrease in viscosity.

To further investigate if this could still be due to entanglements or long structures, oil with even higher viscosity was measured. As seen in figure 17 Spectrasyn oil another PAO (10000mPa s at 40°C) did not display any plateaus.

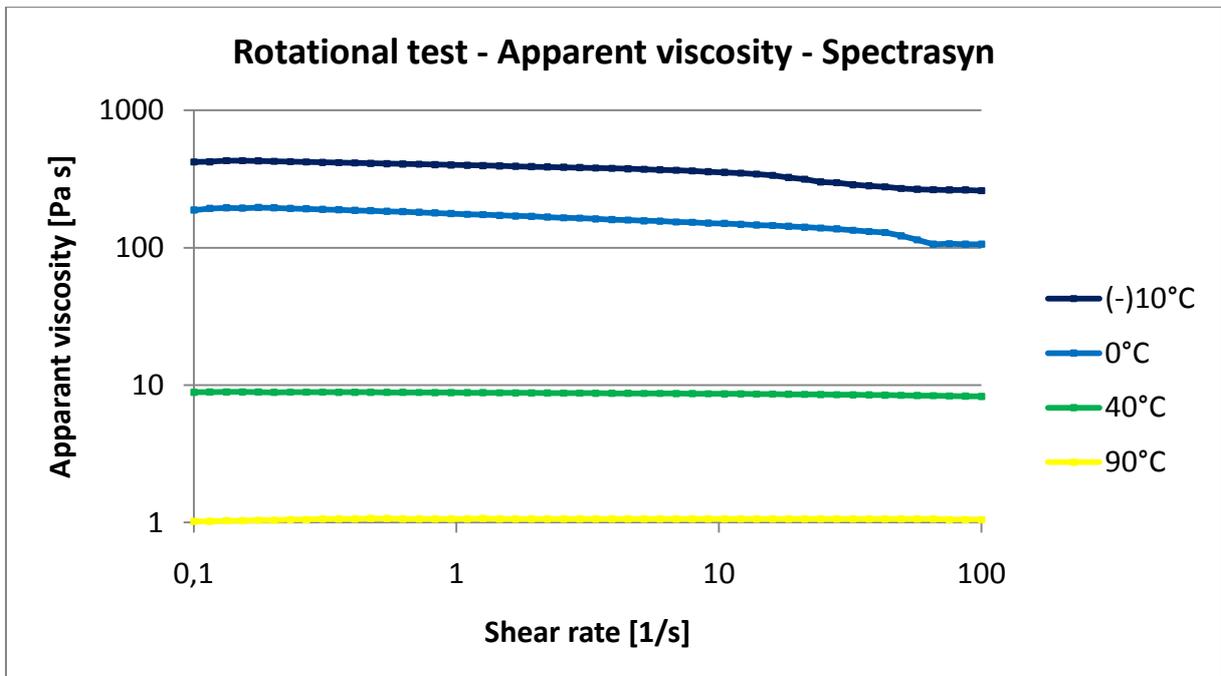


Figure 17 Spectrasyn oil did not form plateaus when subjected to rotational forces.

Perhaps this behaviour could depend on the multiple phases in grease where long bulky molecules interact with shorter or smaller molecules. To investigate this further silica powder was mixed in base oil. However this homemade gel structure became too brittle which made it difficult to run in the MCR and even more difficult to interpret. Instead a silica grease manufactured in the factory was tested. The result is displayed in figure 18 where the expected plateaus are seen. However the look of the plateaus and especially the temperatures where they occur were not the expected.

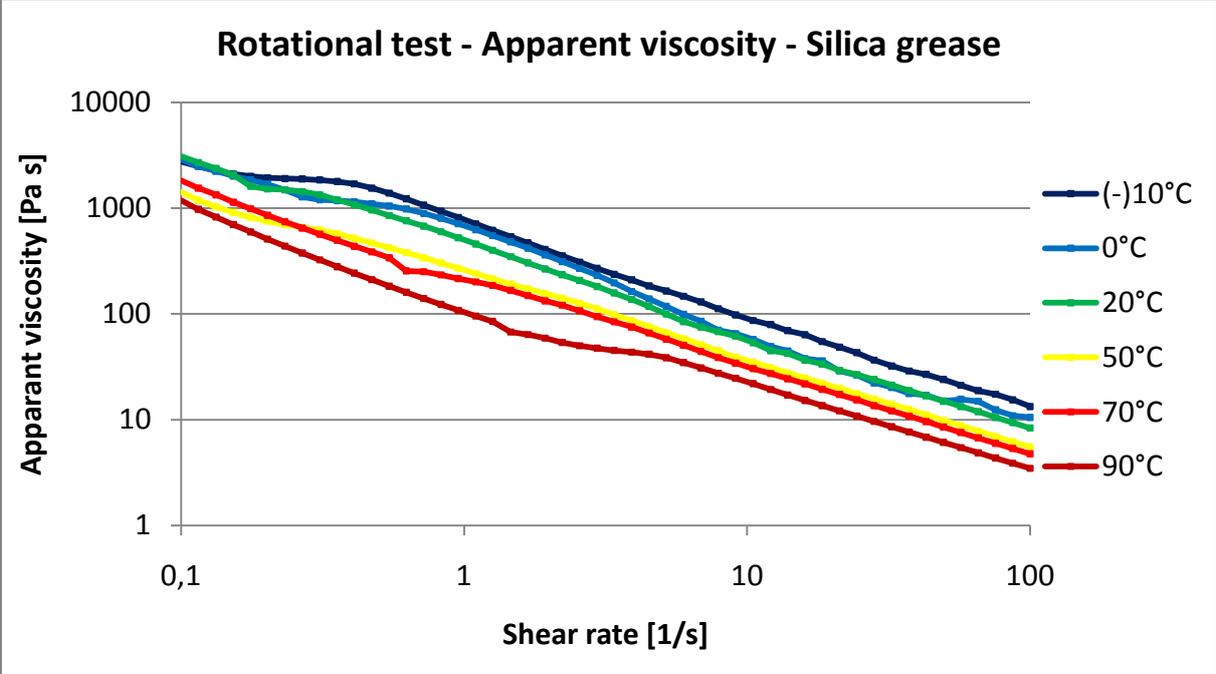


Figure 18. Silica gel forms plateaus at a wide range of temperatures.

To compare different greases with each other apparent viscosity at shear rate 1.0 was plotted towards temperature. In figure 19 it can be seen that the different greases appear in pairs. The pairs consist of the pure grease and the corresponding grease with an additive. In this case the additives are either polymer (3830, 3831) or elastomer (3693). As seen in figure 19 all six greases possess lower viscosity at higher temperatures. This is the expected behaviour for fluids and is apparently so also for lubricating greases, at least when the grease is subjected only to rotational forces.

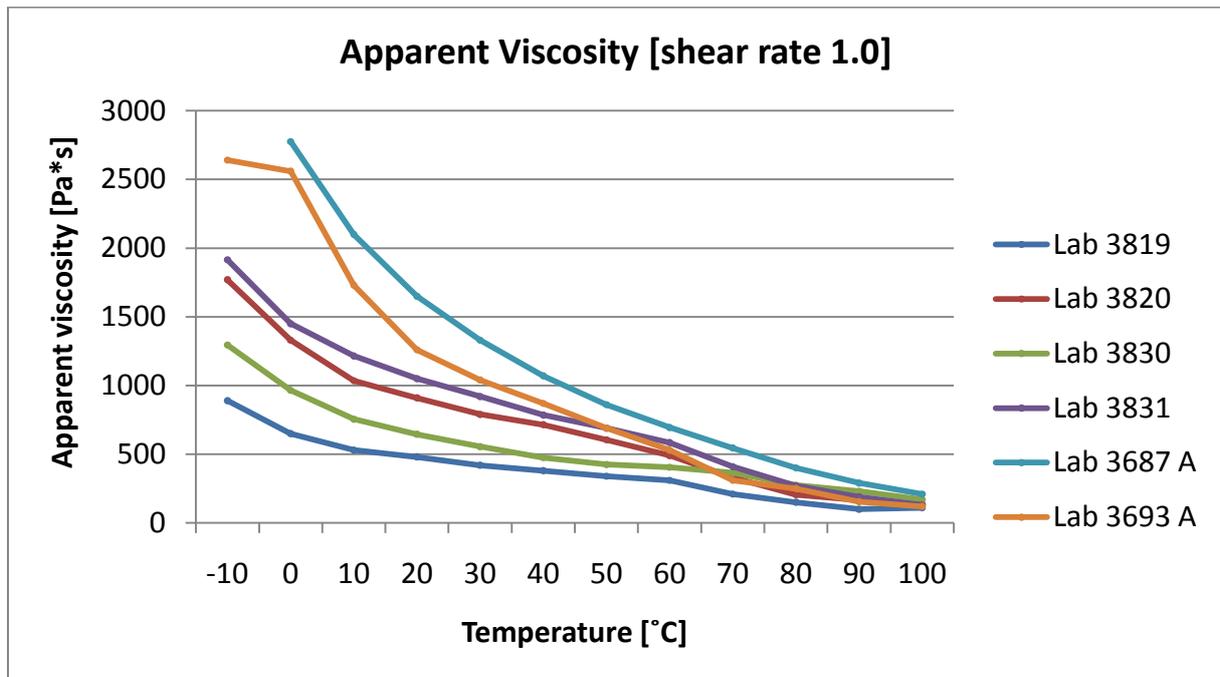


Figure 19. Three different simple greases together with their modified twins plotted towards each other.

## 6.2 Flow point

When analyzing flow point the expected behavior of grease is decreasing flow point with increasing temperature. This expectation is based on increasing internal movements within the grease structure with increasing temperature. The relaxation time of 300 s would provide thermal energy to the grease forcing the storage modulus (stored deformation energy) and the loss modulus (used deformation energy) to stabilize.

These assumptions were found to be true for some of the more complex greases (polymer thickened, Lab 3687 A and 3693 A) but not for all greases produced within the project. The four greases produced within this project displayed a minimum in the temperature region at 30 to 60°C. The flow point decreased at low temperatures in contrast to what was expected. The same assumptions were made for the amount of strain needed to reach flow point; increasing temperature would decrease the amount of strain. This expected behavior was, in correlation to flow point, found to be true for the complex greases but false for the simple greases. Instead of displaying a minimum, as was the case when looking at flow point, an optimum was reached in the same temperature region (30-60°C).

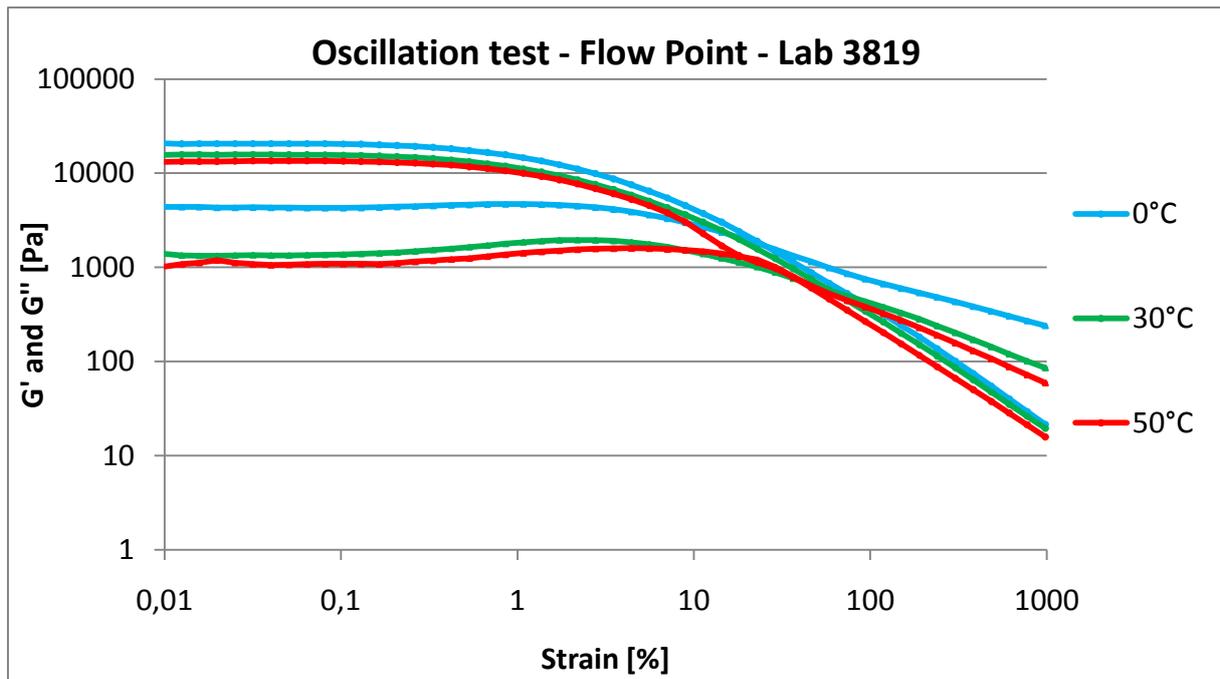


Figure 20. Oscillation test of Lab 3819 at 0°C, 30°C and 50°C

As figure 20 shows, the flow point of 0°C requires the highest amount of strain and force [Pa] to reach its flow point whilst 50°C demands less force and strain, this due to additional thermal energy stored at 50°C compared to 0°C. This was, as previously mentioned within logical behavior; however 30°C displays a deviating behavior. At that temperature the largest amount of strain was needed to reach flow point. It also occurs at a lower force compared to the other two temperatures.

This unexpected behavior was further investigated by running tests on lab 3820 to see if it had something to do with the complexity within the grease lattice. The same assumptions that were made for lab 3819 were also applied to lab 3820 but because of the more complex lattice structure built up by the thickener system, it would theoretically require more force and strain for the grease to reach its flow point.

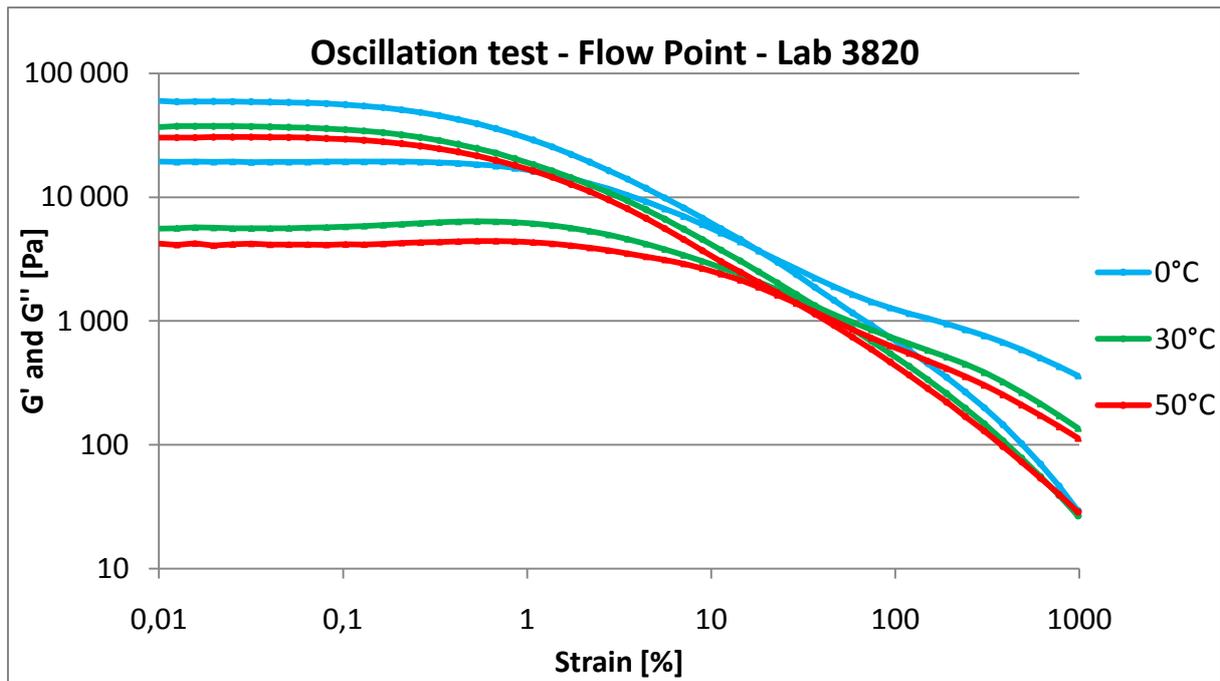


Figure 21. Displaying flow point of lab 3820 at 0°C, 30°C and 50°C

Figure 21 shows that the previous assumed prerequisite in terms of required force was found to be true. The flow points shifted along the y-axis for each temperature compared to lab 3819. This analyze indicated the same scenario, at lower temperature, the amount of force and amount of strain was decreasing as temperature was increasing. Nevertheless, lab 3820 showed the same behavior as lab 3819 with a flow point minimum displayed in the region 30 to 40°C. Since the amount of strain and flow point is interdependent it also rendered an optimum for strain at the same temperature.

To further investigate possible reasons for this strange behavior the two lubricating greases with added polymer, lab 3830 and lab 3831, were analyzed. It was found that adding polymer did not affect the behavior. The only noticeable difference between the pure greases (lab 3819 and 3820) and the ones with added polymer was that they kept their viscoelastic properties at a higher strain before they reached flow point (i.e. loss and storage modulus appeared at a greater distance from each other).

Since all of the flow point tests performed were solely conducted on mineral oil-based greases it was decided to run subsequent tests on polymer-thickened grease. This was done to see how the different lattice formed by polymers influenced the rheological behavior. The complete temperature series were run on lab 3687 A (with elastomer) and lab 3693 A (without elastomer). Flow point and strain data was collected and plotted together with the metal soap thickened greases in figure 22 and 23.

