IDENTIFICATION AND ANALYSIS OF WEISSENBERG EFFECT AND THIXOTROPY OF A HIGH VISCOSITY SILICONE OIL IN RHEOLOGICAL MEASUREMENT'S RESULTS

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ABSTRACT

Silicone oils, which are widely used in the basic and applied researches as well as in the automotive industry, belong to the non-Newtonian, pseudoplastic fluids with strong viscoelastic properties. Technical modelling and simulation of its behaviour needs material laws in the entire range of the operations. Material model development for advanced engineering calculation purpose requires reliable and accurate rheological measurements. The actual viscosity of this kind of artificial and neutral fluid depends not only on the actual degradation status due to the high thermal load, shear rate, instantaneous temperature and pressure. During rotational rheometer measurements, also Weissenberg effect and thixotropy have significant impact on the measured viscosity results. (The Weissenberg effect is an unwanted and curious phenomenon when the fluid leaves the container and climbs up on the rotating measuring head of the rheometer by hindering the measurement. The thixotropy means a special behaviour when the viscosity of the fluid changes over time under relaxation after shearing or under continuous shear rate due to the restructuring of molecular chains.) Hence, the aim of the present paper is to discuss in detail the thermal degradation mechanism of silicone oils and to identify the Weissenberg effect and thixotropy in rotational rheometer measurement results conducted on AK 600 000 STAB high viscosity silicone oil used in torsional vibration dampers. Simple shear tests with concentric cylinder measuring head were performed on Anton Paar Physica MCR 501 rotational rheometer in 25 – 200 °C temperature and 0.1 – 100 1/s shear rate ranges. The influence of temperature, shearing time, resting time and sample quantity on the recorded viscosity results have been thoroughly analysed. Based on the measurement results, methods have been suggested to reduce or eliminate the Weissenberg effect and proposals have been made for the correct interpretation of thixotropic behaviour of silicone oils in rotational rheometer measurement results.

Keywords: equilibrium viscosity, molecular regeneration, PDMS, rheology, thermal degradation, thixotropy, Weissenberg effect

1. INTRODUCTION

Silicone oils belong to the group of polymeric organosilicon compounds which are liquid polymerized siloxane with organic side chains. Polydimethylsiloxane (PDMS) is the most important member of this group which is considered a water-clear, chemically neutral, hydrophobic, non-toxic, non-flammable, odourless and pure technical fluid. PDMS is a synthetic and versatile material with favourable thermal and physical properties leading to a wide range of application fields. Considering its relatively high thermal stability, advantageous heat transfer properties, special rheological behaviour and frictional properties, PDMS is an essential test fluid for calibrating instruments, validating new rheological theories and measurement methods and is the most suitable medium in vibration damping technology [1]. In the automotive industry, it is used primary in torsional vibration dampers for controlling the unwanted torsional vibration and extending the service life of the crankshaft in high performance internal combustion engines. Understanding the thermal degradation process of silicone oil is a crucial step before applying rheological measurements and developing material models for numerical calculations.

1.1 Thermal degradation mechanism of silicone oil

Thermal stability determines the resistance of the material against heat and affect its lifetime. In case of thermal stability fails, degradation of the material occurs. Under the term ‘degradation’ the authors mean a final and permanent deterioration process of the molecular structure when the initial intact state cannot be recovered. The most appropriate method for monitoring the degradation of a fluid is determining its viscosity. Viscosity describes the degree of internal friction among the fluid layers which lead to energy loss.
The chemical formula of PDMS oil can be written as (H3C)3[Si(CH3)2O]nSi(CH3)3, where n is the number of repeating monomer units. Increasing n leads to initial viscosity increment. Fig. 1 shows the molecular (left) and chemical structure (right) of the silicone oil. As far as torsional vibration dampers application is concerned, the highest effect on molecular structure, on the viscosity and on the lifetime of silicone oil is the temperature.