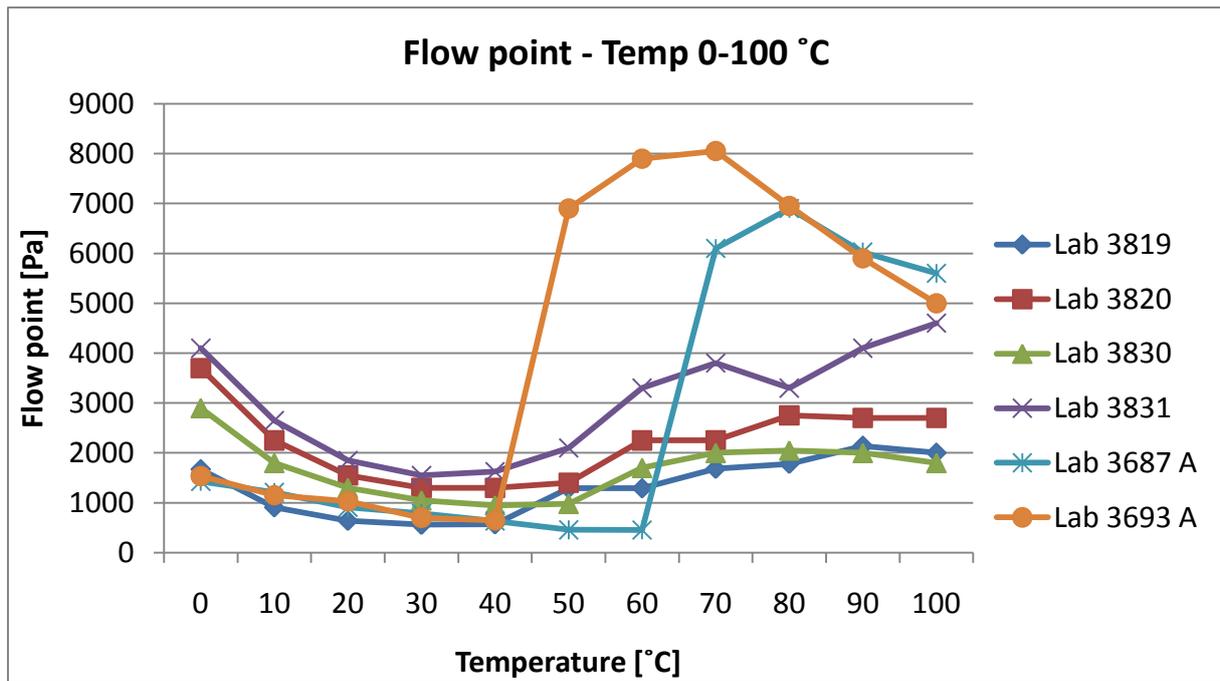


Figure 22. Comparing flow points of soap- and polymer thickened greases.

Figure 22 displays the flow point minimum for all the metal soap based greases. Also illustrated in the figure is that this minimum is increasing when the complexity of the thickener system (comparing lab 3819 with 3820) and additive increases (comparing lab 3820 with 3831). The added polymer also influences the amount of force required to reach flow point. When comparing lab 3819 and 3830 it is obvious that lab 3830 (with polymer) needs more force in almost every measuring point. The same scenario is displayed for lab 3820 and 3831 (with polymer), here every measuring point is even more distinguished from each other. It is clearly visualized that the amount of force to reach flow point is significantly increasing around 50°C for all the metal soap based greases.

Looking at the polymer thickened greases, lab 3687 A and 3693 A, They both show the same trend at low temperatures, decreasing amount of force to reach flow point. However, when lab 3693 A reached 50°C (corresponding temperature was 70°C for lab 3687 A) there was a very clear difference compared to the metal soap based greases. The amount of force increased rapidly at that temperature which shows that at high temperatures the difference between polymer thickened and metal soap thickened greases is more significant than at low temperatures. Before this significant increase both polymer thickened greases show a descending trend. This can be considered as in similarity with the metal soap based greases. The desired effect of adding elastomer is clearly seen in figure 22. Lab 3687 A (with elastomer) keeps its low flow point 20 degrees longer than lab 3693 A (without elastomer), which proves that adding elastomer actually effects the grease which has previously been proved by other testing methods.

Looking at the amount of strain needed to reach flow point for lab 3687 A and 3693 A it was according to the behavior expected, but not seen in the four greases produced within this project as figure 23 shows.

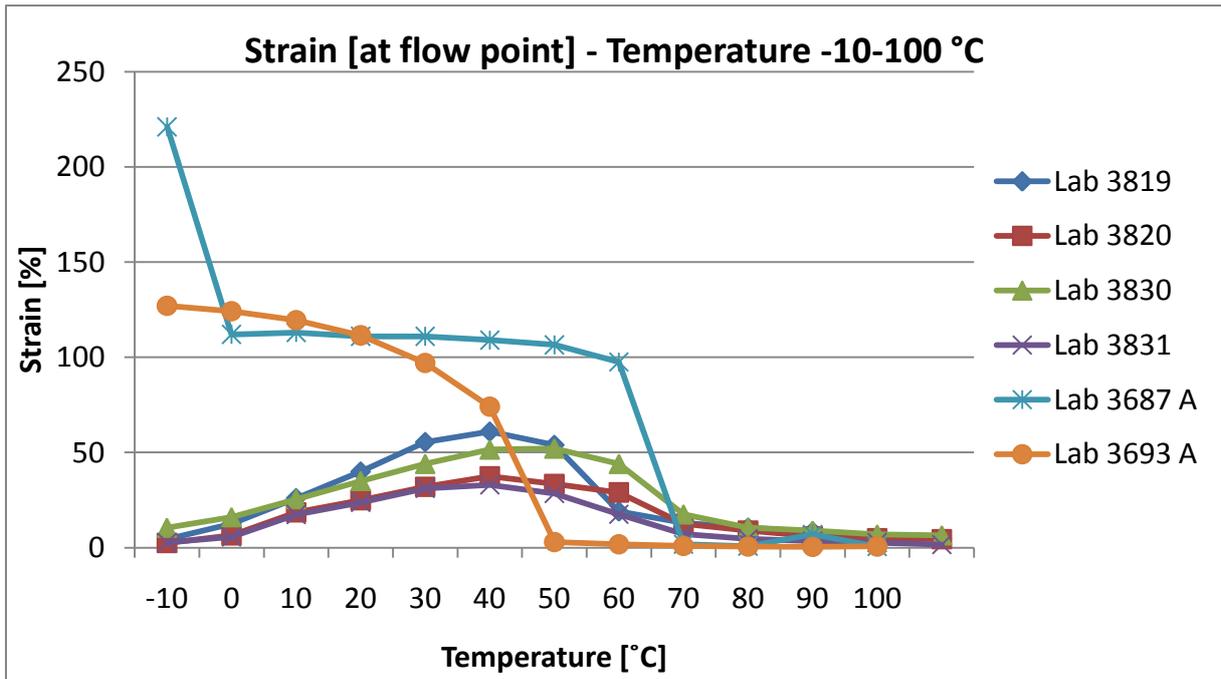


Figure 23. Displaying the amount of strain needed to reach flow point

When analyzing figure 23 above, the correlation between strain and flow point is prominent. As seen, lab 3687 possessed great mechanical stability in the temperature region 0 to 60°C. This is exactly the property it was designed to have. This behavior confirmed results seen in other tests, showing an increased mechanical stability of polymer thickened greases when elastomer is added. As for lab 3693 A, it possessed the behavior expected, but not seen in the four base greases. This was decreasing amount of strain with increasing temperature. Whilst for the metal soap based greases an optimum in the amount of strain was displayed. This optimum can easily be compared to the minimum displayed in figure 22. The figures show that the amount of strain and the flow point are interdependent for all the six greases.

Another observation that was found to be of high interest was the small peak of loss modulus shortly before the sample started to flow. This phenomenon was displayed when the amount of strain was big enough for the grease to lose its viscoelastic properties and the lattice structure started to deform. This is best illustrated by the red circle in figure 24 below.

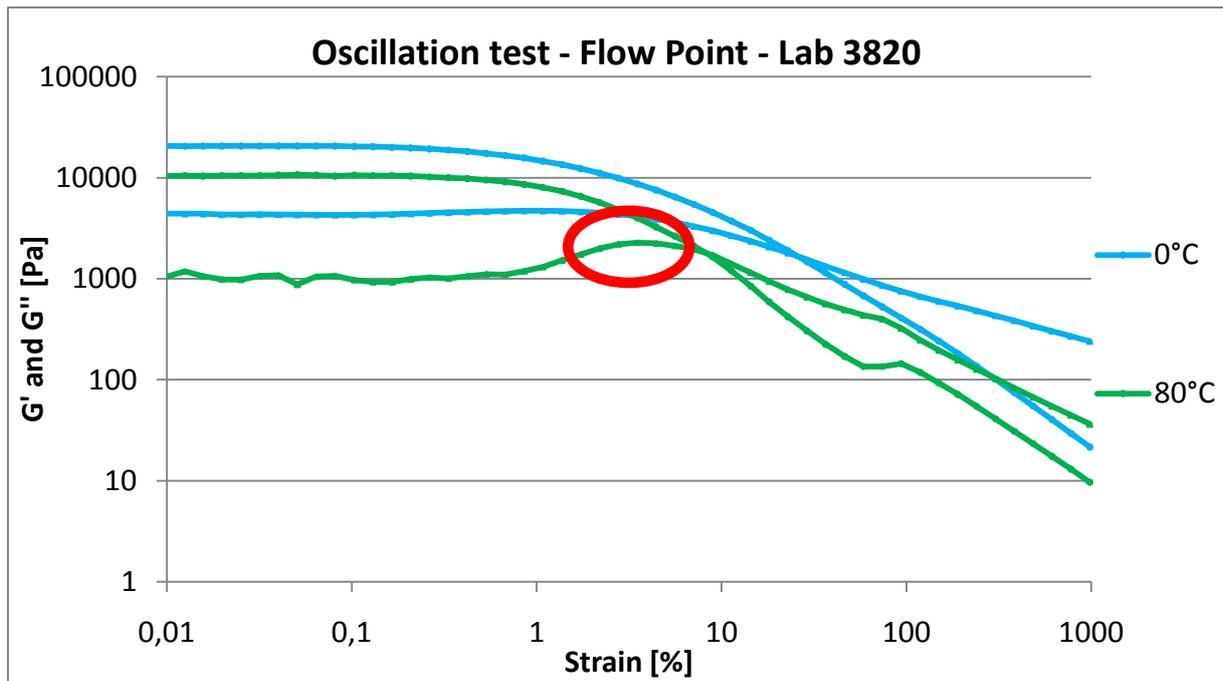


Figure 24. Illustrates the peak for loss modulus right after the deformation of the grease for

The peak displayed in figure 24 for lab 3820 is at 80°C and is clearly distinguish from 0°C, which displays no such behavior. This phenomenon was reproducible for all the greases incorporated in this project. The peak was exhibited slightly above ambient conditions, somewhere in the temperature region of 30 to 50°C.

To achieve deeper understanding of the unexpected behavior of the four base greases i.e. the flow point minimum, the strain optimum and the intriguing peak for loss modulus it was decided to conduct additional oscillating analysis. A new test, oscillating temperature ramp, was programmed (see section 5.1 c).

The intention was to find, at a specific strain, the most favorable temperature before the grease's lattice structure started to change. This is shown for lab 3820 in figure 25 below (for full series, including strain 10.0, see appendix figure 43-48).

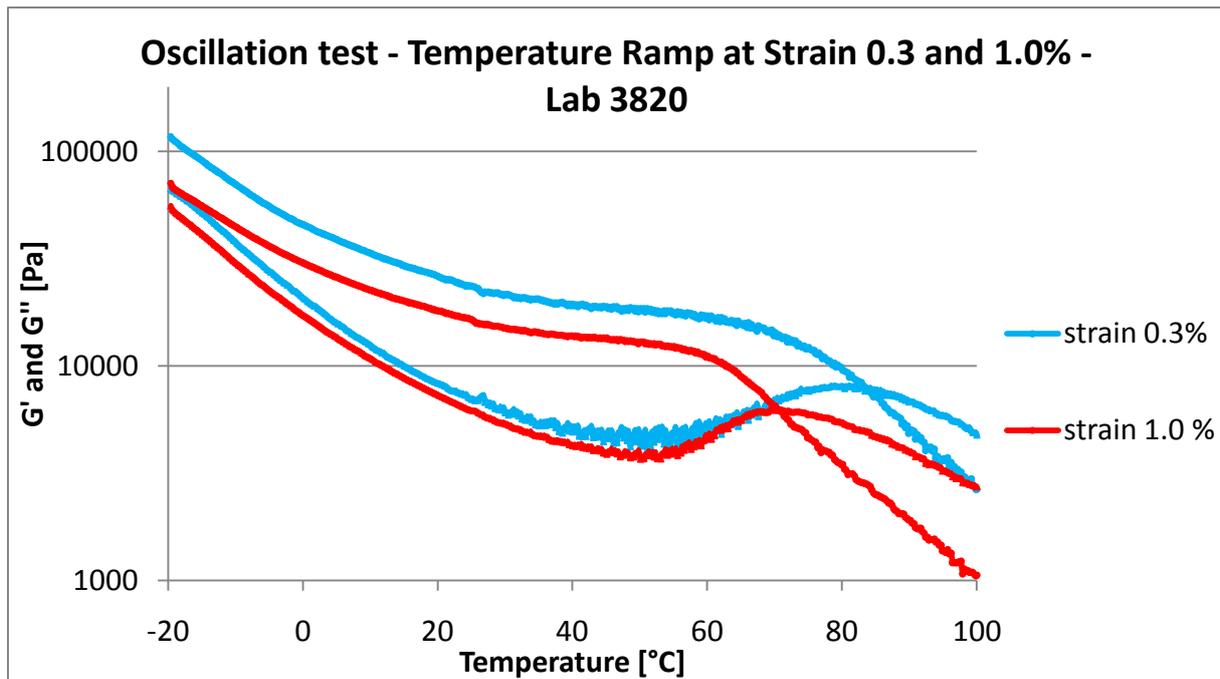


Figure 25.....

As shown in figure 25 flow point is reached within the temperature interval even for a small amount of strain (0.3 %) although it requires a high temperature before affecting the greases linear viscosity. It can also be seen that the irreversibility of the grease structure occurs gradually somewhere in the temperature region of 60°C to 80°C. This observation applies for all the four base greases. However, for the two polymer thickened greases the thermal energy and the lower strain 0.3% is insufficient to break the grease lattice. When the strain is increased to 1.0% flow point is reached within the temperature interval (see APPENDIX oscillation-tempramp lab 3687A & 3693 A).

### 6.3 Comparing greases

One of the intended benefits with having a standard method for evaluating lubricating greases was to facilitate an easy and reliable comparison between different greases. A real life situation when this comes in handy is when a lubricating grease manufacturer is asked to replace an existing lubricant from another company. The customers are normally not eager to change the present product if working properly. Economical or environmental reasons might make this change of product beneficial. A sound way of approaching such a change would be to re-evaluate which type of grease would be the best and try to tailor the grease perfectly for the application in question. However since the customers sometimes are hesitant to changing a working concept grease manufacturers might be asked to produce other greases with equivalent properties.

During the project grease from Axel Christiernsson was compared to grease from another manufacturer to evaluate whether one of Axels' existing greases could be used to replace grease previously bought from another grease manufacturer in this report called Li-X grease II.

The only thing known about Li-X grease II, was that it contained lithium complex and was labeled NLGI 2. It was reasonable to believe that the grease contained at least one additive since almost all modern greases contain one or more additives.

Looking at the well known and widely used NLGI method a wide variety of greases seemed feasible. However when consulting the more reliable data from the rheometer the differences were obvious. As seen in figure 26 the optimum, discussed in detail earlier, appears at a different temperature and has an altering appearance.

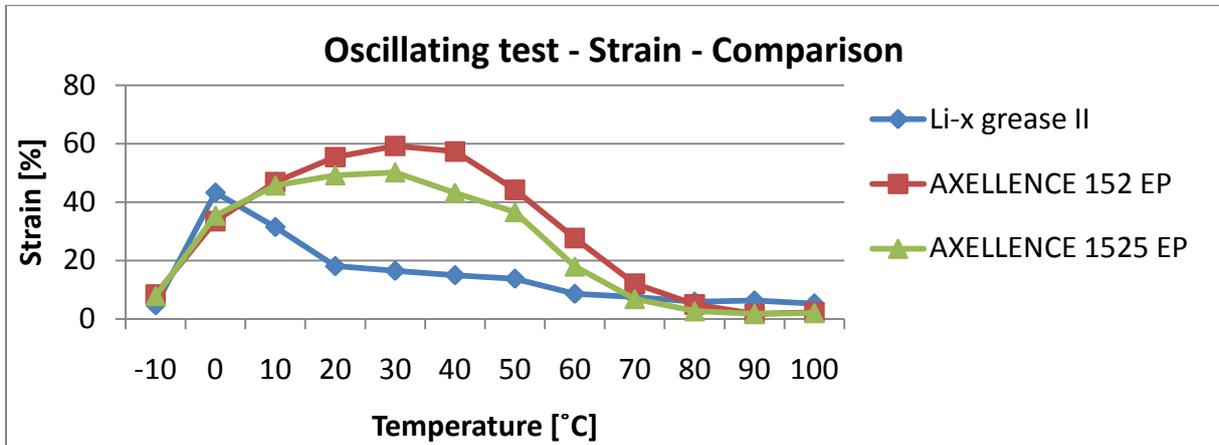


Figure 26. A different type of optimum appeared in Li-X than most of the greases produced by Axel CH here represented by two lithium complex greases.