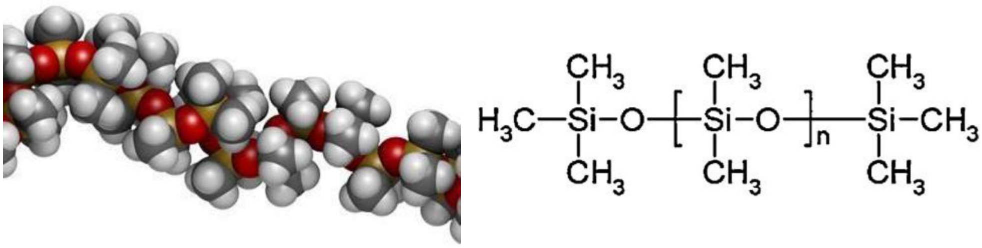

Fig. 1 Molecular structure (left) and chemical structure (right) of PDMS fluid [2][3]

Camino et al. [4][5] performed a detailed research with kinetic formal treatment and numerical simulation on silicone oil to reveal its thermal degradation mechanism. It turned out from their studies, that at lower temperatures (0 – 400 °C) thermal volatilization and weight loss of PDMS oil occurs with formation of cyclic oligomers through Si-O backbone scission. This kind of decomposition is directly proportional to the amount of heat increase that takes place and is determined by the diffusion and evaporation of produced oligomers following first order Arrhenius behaviour and having activation energy of ~ 27 kcal mol^{-1}. At higher temperatures (above 400 °C), a radical mechanism occurs through homolytic Si-CH_3 bond scission followed by hydrogen abstraction and leads to the formation of methane. The flexibility of PDMS chain is reduced by the cross-linking of macro-radical groups and further splitting of cyclic oligomers is restricted. The oxidative cross-linking and the reorganisation of split bonds improve the thermal stability of the oil and produces ceramic silicon-oxycarbide.
An extended silicone oil thermal degradation analysis [6] has been conducted at Knorr-Bremse Braking Systems Ltd. to describe the degradation mechanism in mathematical

way and to develop a lifetime calculation method for PDMS fluid used in viscous torsional vibration dampers. A theoretical variable (Degradation Factor) has been introduced that provides information about the rate of change in oil's viscosity exposed to a constant temperature for one day.

1.2 Weissenberg effect in silicone oil

Silicone oil is a non-Newtonian fluid, the relationship between the shear stress and shear rate in the fluid is nonlinear. Silicone oil belongs to the pseudoplastic fluids (also known as shear-thinning fluids) where the increasing shear rate makes the fluid more fluent by decreasing its viscosity.

Weissenberg or rod-climbing effect can be described as follows. When a vertical rod is partially submerged in a fluid, assuming the fluid wets the rod, surface tension causes the fluid surface to slightly rise up on the rod. By rotating the rod in Newtonian fluid (for instance water), inertia dominates, and the fluid moves to the boundaries of the container, far away from the rotating rod. In the case of non-Newtonian viscoelastic fluid (for instance molten polymers and silicone oil), the elastic forces generated by the rotation of the rod (and the consequent stretching of the polymer chains in the fluid) result in a positive normal force (towards the centre of the rod) and the fluid climbs up the rod, relaxes on it, and then overcomes the force pushing from below (see Fig 2.). [7]

Climbing up is driven by the influence of normal stress differences in the variation of the pressure value in the radial direction and as the normal stresses cause the total normal pressure to decrease in the radial direction, secondary flows develop around the rod (see Fig. 3). [7]

Fig. 2 Up climbing (left), relaxing on top (center) and down falling (right) phases of Weissenberg effect [8]

Figueiredo et al. [9] performed numerical simulations for isothermal and incompressible flow to provide an insight into the fluid dynamics of the Weissenberg effect and its inherent unsteadiness in viscoelastic fluids. The applied numerical solution is based on the combination of Volume-of-Fluid and Continuum Surface Force methods completed with a generic kernel-conformation tensor transformation to make the simulation more stable at high Weissenberg numbers. The outcome of the calculation is compared with experimental data from the literature related to low angular velocities. To investigate the elastic instabilities at high angular velocities, the Oldroyd-B model has been used. The relationship between the climbing height and angular velocity has been analysed. Also, the secondary flows and secondary vortex motions (perpendicular to the stirring main flow generated by the rotating rod) near the rod have been presented with 2D streamlines as it can be seen in Fig. 3.