This obvious difference does indicate that there are differences in the greases. However this difference does not indicate that the grease would not be suitable for the application. This is just one part of the complicated selection of which grease would be suitable for a specific application.

The other result from the oscillating test; flow point looked a bit more promising. As seen in figure 27. Axcellence 1525 EP exhibited if not identical, at least similar properties as Li-X grease II. The major difference is the peak at higher temperatures seen in figure 27.

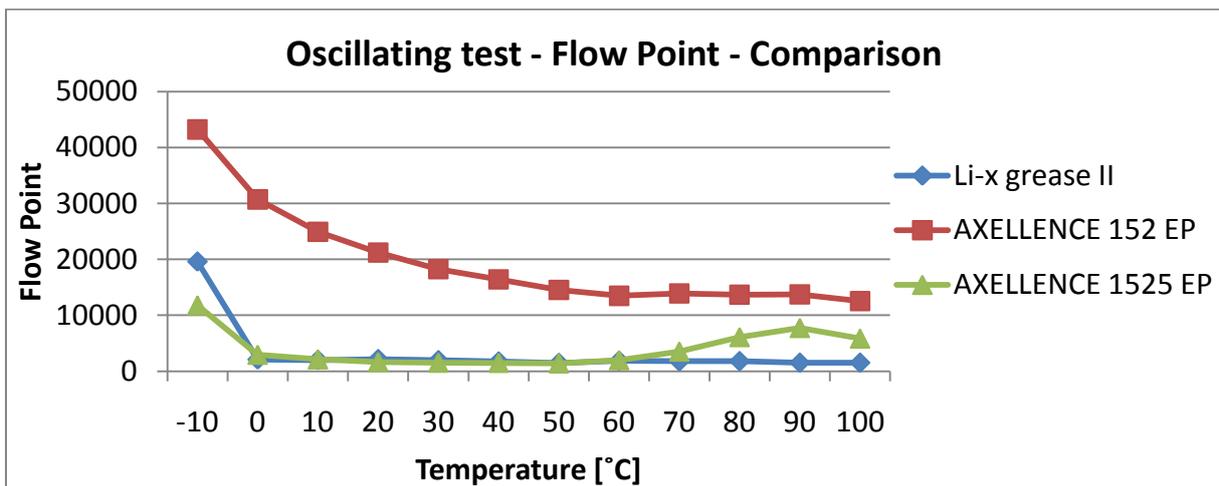


Figure 27. Looking at flow point it is evident that Axcellence 1525 EP is a better choice to replace Li-X grease II compared to Axcellence 152 EP. Also note the peak above 70°C for 1525 EP.

The Difference between Axcellence 152 EP and 1525 EP is really their consistency. 152 EP has NLGI 2 while 1525 EP has 2.5. As seen in figure 27 this consistency difference made the stiffer Axcellence look more similar to Li-X grease II. In fact Li-X grease II was analyzed with cone penetration which proved that it was stiff as a 2.5 (NLGI) even though it was labeled 2.0.

Comparing the same three greases with the rotational method it was possible to say that for each measuring point all three greases were rather close to each other. In a larger perspective though, it is evident that Li-X grease II displayed a smoother and more logarithmically looking curve see figure 28.

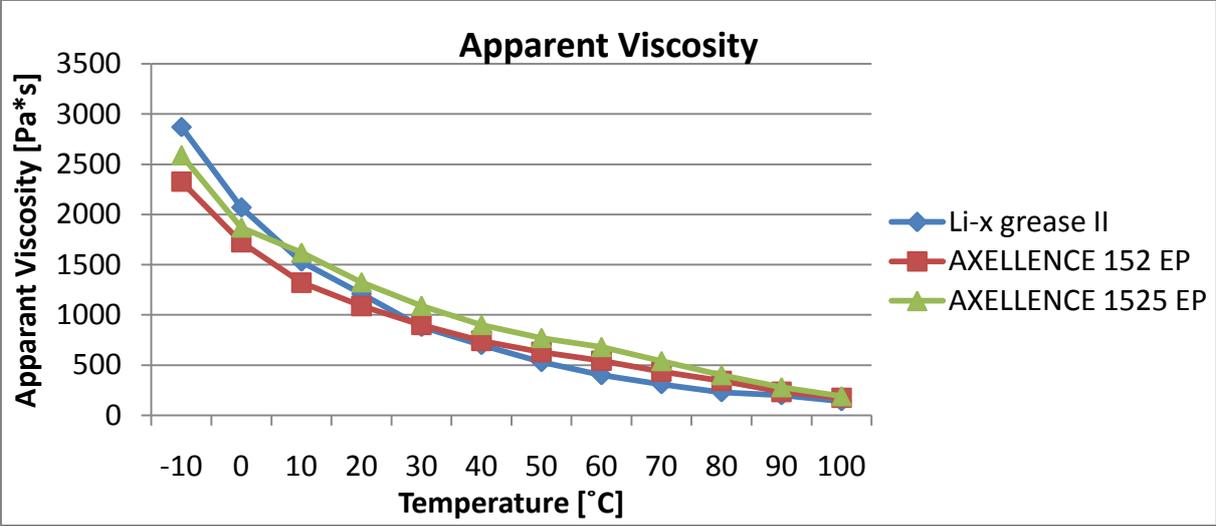


Figure 28 Li-X grease II shows a smoother curve than the two Axcellence's.

In general this very simple comparison of rather similar greases indicates that different greases even similar ones are clearly distinguishable from each other and exhibits measurable and comparable rheological properties. To be able to substitute one grease for another, expertise and experience is still needed but are in this way backed up by reliable data.

## 7 Discussion

The reproducibility was easy to test and the results were positive. It is possible to repeat both the expected straight slopes and the unexpected so-called plateau behavior for apparent viscosity. The same goes for the peak, the optimum and the minimum that occurred when measuring flow point. As long as the grease is not subjected to severe mechanical force before it is analyzed, the relaxation time of 300 s is sufficient to compensate for any structural degradation.

### 7.1 Apparent viscosity

The results from the rotational test were not the anticipated. The encountered plateau is problematic since it occurs at shear rate 1.0% where data is normally collected and compared. If data is retrieved at different shear rates for different greases it would not be such a standardized test. Moreover it would be, if not impossible, at least extremely difficult to compare different lubricants. When this plateau behavior was found it was considered interesting since it was an unexpected behavior.

A number of different greases were carefully studied to test whether the same behavior occurred in all of these. Since the result of this study was positive a simplified test with fewer temperatures was performed on a larger number of greases. The greases studied were chosen based on which components they contained and what properties they had to find an explanation to the encountered plateaus. This rendered in a wide range of greases from different subgroups containing different thickener systems including soap, gel, particle and polymer thickened greases. All these greases generated some kind of a plateau at higher temperatures. Seeing that the plateaus existed in such a variety of greases made the task of understanding this behavior more difficult. Since the behavior was seen in greases both with and without a soap thickener the soap thickener alone could not cause the formation of plateaus. If the plateau derives from the thickener system it does not matter what kind of thickener it is more likely an effect of the thickeners mobility in the base oil. This indicates that all thickener system have something in common. This common ground is probably on a molecular level. Perhaps it includes molecular interactions within the thickener structure.

On the other hand these molecular interactions could just as well take place outside the thickener system or with the thickener system together with the base oil. Since the interesting plateau was found even in greases without additives the root of it needs to be found in either the base oil, the thickener or the combination of these two. To see if the behavior appeared without thickener, high viscosity base oils were tested. Pure base oils did not form plateaus. This was rather expected since rheological measurements are more common in the lubricating oil business than in the corresponding grease business. No indications of interfering plateaus have been found in previous studies. Thus the result indicates that it is the thickener system alone or in combination with the oil that causes this peculiar phenomenon.

When looking at the combination of thickener and oil, these systems have very different character. Systems where the thickener is built by a metal soap is fundamentally different than the ones built by particles, which in turn are just as different from the ones built by polymers. Silica and clay thickened grease both display clear plateaus.

Polymer solved in base oil showed a different behavior at low temperatures. This behavior appears like a drop of apparent viscosity at high shear rates and low temperatures and is thus not considered as a plateau. It could however theoretically have the same roots as the plateaus at high temperatures but more likely it is due to freezing and brittleness caused by the low temperatures.

## 7.2 Flow point

The extensive analyses of flow point and its interconnected amount of strain rendered in quite unanticipated results. From the results it was clearly shown that all the four base greases had a minimum flow point between 30 to 60°C. It indicates a low temperature effect before it reached the minimum and a high temperature effect after that. What can be interpreted from the flow point measurements is that there is a distinct difference between almost every measurement point of the metal soap thickened greases. They contain different components and have different soap content but are produced in a similar way.

One aspect that should be taken into account, at low temperatures, is the adhesion of the greases to the bottom plate and probe of the rheometer. During low temperature conditions there might be some components within the greases lattice that freezes. This could cause the grease to become more brittle which in turn can make the grease more prone to break and reach its flow point at an earlier stage.

At higher temperatures flow point and the interdependent amount of strain display a minimum and optimum respectively. This could possibly be thickener-related. Previous studies has been conducted that showed different rheological behaviors depending on which type of thickener used [11]. This study also indicated that at low frequencies (low strain) and high temperatures the behavior of greases is highly entangled and structured. This could be a possible cause of the high temperature effect displayed for the metal soap based greases within this project.

The expected behavior was that increased thermal energy would generate more molecular movement and the greases would be more prone to reach flow point. Instead the greases behavior seemed to exhibit a more entangled system with intermolecular interactions which caused the amount of force to increase with increasing temperature.

Another aspect that should be taken into consideration is the loss of sample from the rheometers contact area. As mentioned before, when temperature increase more intermolecular movements appears in the greases structure. This enhanced movement caused the greases to be less viscous and therefore more prone to slip, reducing the amount of sample on the plate-to-plate geometry.

Another conducted study shows how greases are dependent on their base oil formulation. It displays how the different paraffinic and naphthenic components in the base oil are affected by different parameters (e.g. oxidation). This affects the fibers and the structural breakdown of the thickener system[6].

Looking at the results of the measurements with a temperature ramp supported the method used for measuring flow point. When more strain was applied the greases reached their flow point at a lower temperature. Again there is a clear difference between metal soap and polymer thickened greases. The polymer thickened greases are unable to reach flow point at low strain (0.3%) whilst applying the same strain to metal soap thickened grease usually causes it to flow. Applying high strain (10%) to

the metal soap thickened greases caused an almost immediate altering of the structure and flow point was reached already at temperatures below 0°C (see appendix figure 43 and 44). This proves that greater stress causes a greater effect on the grease thickener structure. However, loss modulus has less effect on the base oil which in turn will have less effect on the viscous properties of the greases.

### **7.3 Comparing greases**

The differences in strain when comparing Axellence and Li-X grease II is not so surprising since they are made in different factories and most probably with different methods. However it is quite surprising that most of the greases made at Axel Christiernsson have a similar behavior.

It was found that the grease stiffness influenced the flow point; this is not so surprising since the consistency is partly dependent on flow point and partly on apparent viscosity.

The peak at higher temperatures is interesting and could at least for some applications prove to be problematic since this might affect pumpability even at high temperatures.

The optimum displayed at temperatures just above ambient conditions could if yet less problematic still be considered as very interesting (see earlier discussions). The most interesting seen in this comparison is that Li-X grease II did show the expected behavior at least above 0°C. The only other grease encountered with this logical behavior was the polymer thickened greases (see section 6.2, figure 22 and 23). It is thus evident that the same thickener system can give different rheological responses.

## 8 Conclusion

Some of grease rheological properties can easily be analyzed by conducting the measurements suggested within this project. These tests constitute an excellent contribution to other standard methods. The results show differences both in terms of flow point and apparent viscosity for all analyzed greases. Greases produced at different production plants displayed greater differences. This indicates that the final grease product is significantly affected by a number of different parameters in the production. There are many parameters that can be altered to change the properties and rheological behavior of the greases. According to our study there are two that seem to be responsible for the greatest effect on grease:

- Process method
  - This includes temperature, addition rate of components, milling and deaeration. The final product is strongly dependent on, at which temperatures the components are added and how long the homogenization processes are. This together with rate addition rate of components can change how the grease is built up and how the thickener lattice is formed.
  - The largest difference between the rheological behaviors is seen when comparing Axcellence with Li-X grease II. These are both lithium complex soaps but made in different factories and probably with different base oils and fatty acids.
  
- Thickener
  - The thickener lattice is the most important component in lubricating greases. If created incorrectly it will most certainly affect the lubrication and the resistance against water spray-off. All these parameters are affected by the previous mentioned process method.

When the combined results were analyzed it seems like the most probable cause of the local minimum, for apparent viscosity as well as flow point, is to be found in the thickener system.

The results of this study constitute a starting point for extended studies of how lubricating greases can be understood, compared and labeled. To develop a standard method, further studies need to be conducted to comprehend how each component affects its rheological behavior.

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## **Appendix 1 Rotational test**

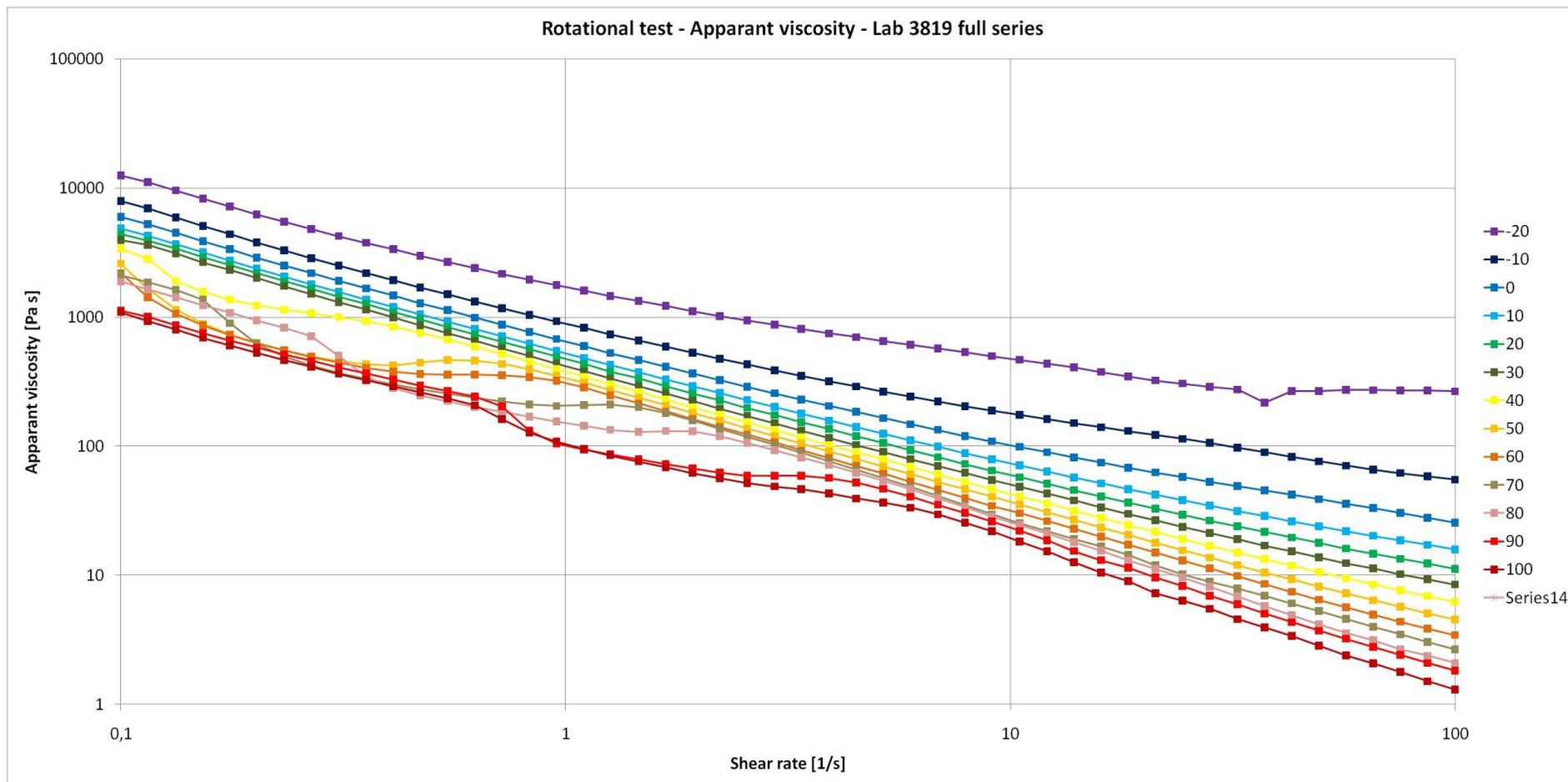


Figure 29 Lab 3819 full series

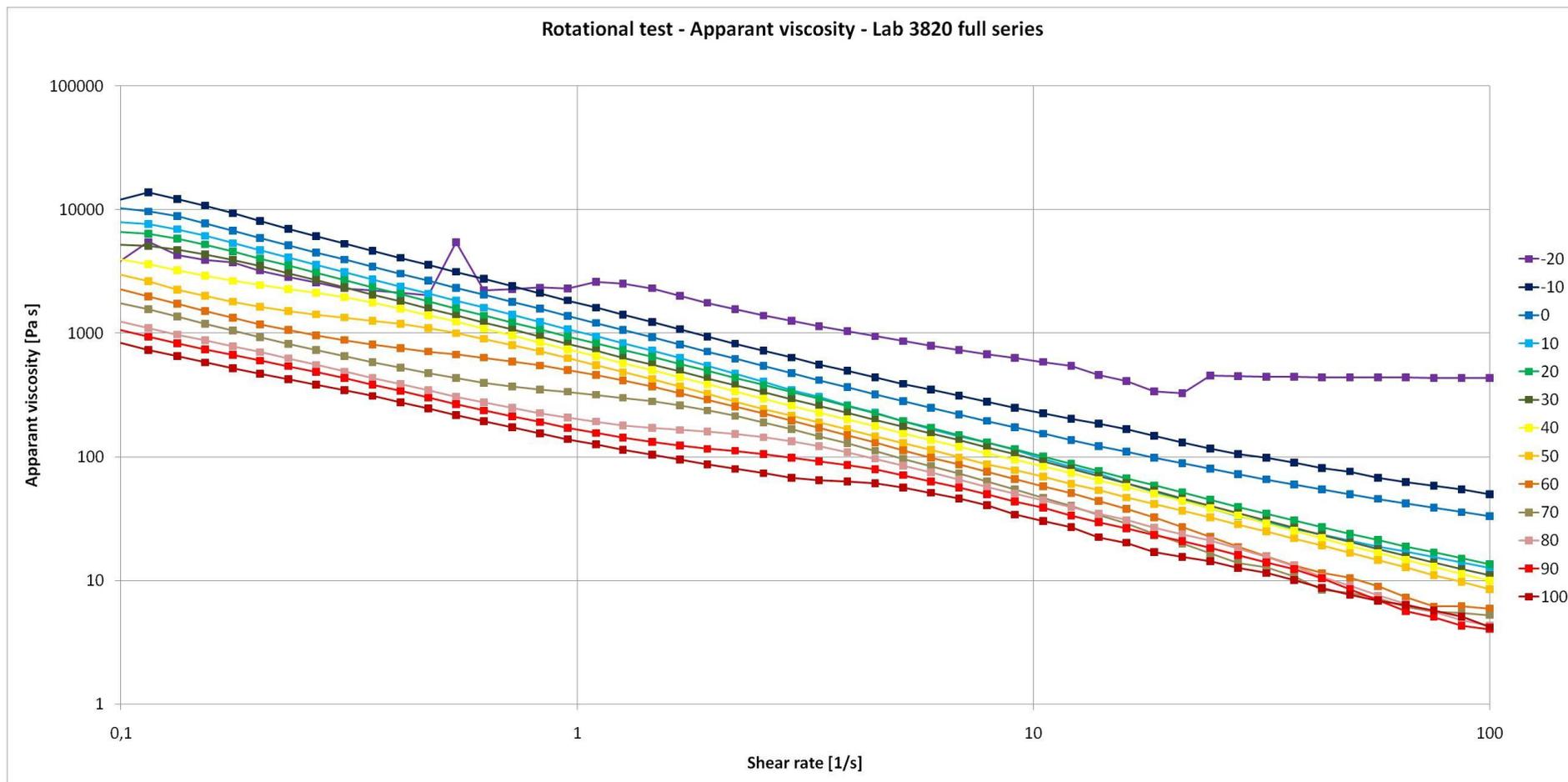


Figure 30 Lab 3820 full series

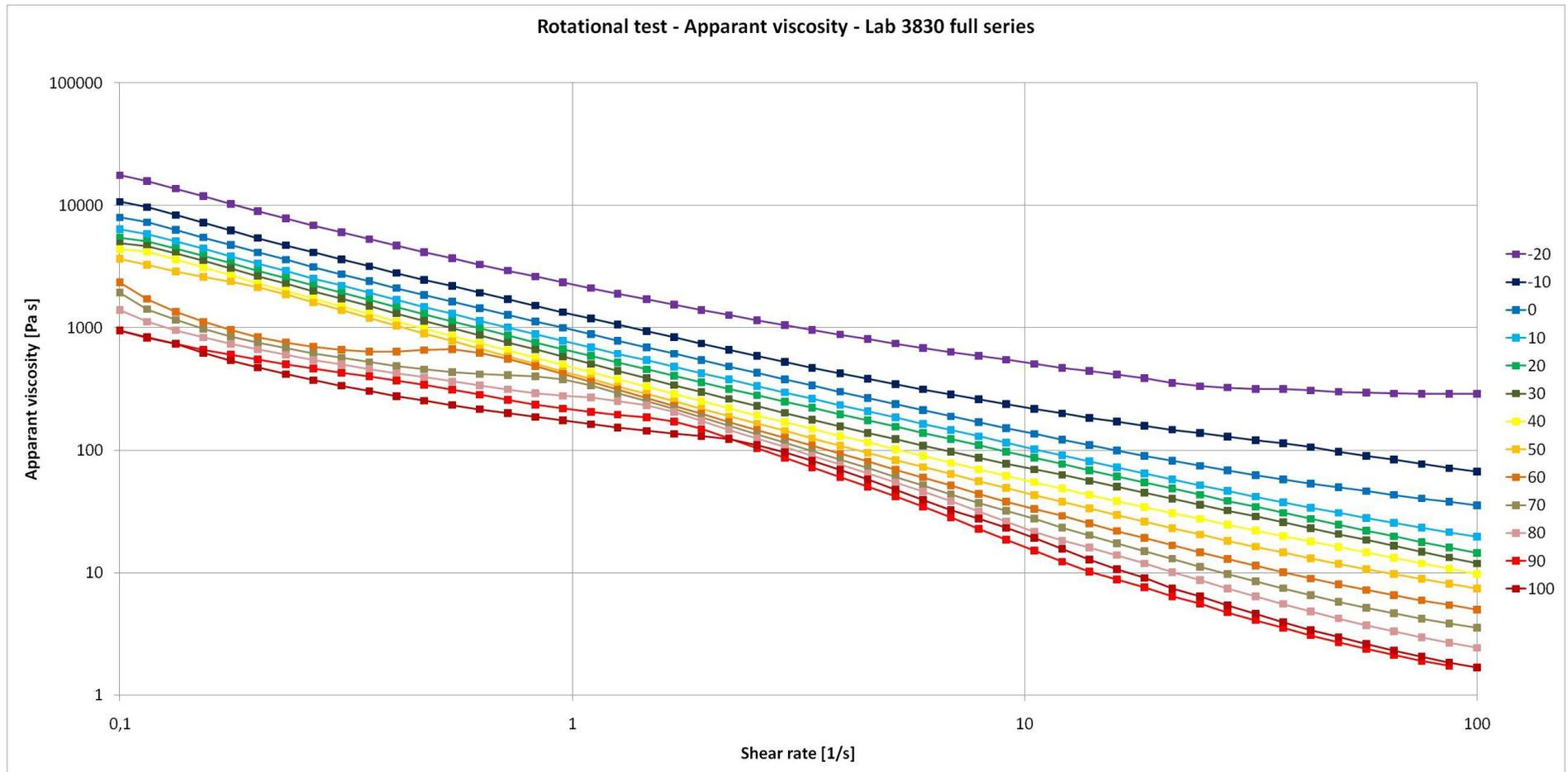


Figure 31 Lab 3830 full series

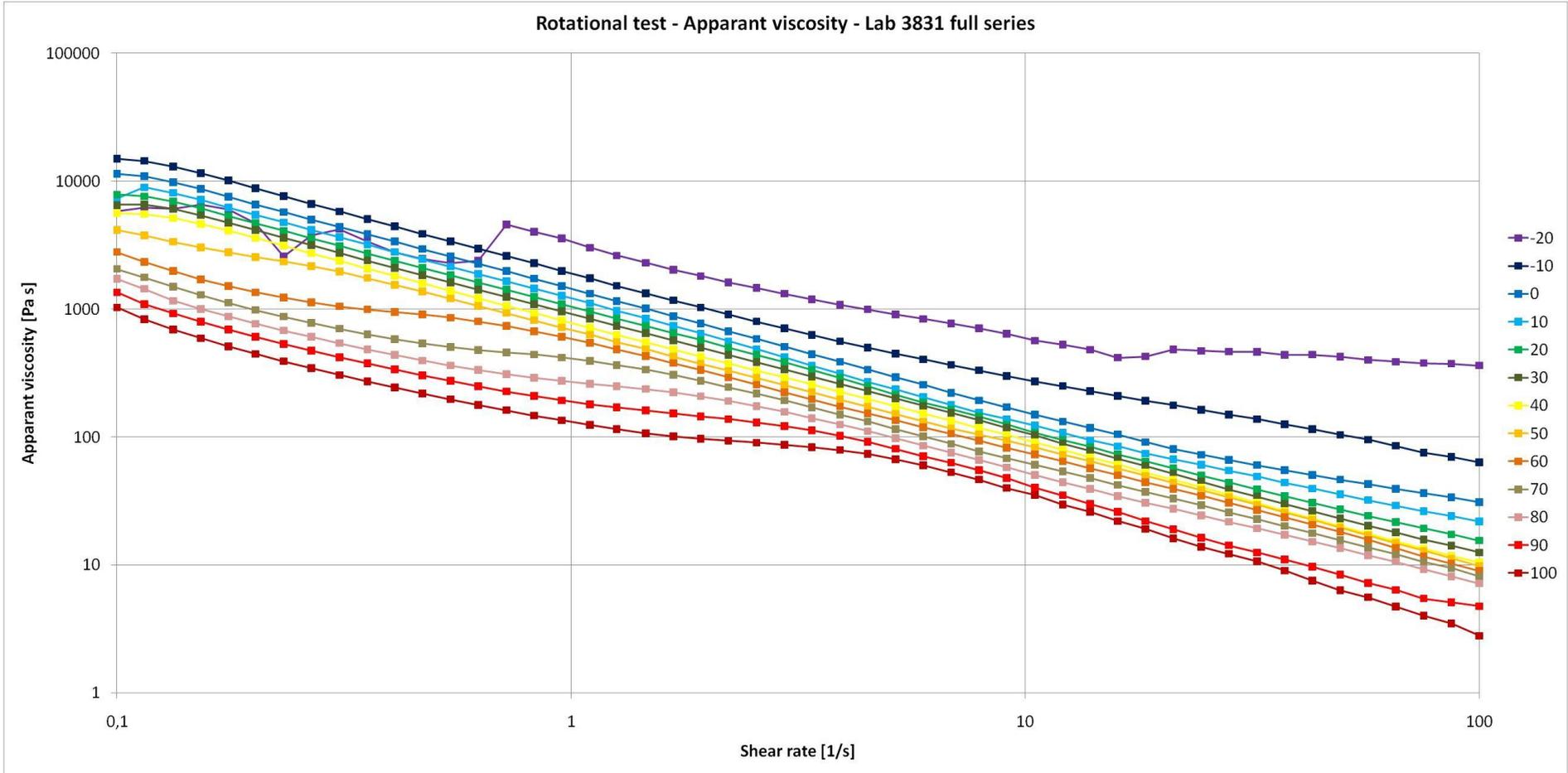


Figure 32 Lab 3831 full series

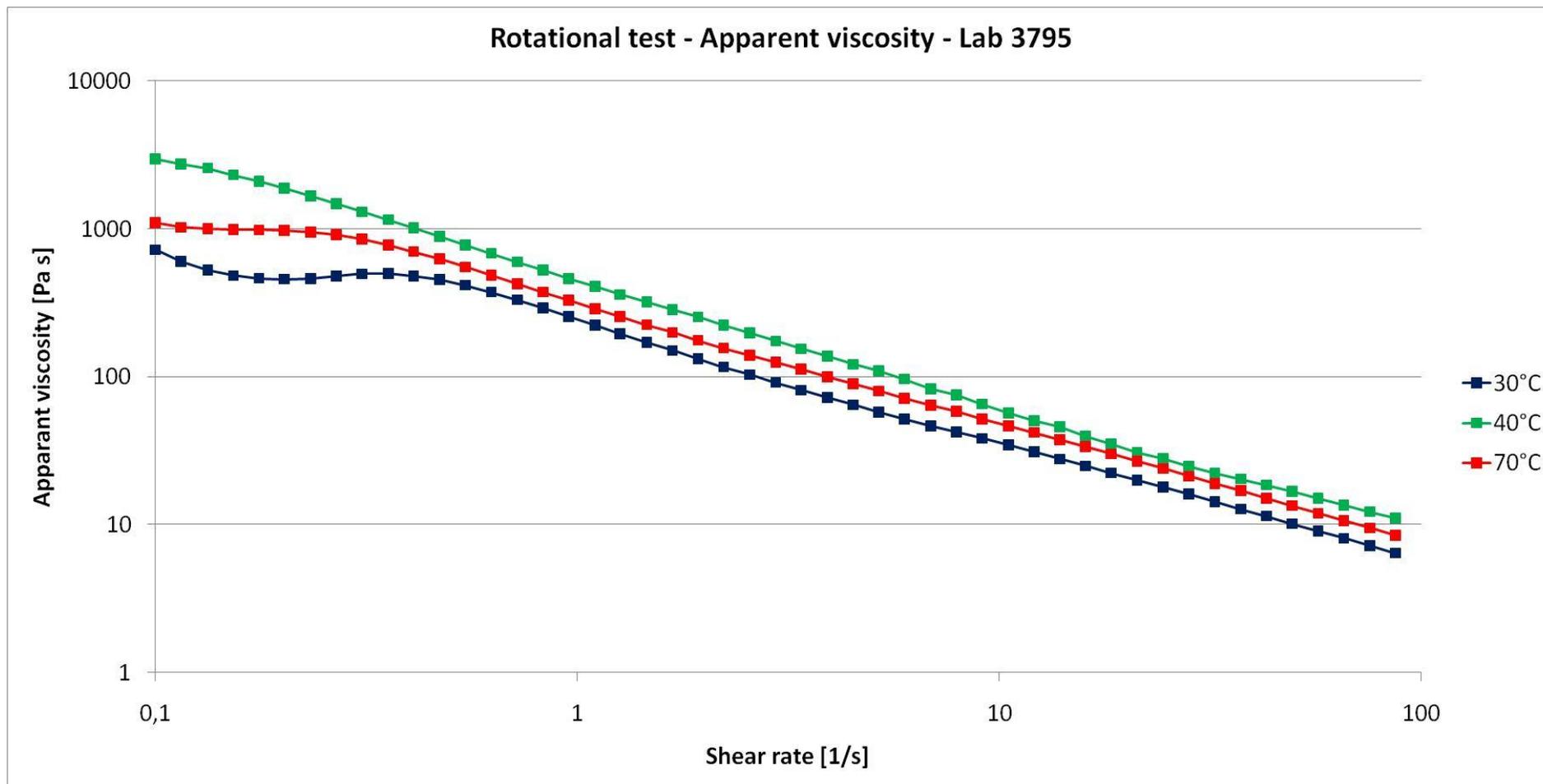


Figure 33 Lab 3795 plateau