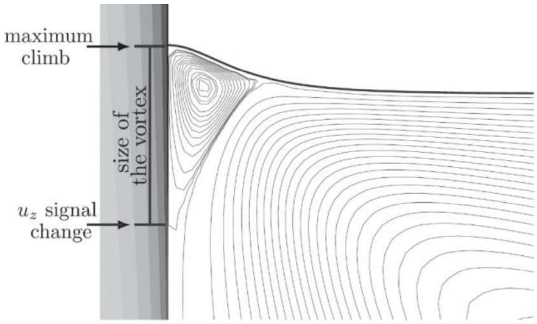

Fig. 3 Secondary flow and secondary vortex motion of Weissenberg effect in viscoelastic fluid [9]

1.3 Thixotropy in silicone oil

Thixotropy is a time-dependent shear thinning property of pseudoplastic fluids where the viscosity changes as the fluid sample is sheared and must recover after a certain period of time when the applied force is removed. If the thixotropy is not taken into account during a rheological measurement, the reproduction of the measured results can be hindered or incorrect results will be recorded.

The term thixotropy origins from the Greek words ‘thixis’ (to touch) and ‘trepein’ (to turn). The reason for the appearance of the mentioned phenomenon is a finite time required for the fluid molecules to organize and attain equilibrium viscosity when introduced to a steep change in shear rate. For instance, toothpaste or silicone oil flows out of the tube like a liquid when they are under pressure. According to Fig. 4, their viscosity becomes lower as force is applied. After the force lessens, the viscosity of both fluids recovers to its initial stiff state [10] [11].

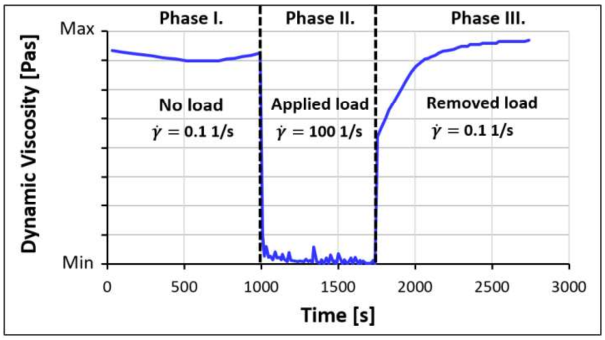

Fig. 4 Viscosity of silicone oil in initial state (Phase I.), in used state (Phase II.) and in regeneration state (Phase III.) where shear rate is denoted by $\dot{\gamma}$

Kőkuti et al. [12] investigated the thixotropic behaviour of AK 1 000 000 STAB high viscosity silicone oil at 30 °C and 80 °C temperatures during long time steady shear flow test with constant shear rate of 20 1/s (as the upper allowable shear rate limit is due to the appearance of Weissenberg effect) and small amplitude oscillatory shear test. It turned out from the rheological analysis that the oil sample shows thixotropic behaviour only at the higher investigated temperature and 3 hours was not enough for the oil to regenerate from its used state with 280 Pas of minimal viscosity to its initial state with 400 Pas initial viscosity. A shear stress – shear rate diagram has been also presented to show the hysteresis more explicitly.

Since the Weissenberg effect and thixotropy make the rheological analysis and material model development for silicone oil more complex, the aim of the present paper is to identify the Weissenberg effect and thixotropy in rheological measurement results performed on high viscosity silicone oil, and to make proposals for the correct treatment and consideration of the mentioned phenomena in the assessment of the rheological measurement results.