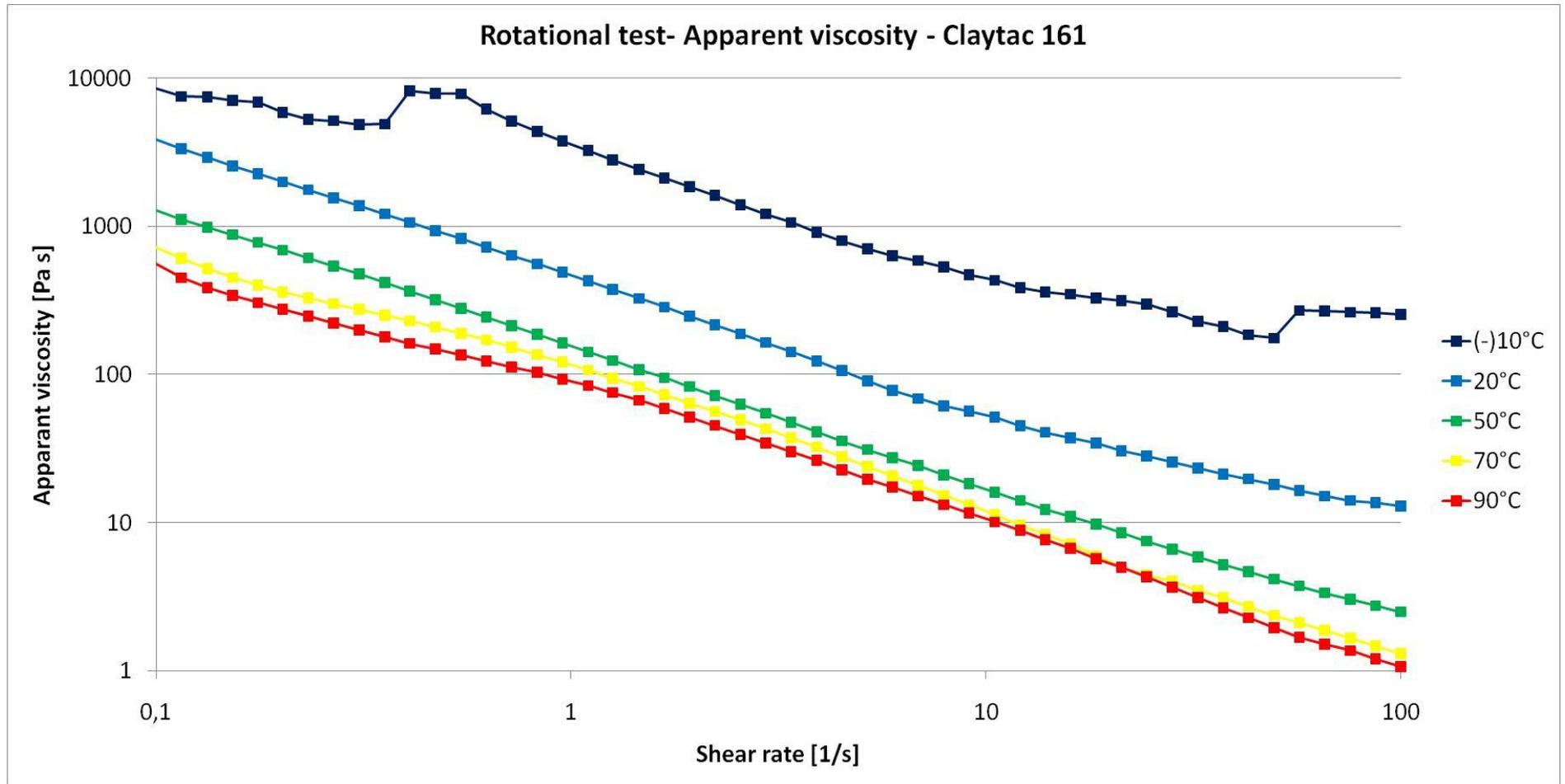


Figure 33 Lab 3795 local minimum claytac 161

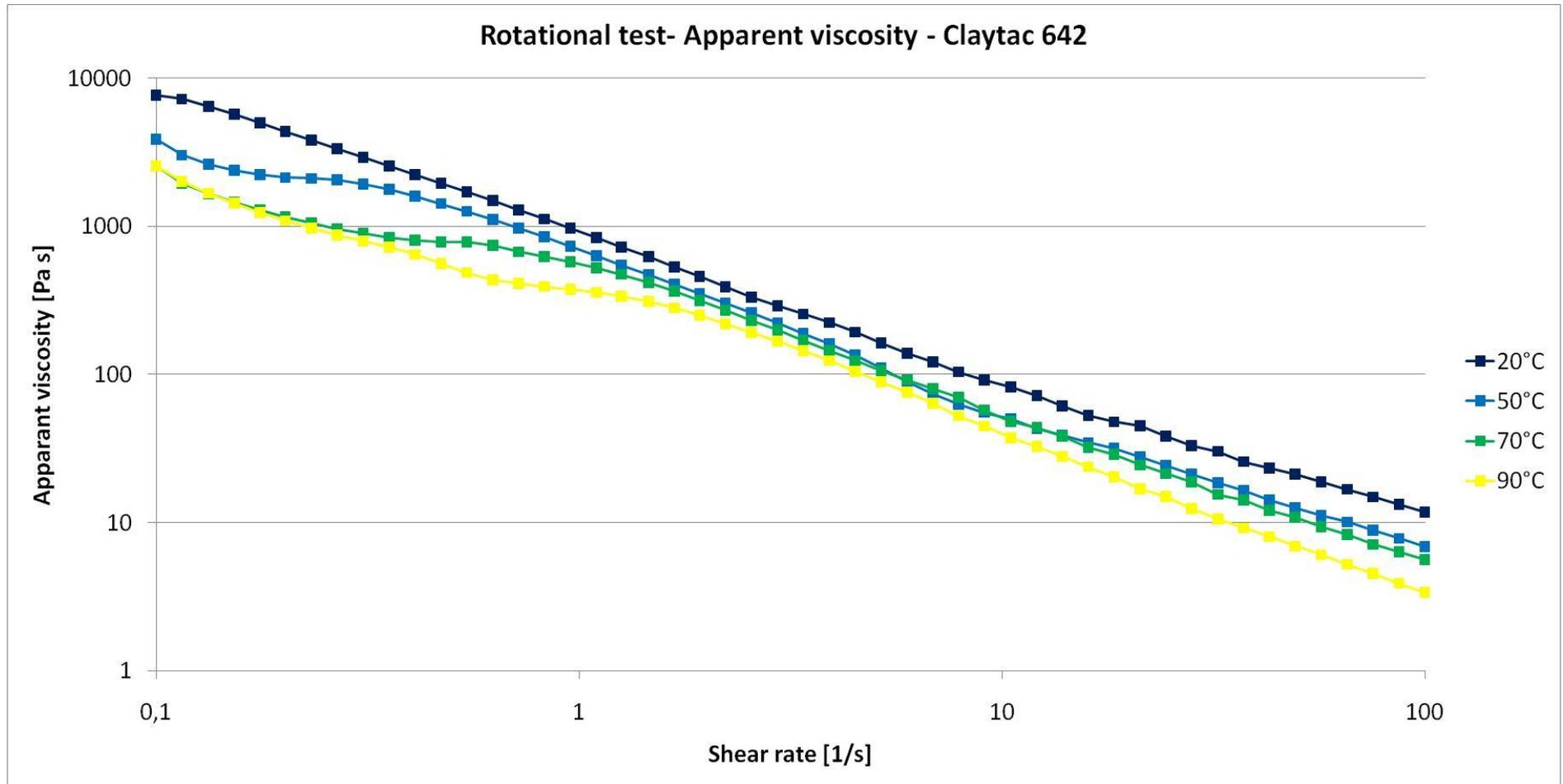


Figure 34 local minimum Claytac 642

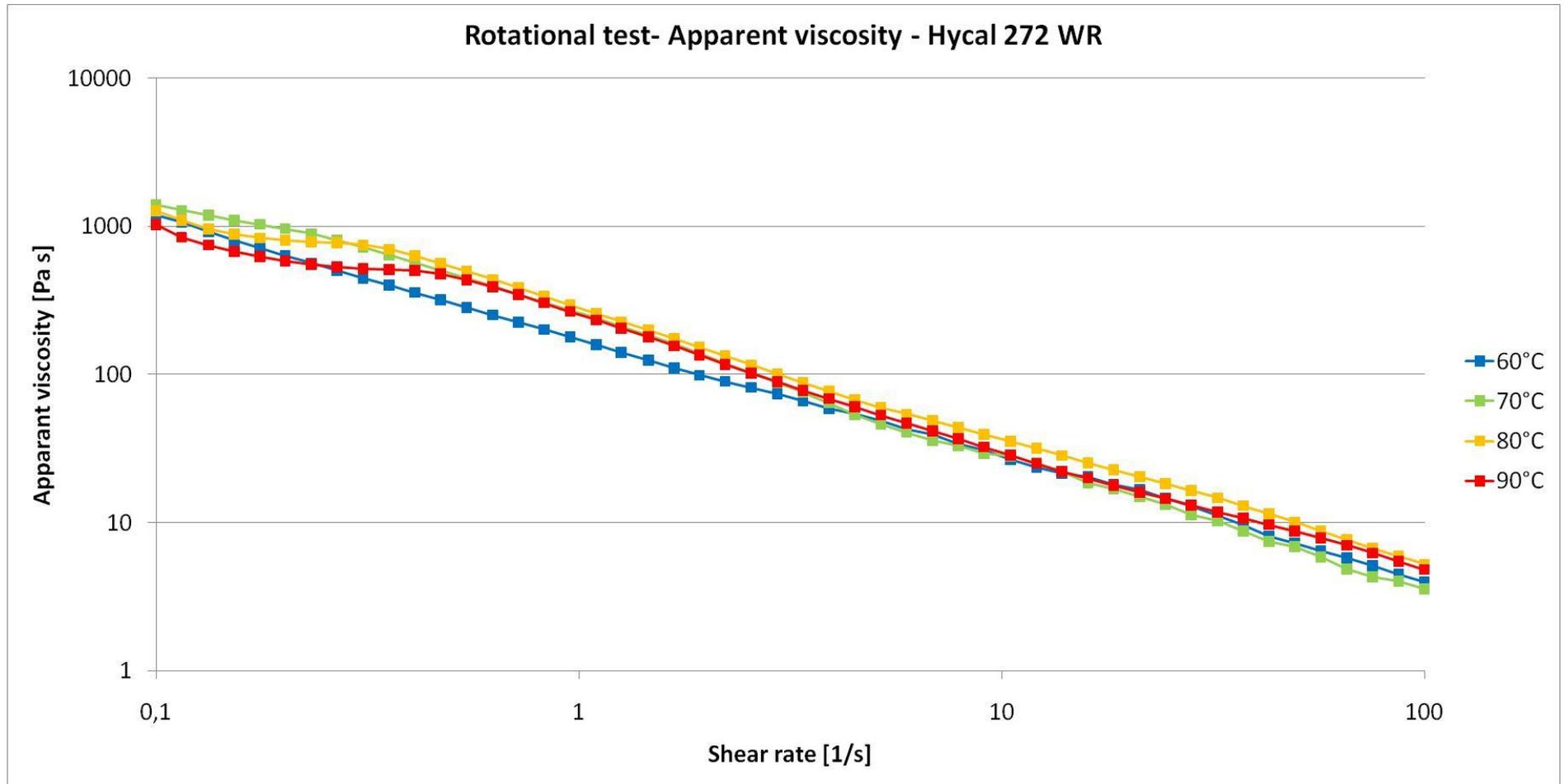


Figure 35 local minimum Hycal 272 WR

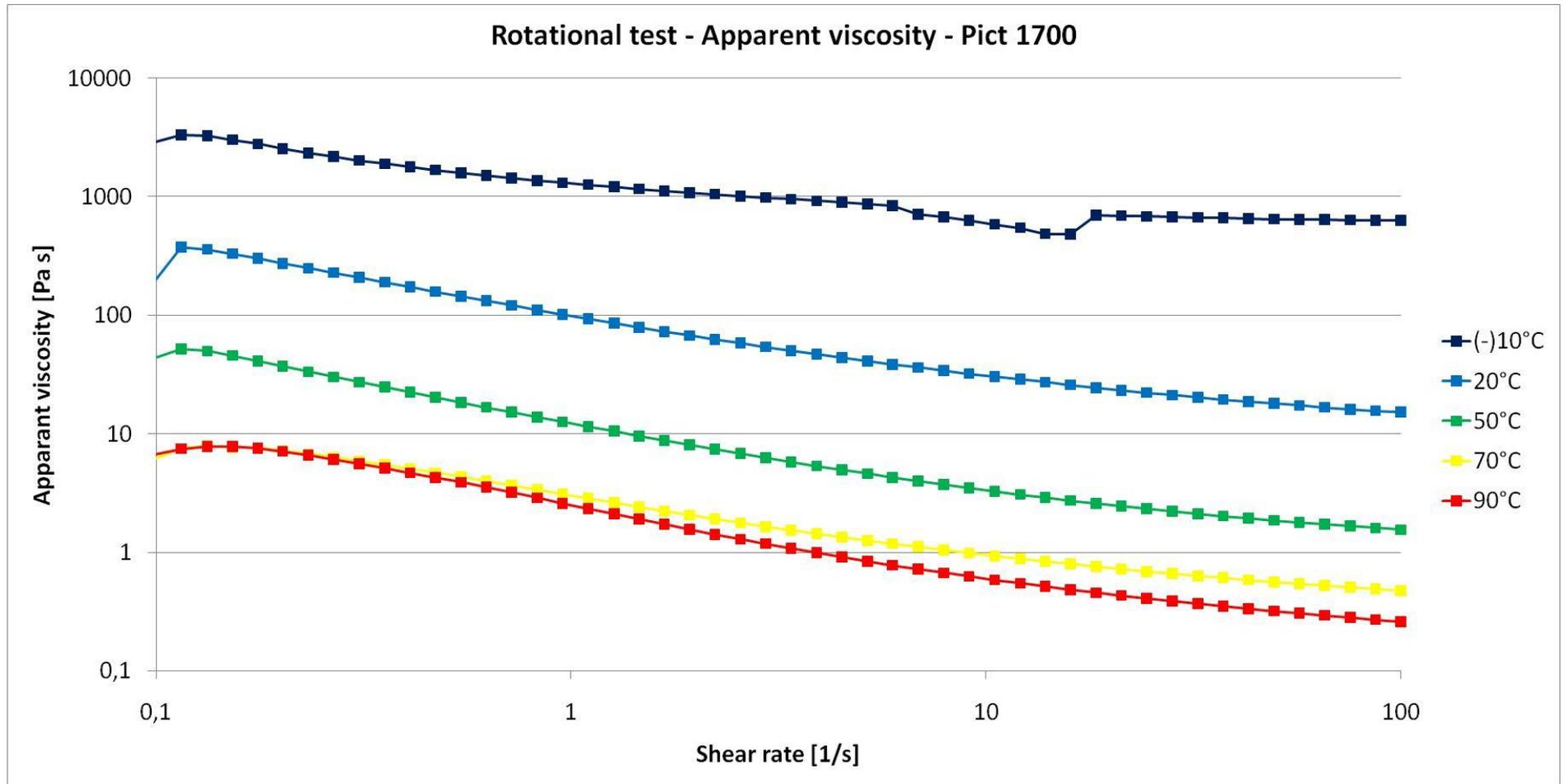


Figure 36 local minimum Pict 1700

## **Appendix 2 Oscillating test**

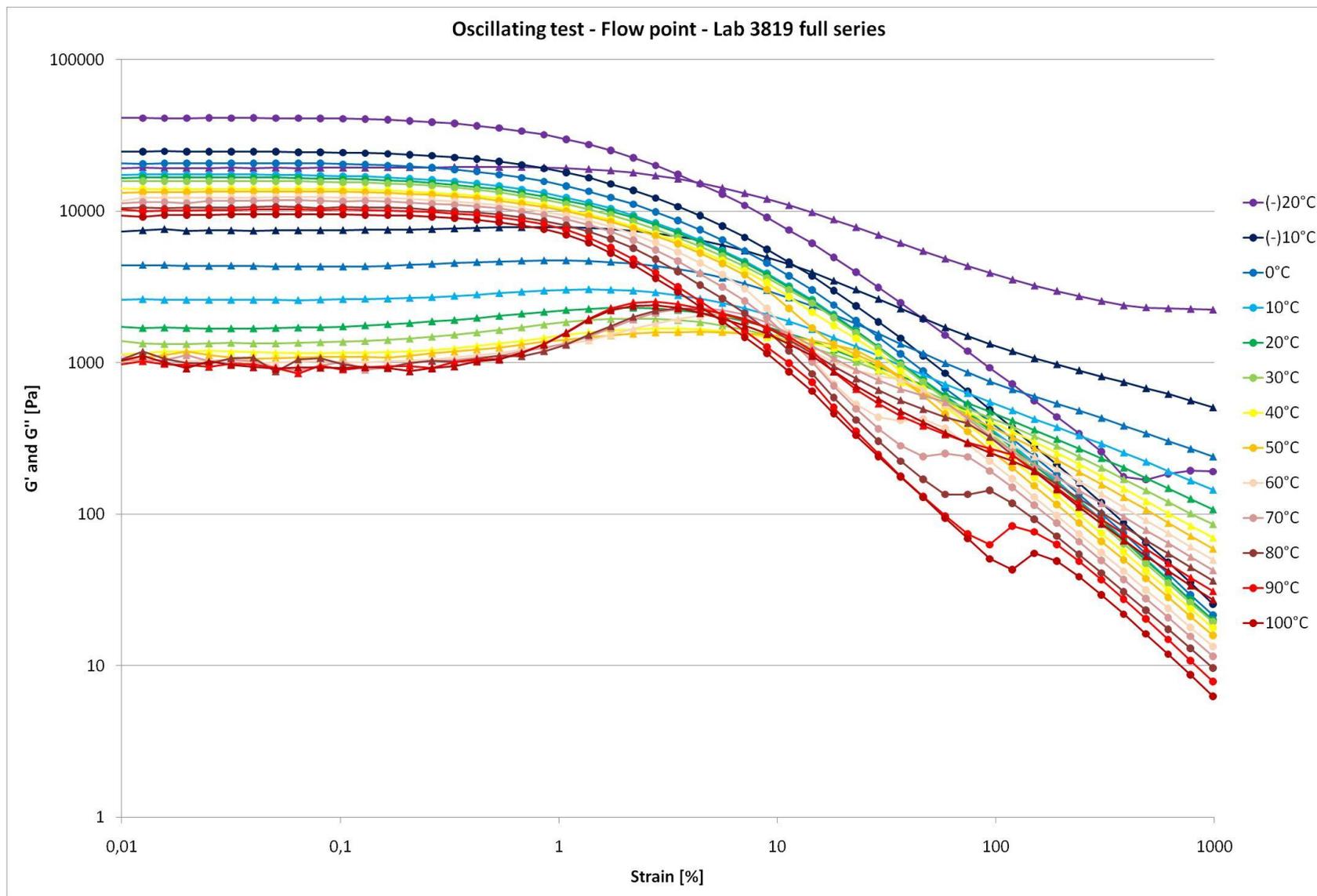


Figure 37 Lab 3819 full series

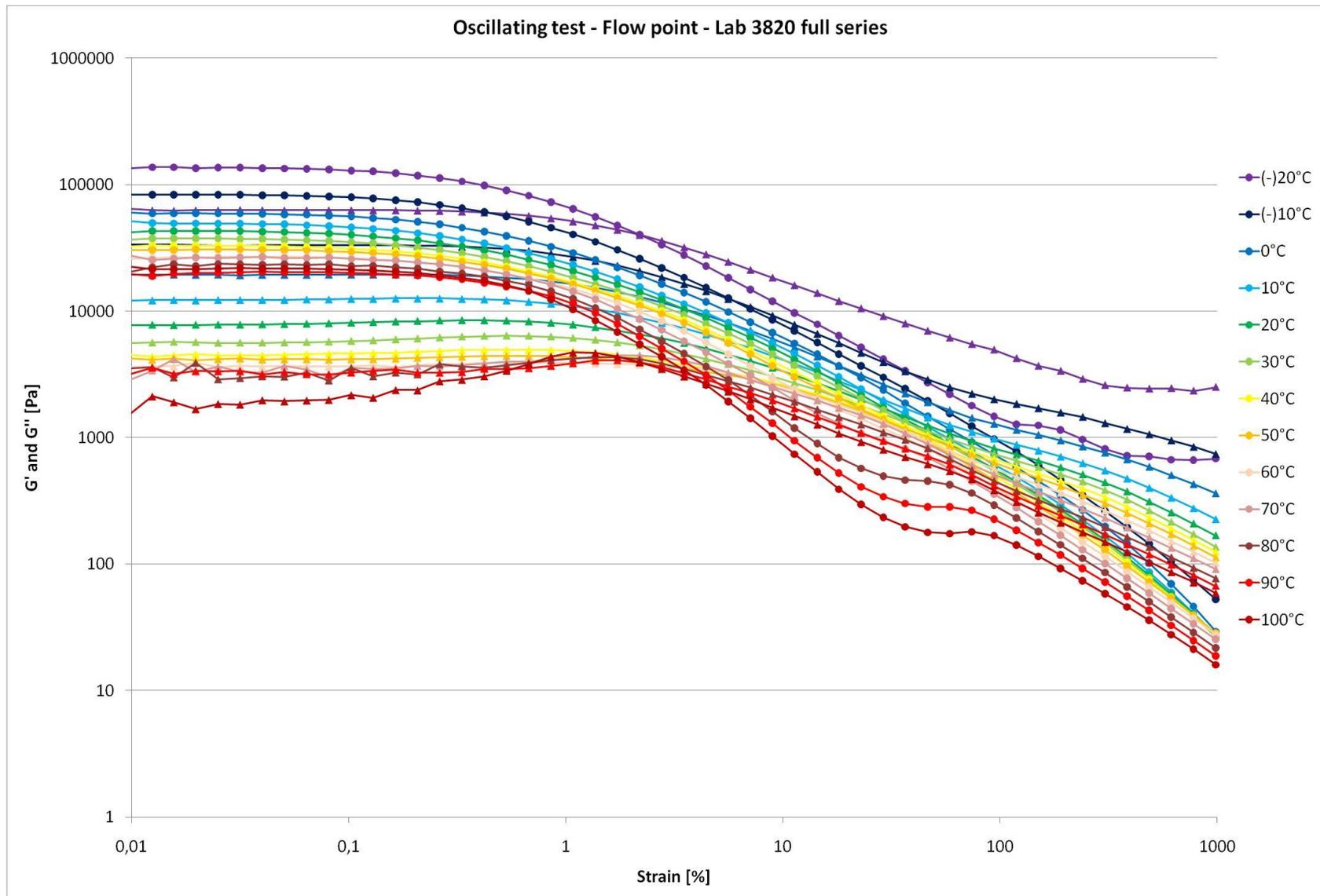


Figure 38 Lab 3820 full series

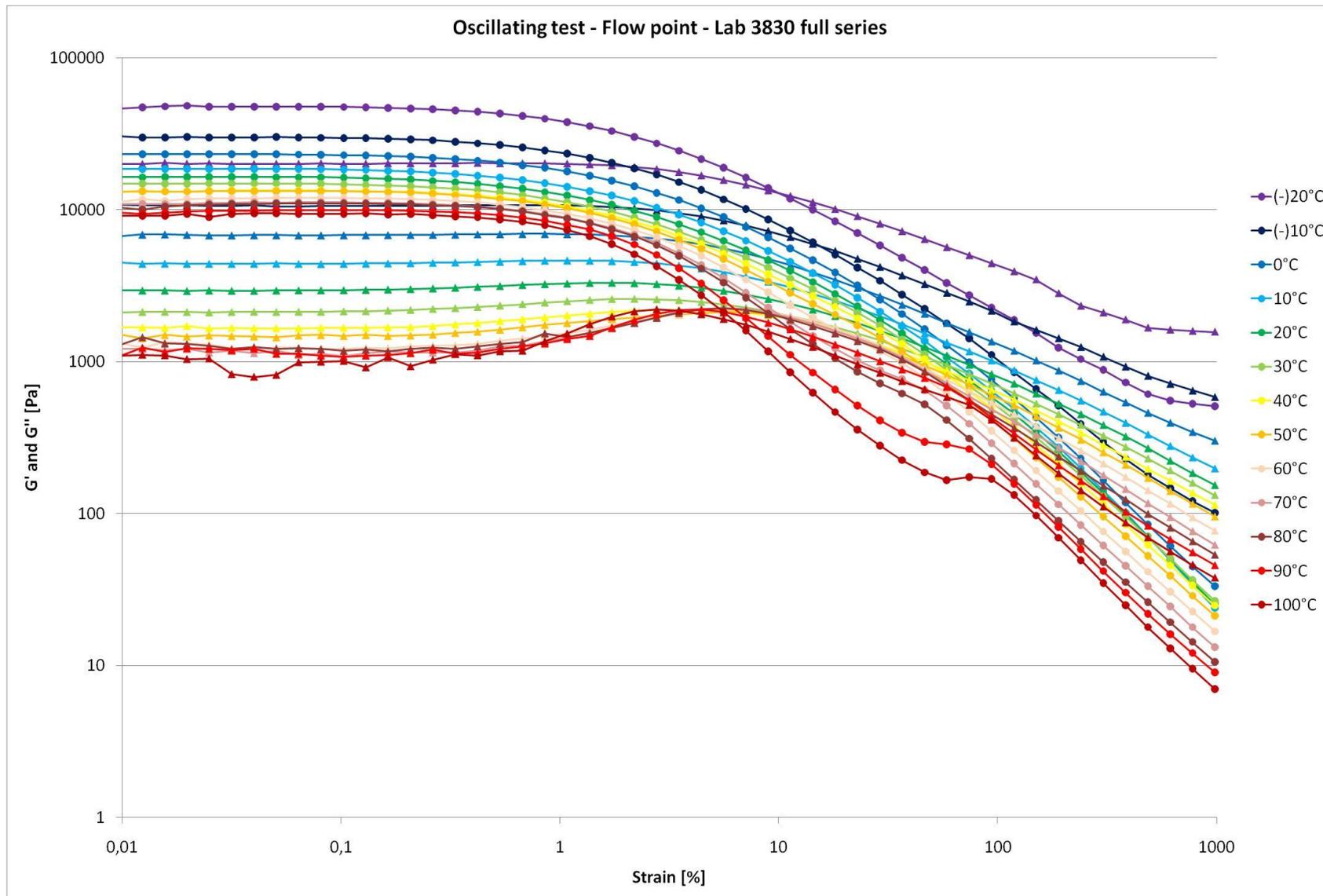


Figure 39 Lab 3830 full series

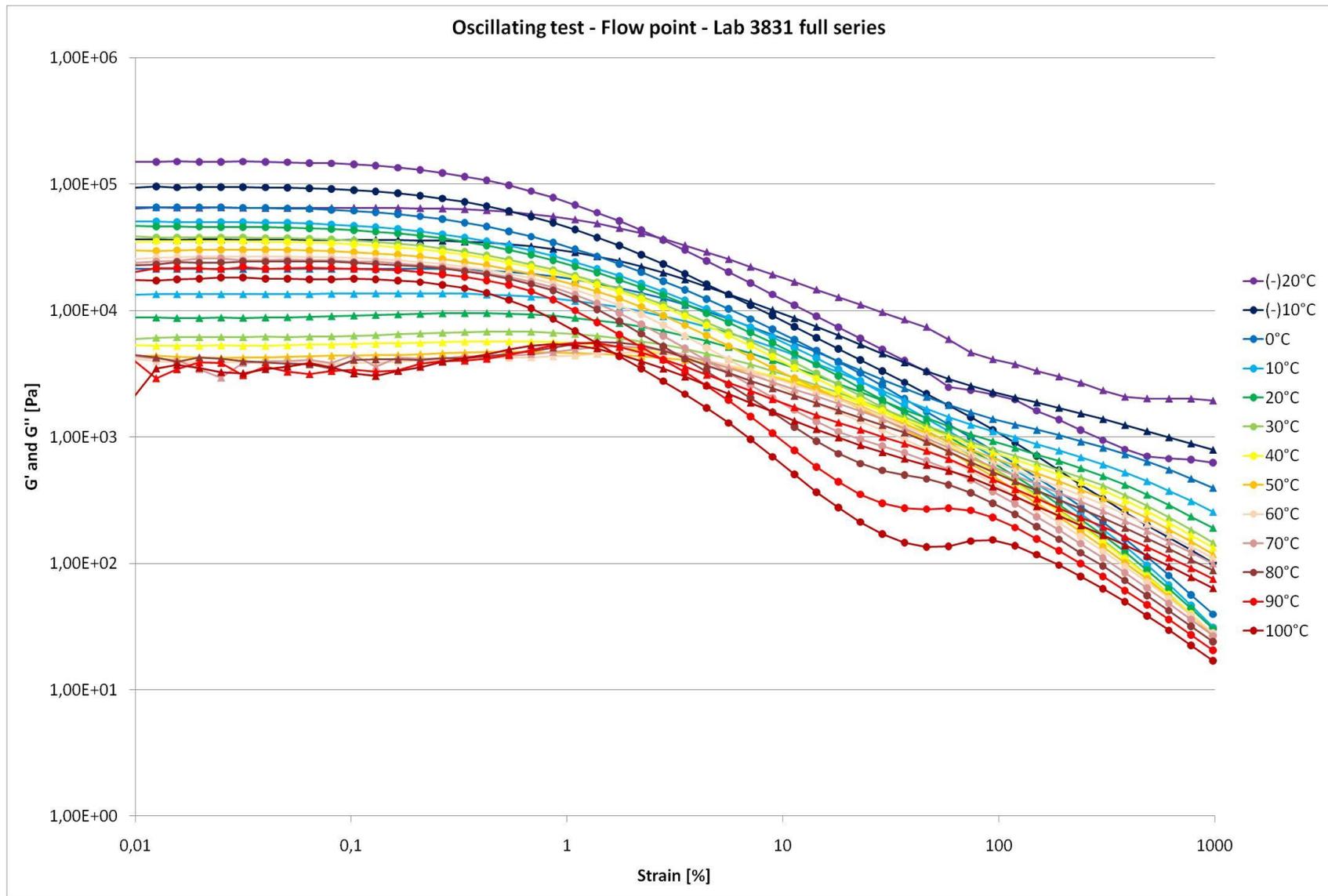


Figure 40 Lab 3831 full series

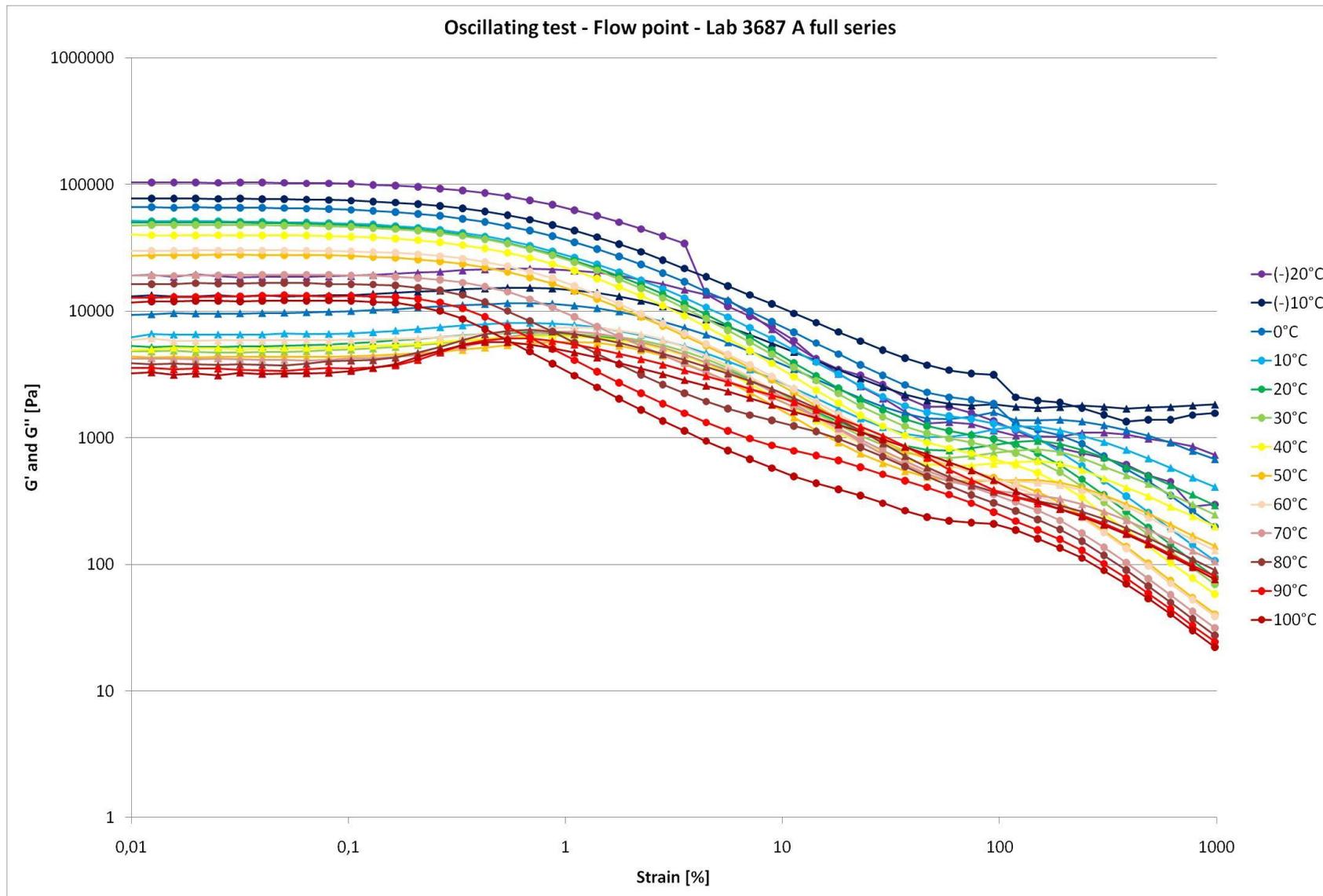


Figure 41 Lab 3687 A full series

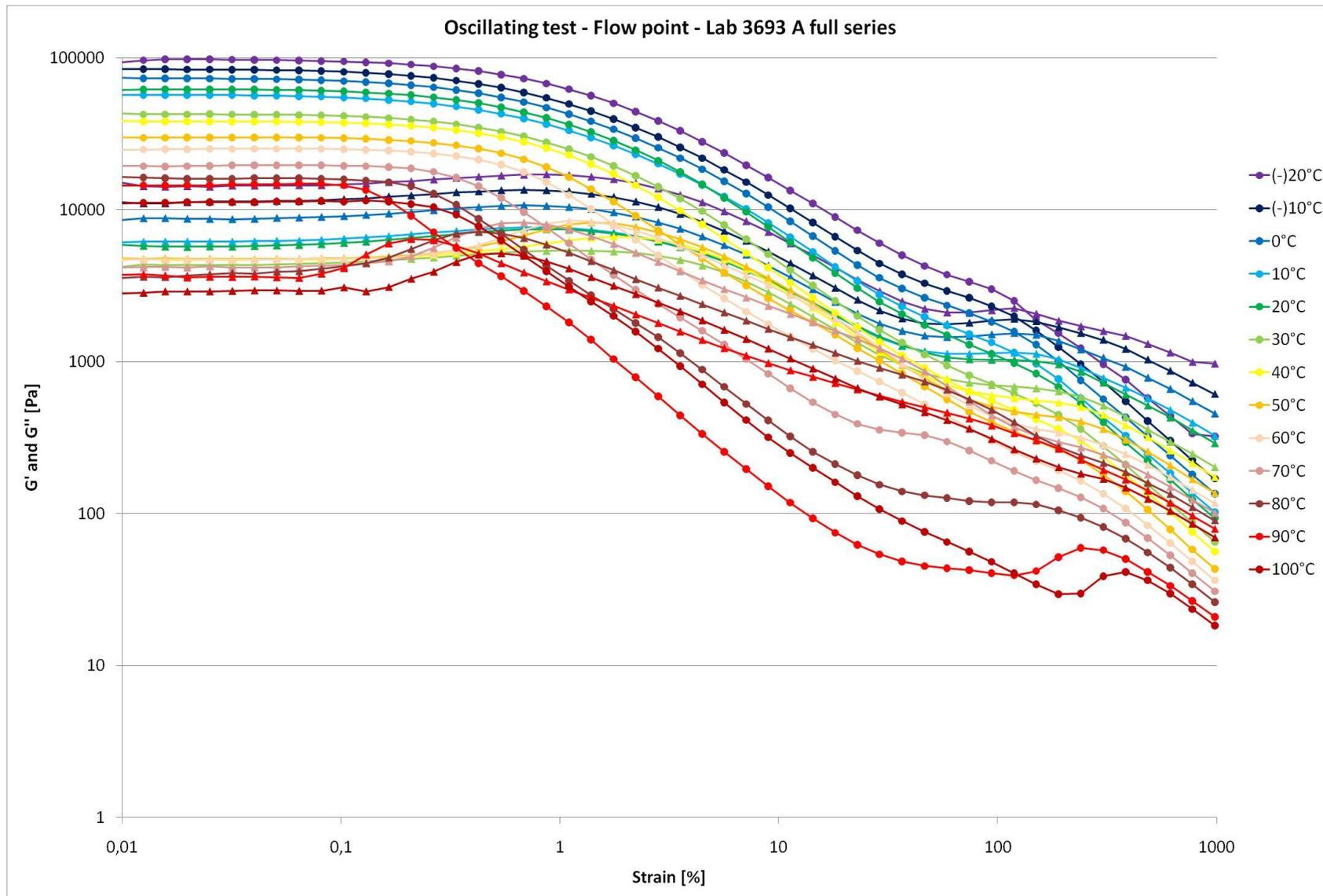