2. APPLIED RHEOLOGICAL MEASUREMENT METHOD

AK 600 000 STAB silicone oil has been used for rheological analysis at Bay Zoltán Nonprofit Ltd. for Applied Research in Miskolc. The investigated high viscosity oil type has 600,000 cSt initial kinematic viscosity or 600 Pas dynamic viscosity at 25 °C and it is preferred to be used as working fluid in torsional vibration dampers in automotive industry [13]. Simple shear tests (where the shear rate is linearly increased while the shear stress is recorded) with CC10 measuring head have been carried out on the oil samples. Anton Paar Physica MCR 501 high precision rotational rheometer has been used for this purpose that is a modern instrument by including air bearing (to minimize the friction loss during rotation of the measuring head) and strain-controlled measuring head (the shear stress in the sample is calculated from the instantaneous value of the current of the electric motor that rotates the measuring head in the sample) [14]. During measurements shear rate, shear stress, dynamic viscosity, temperature, speed of rotation and torque were recorded.

As far as Weissenberg effect analysis is concerned, the samples were investigated on discrete shear rates linearly increasing from 0.1 1/s to 100 1/s (on each discrete shear rate value approximately 50 measurement points were recorded (with 15 s shearing time per points) at different temperatures. At each temperature a shear rate value was highlighted where Weissenberg effect occurred. The influence of sample quantity at the appearance of Weissenbeg effect was also investigated at 25 °C by applying different amounts of silicone oil. The features of the conducted measurement series are found in Tab. 1. During the Weissenberg effect analysis 12 samples (I. – XII.) were taken from the oil (each measurement series is performed on a new sample) and 9320 measuring points were stored.

In terms of thixotropy analysis, three samples were taken from the oil. The samples were heated up and maintained at 100 °C to make them sufficiently fluent, easy to handle and to reduce the total time required for the measurement. Shear rate was increased linearly from 0.1 1/s to 100 1/s and 10 or 30 discrete shear rate values were selected for measurement points in each measurement series. Shearing time lasted for 15 or 30 s in each measurement point. The initial viscosity of the samples was also recorded by ‘zero’ measurements on 0.1 1/s shear rate lasted for 2.5 minutes.

Tab. 1 Conducted measurement series for Weissenberg analysis

Sample	I.	II.	III.	IV.	V.	VI.	VII.	VIII.	IX.	X.	XI.	XII.
Temperature [°C]	25	25	25	25	25	50	75	100	125	150	175	200
Quantity of oil [g]	0.853	1.021	1.178	1.303	1.303	1.303	1.303	1.303	1.303	1.303	1.303	1.303
Shear rate [1/s]	0.1 - 21.5	0.1 - 25	0.1 - 29	0.1 - 34	0.1 - 40	0.1 - 35	0.1 - 60	0.1 - 60	0.1 - 85	0.1 - 100	0.1 - 100	0.1 - 100
Number of recorded points	788	727	900	870	505	544	726	630	900	800	987	943

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Tab. 2 Conducted measurement series for thixotropy analysis

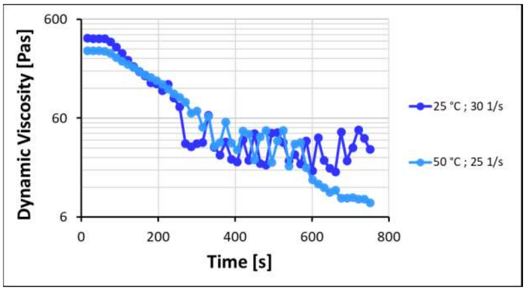
Sample	A				B			C		
Meas. series	0. meas.	1. meas.	2. meas.	3. meas.	0. meas.	1. meas.	2. meas.	0. meas.	1. meas.	2. meas.
Temperature [°C]	100	100	100	100	100	100	100	100	100	100
Shear rate [1/s]	0.1	0.1 - 100	0.1 - 100	0.1 - 100	0.1	0.1 - 100	0.1 - 100	0.1	0.1 - 100	0.1 - 100
Number of recorded points	10	30	30	30	10	30	30	30	30	30
Shearing time per points [s]	15	15	15	15	15	15	30	30	15	30
Total time [s]	150	450	450	450	150	450	900	900	450	900
Resting time [min]	-	0	177	0	-	0	20	-	0	1