Figure 42 lab 3693 A full series

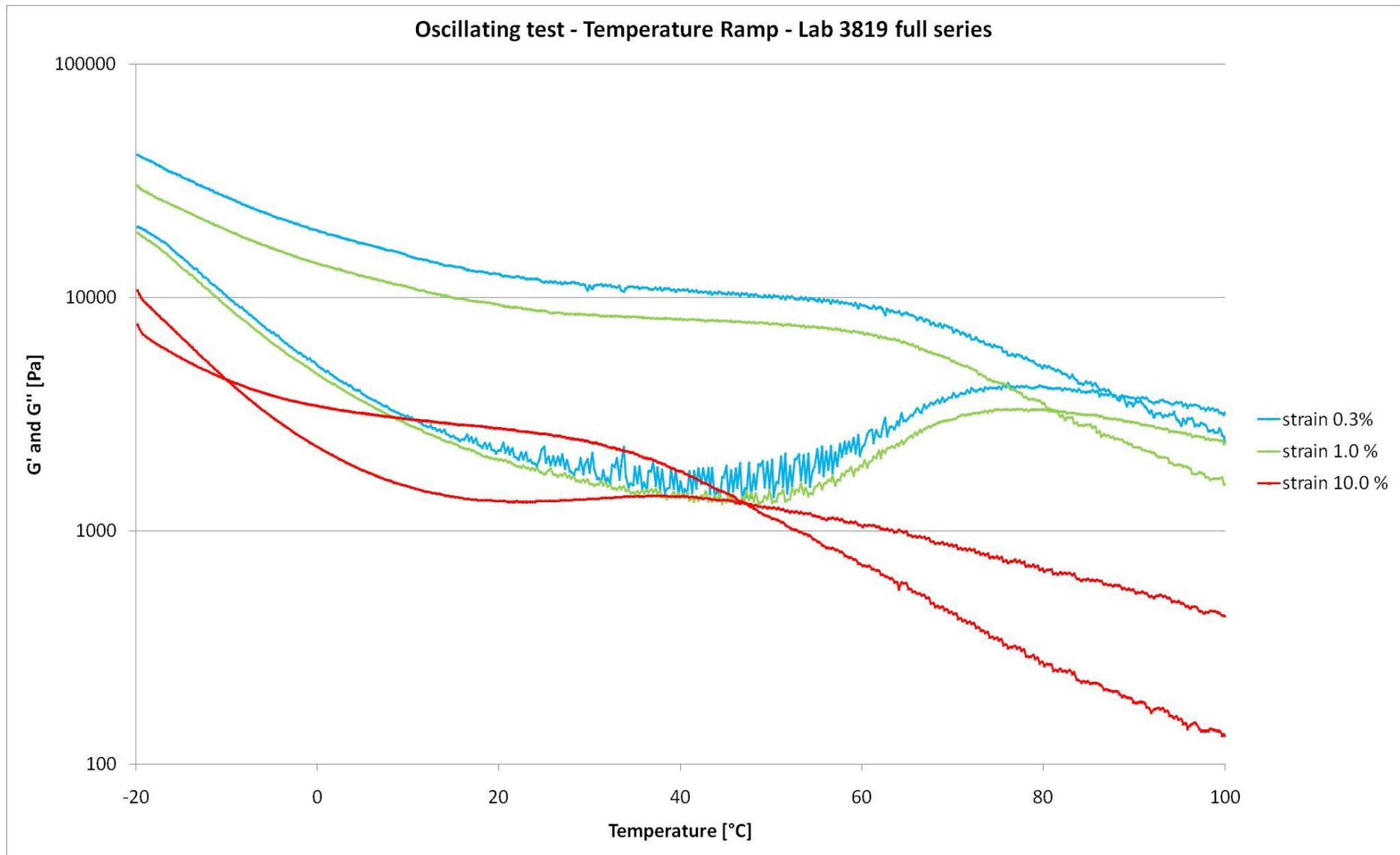


Figure 43 Lab 3819 temp ramp

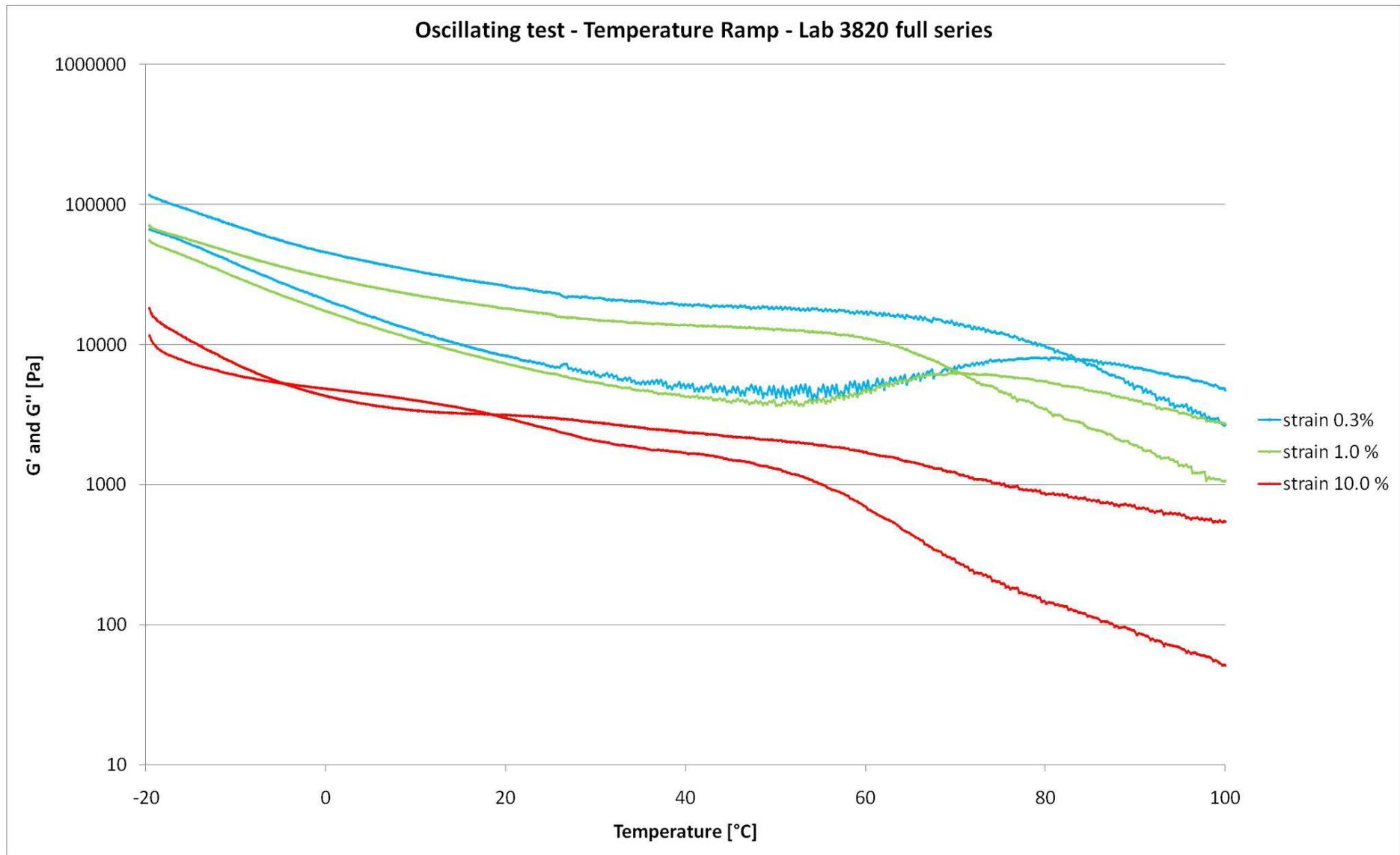


Figure 44 Lab 3820 temp ramp

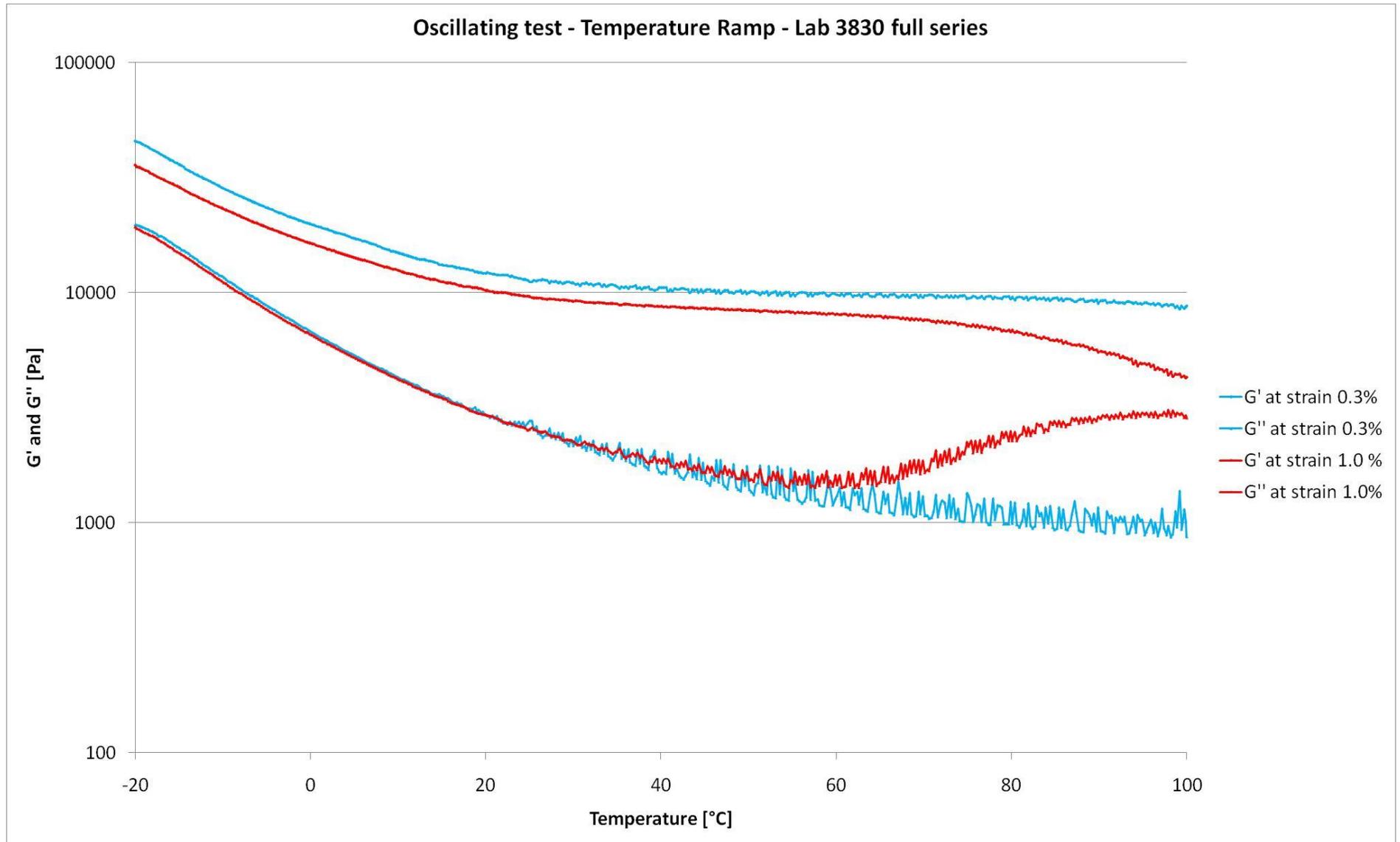


Figure 45 Lab 3830 temp ramp

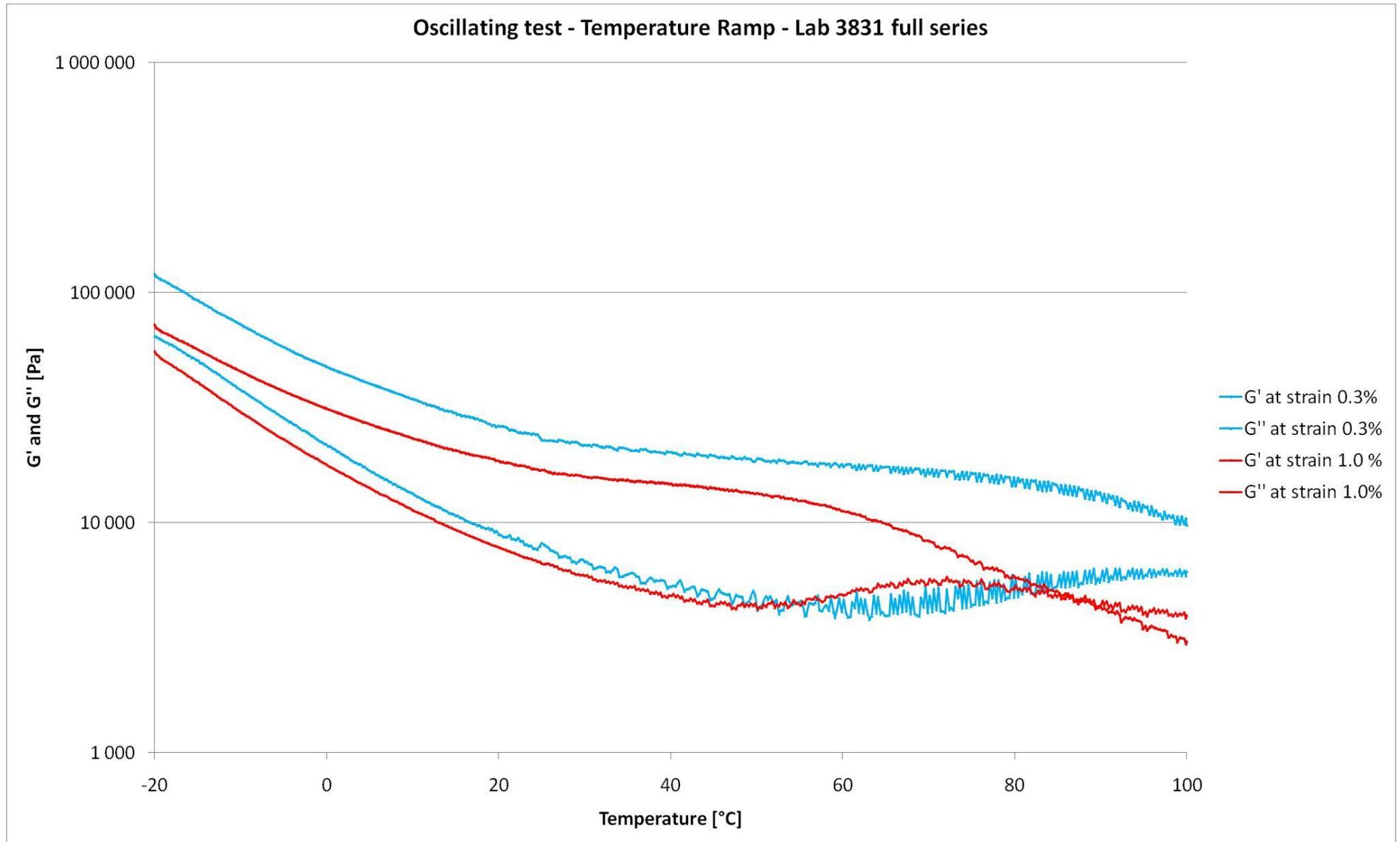


Figure 46 Lab 3831 temp ramp

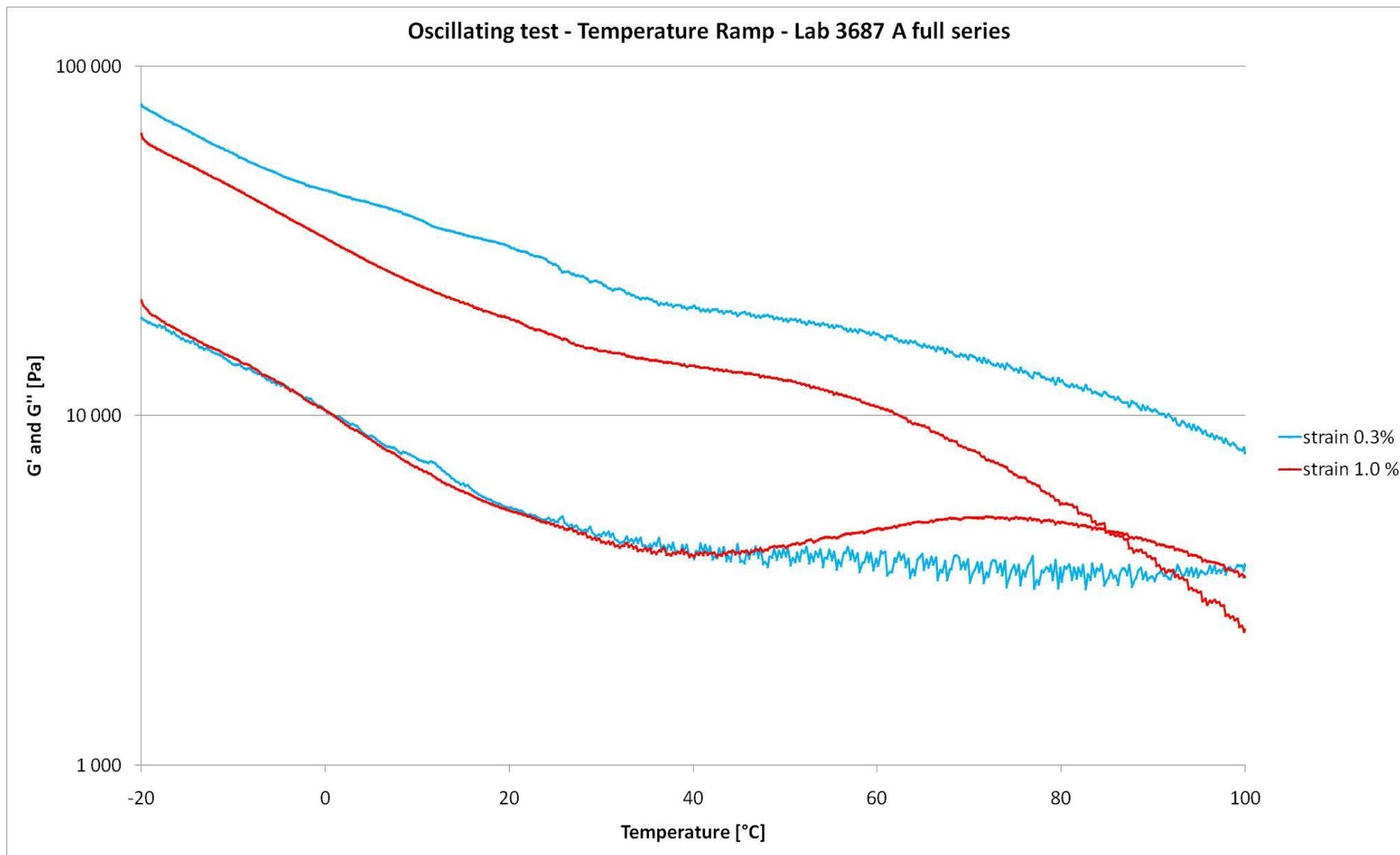


Figure 47 Lab 3687 A temp ramp

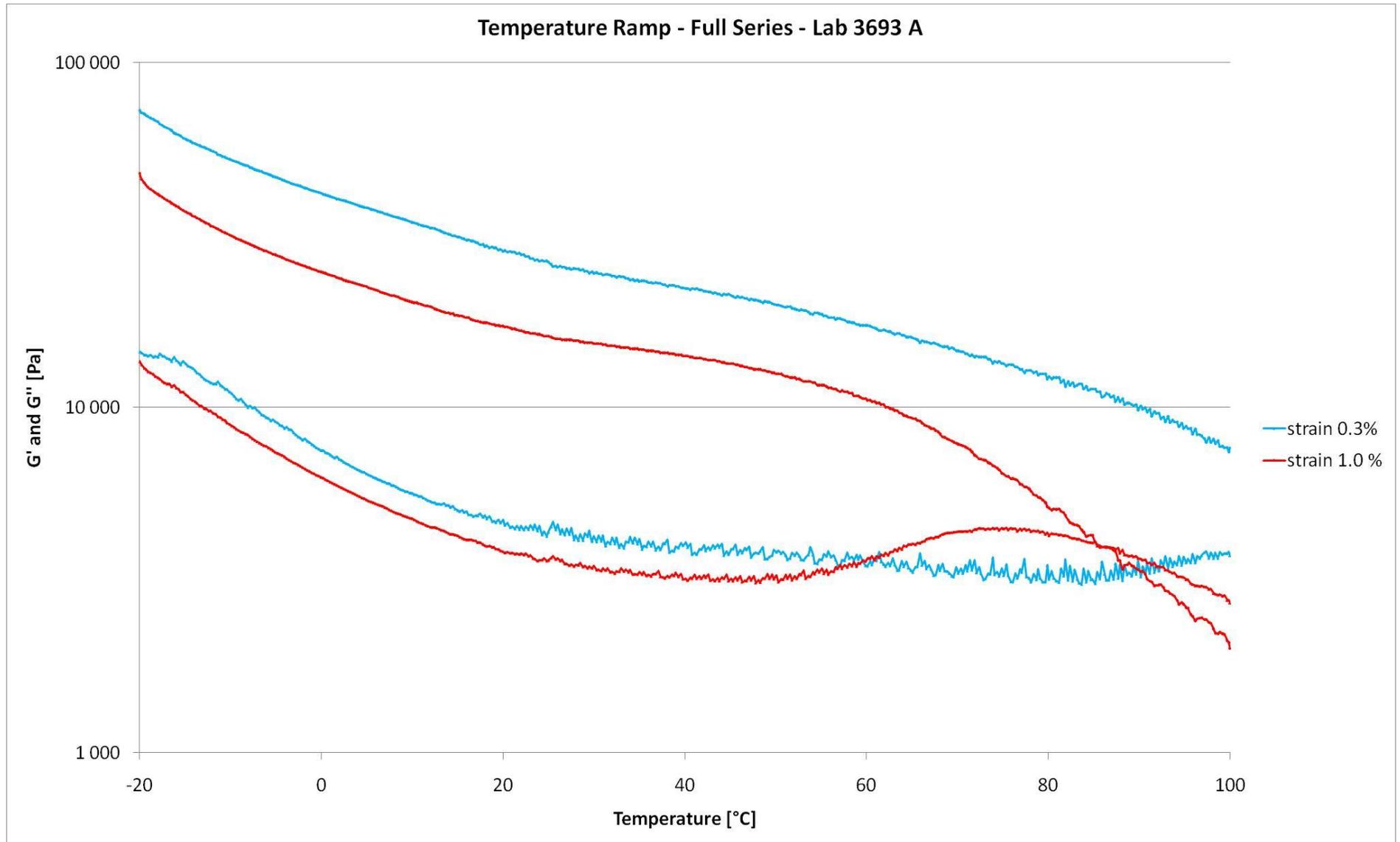


Figure 48 Lab 3693 A temp ramp

## **Appendix 3 Comparing greases**

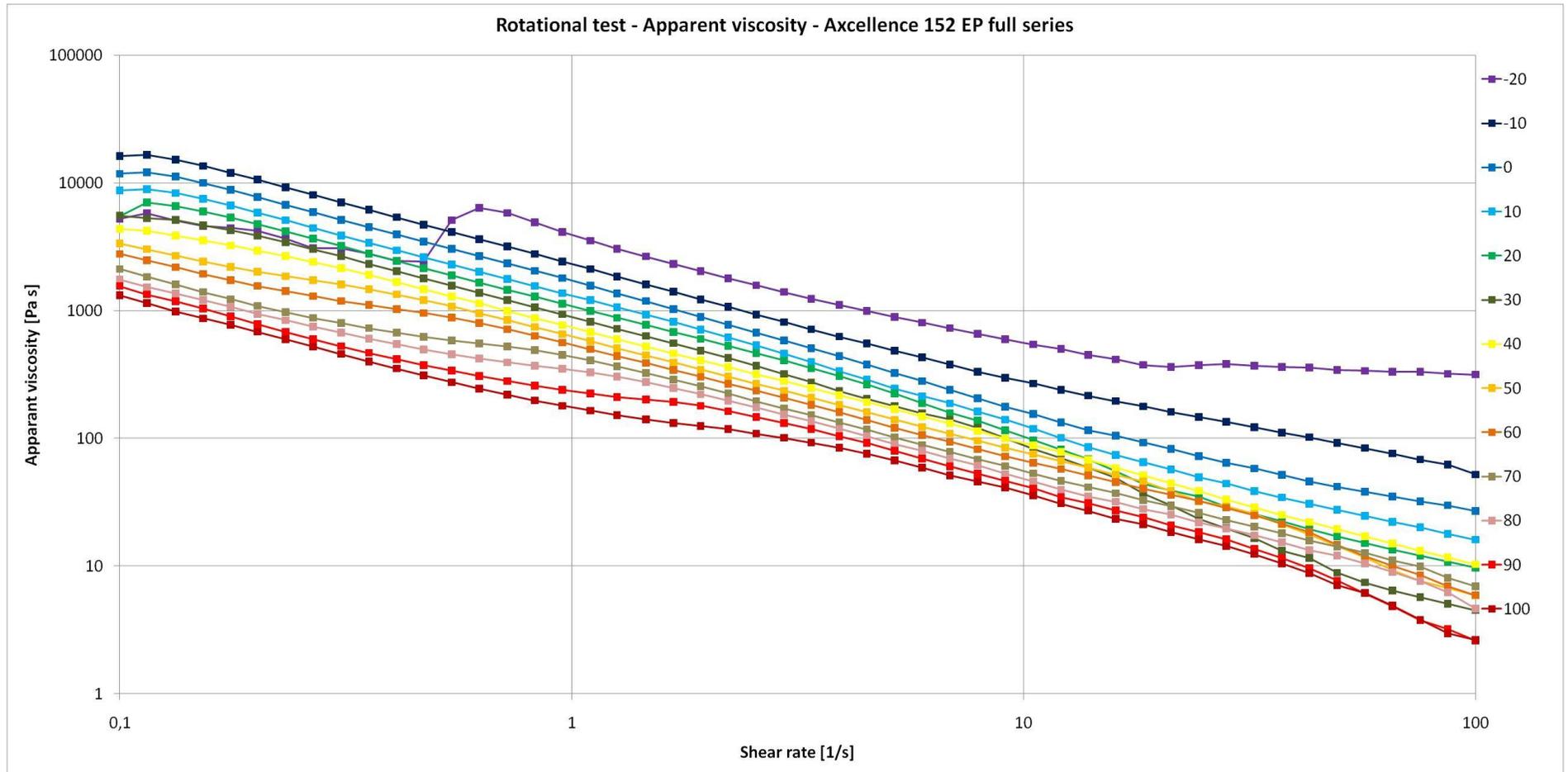


Figure 49 Axcellence 152 EP full series

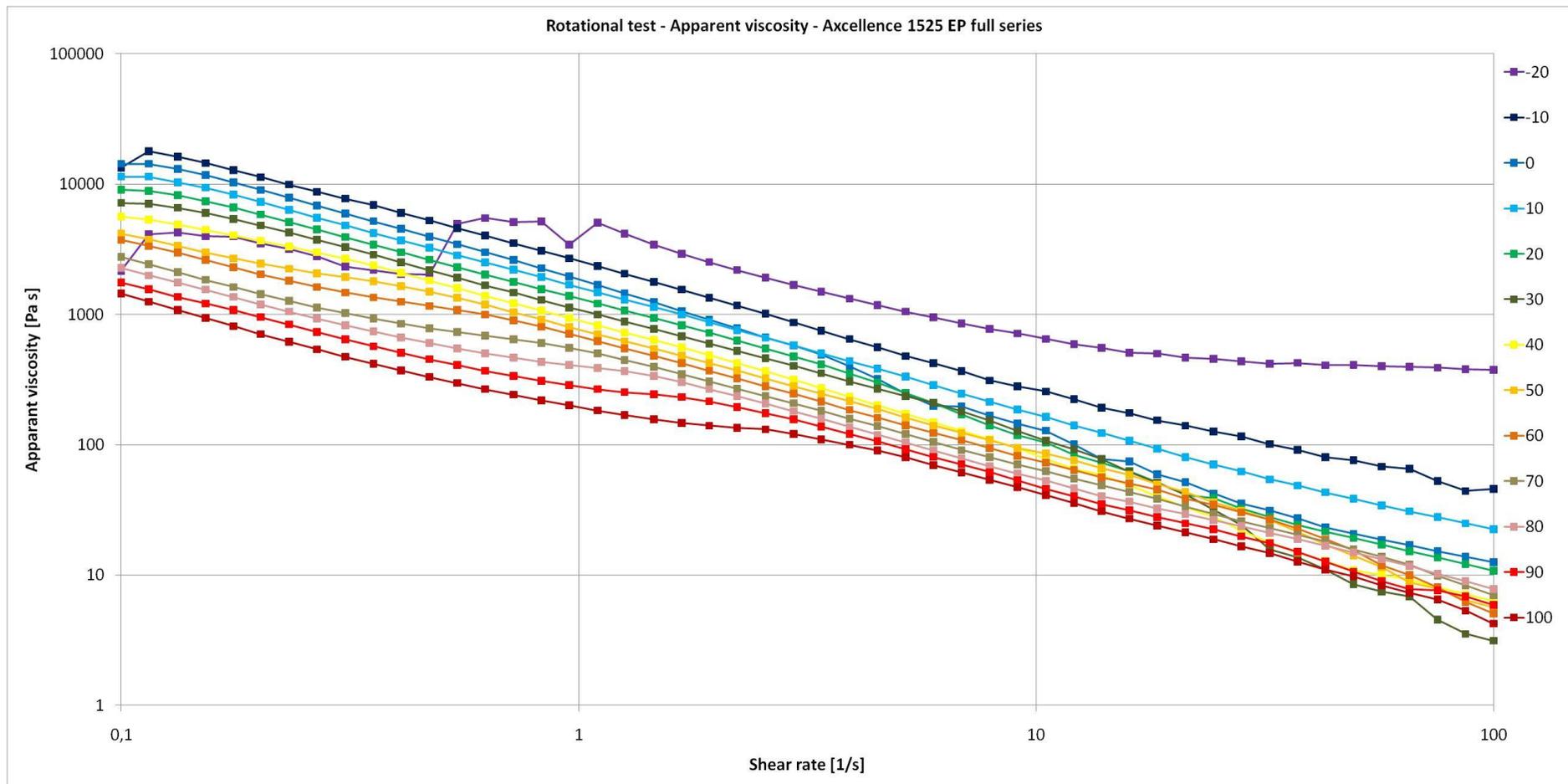


Figure 50 Axcellence 1525 EP full series

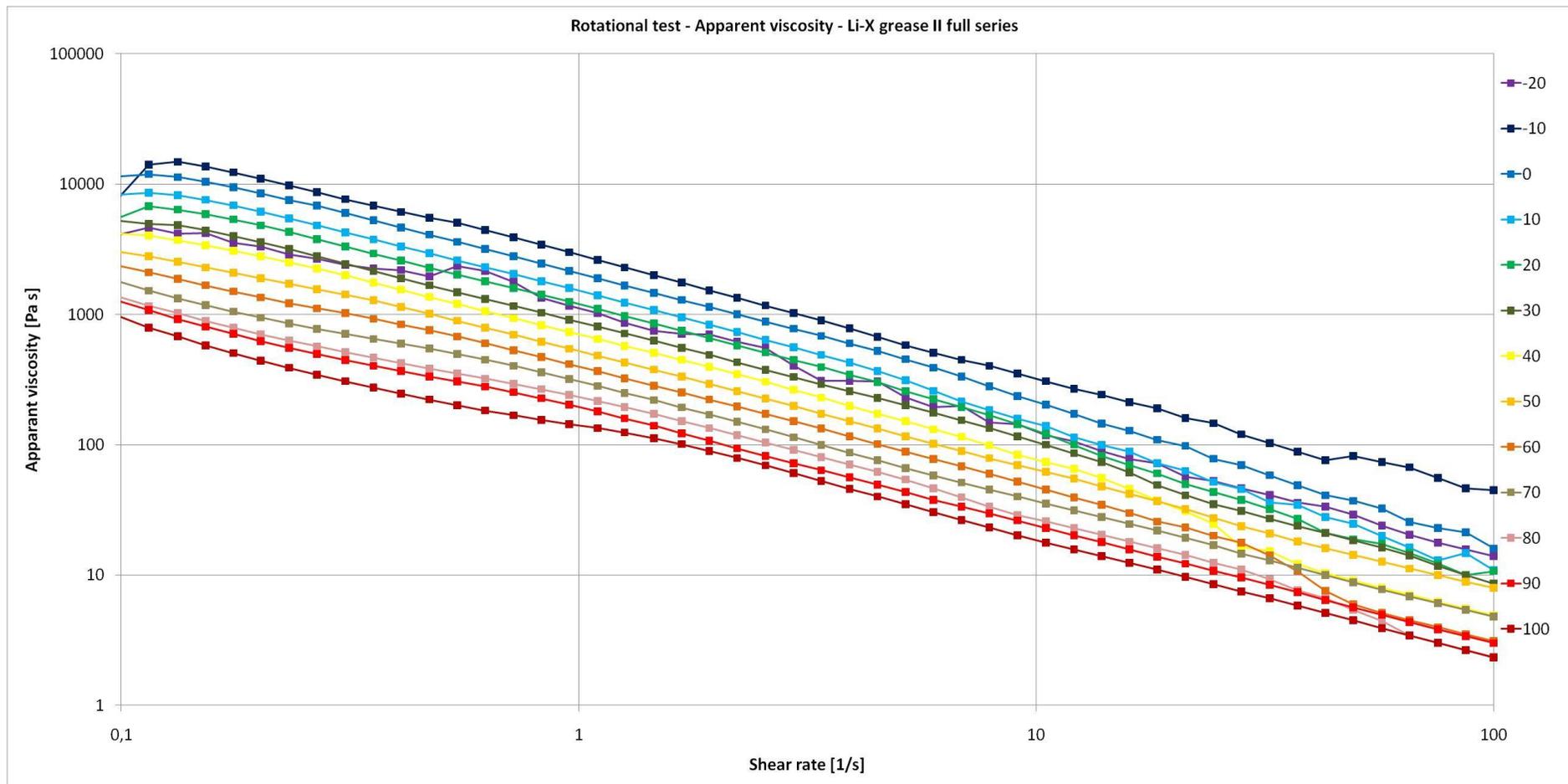


Figure 51 Li-X grease II full series