3. MEASUREMENT RESULTS

3.1 Appearance of Weissenberg effect

Figure 5 presents how the Weissenberg effect can be identified in rheological measurement results. The viscosity on a constant shear rate and at given temperature must show a horizontal line at beginning (provided that logarithmic viscosity axis is used) and after that a monotonically decreasing trend must be seen as the molecular structure of the fluid is reorganized (arrangement of the highly anisotropic chains) due to continuous mechanical load. If the viscosity line has broken and then the viscosity values start oscillating, Weissenberg effect occurs, the oil sample leaves the container and climbs up on the rotating head of the rheometer. It turned out from the results, that in case of AK 600 000 STAB silicone oil Weissenberg appearances at 25 °C on 30 1/s shear rate (after 240 s), at 50 °C on 25 1/s shear rate (after 255 s), at 75 °C on 50 1/s shear rate (after 330 s), at 100 °C on 40 1/s shear rate (after 195 s) and at 125 °C on 85 1/s shear rate (after 225 s).

By increasing the temperature, the rod-climbing effect appears higher and on higher shear rates and the phenomenon can be delayed. Above 125 °C no Weissenberg effect has been observed.



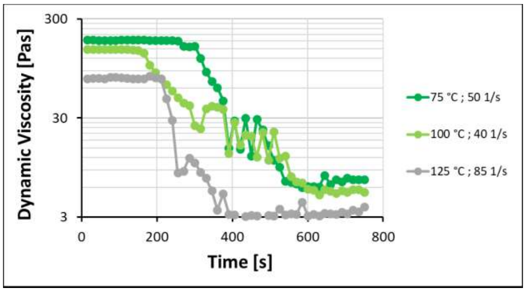


Fig. 5 The influence of temperature on the appearance of Weissenberg effect

Considering the first mark of Weissenberg effect in the viscosity results, Fig. 6 (left) shows only the measured viscosity values before the rod-climbing occurs. It is clearly seen that curves of 150 °C, 175 °C and 200 °C cover the whole investigated shear rate range without any mark of the Weissenberg effect. Figure 6 (right) highlights the influence of the investigated amount of oil sample onto the appearance of Weissenberg effect. According to the results of AK 600 000 STAB silicone oil, rod climbing occurred with 0.853 g quantity of oil on 15 1/s shear rate, with 1.021 g quantity of oil on 20 1/s shear rate, with 1.178 g quantity of oil on 28 1/s shear rate and with 1.303 g quantity of oil on 30 1/s shear rate.

By increasing the amount of investigated oil, the rod-climbing effect appears on higher and higher shear rates and the phenomenon can be delayed.

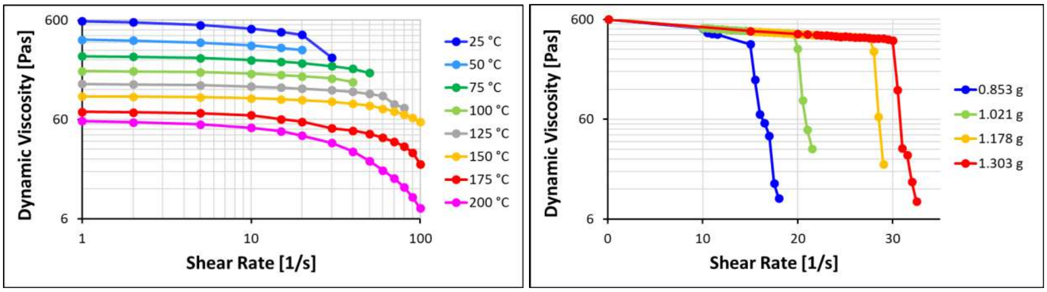


Fig. 6 Measured viscosity values without Weissenberg effect (left) and the influence of sample quantity on the appearance of Weissenberg effect (right)

As reported in [15], by applying parallel plate measuring head instead of concentric cylinder, the rotational rheometer can be converted into a normal force measuring instrument and the up-climbing phenomenon will not appear.

According to [16], the rod-climbing effect occurs only in case the measuring head rotates continuously in the fluid. By applying small amplitude oscillatory shear test (SAOS) instead of simple shear test, the appearance of rod-climbing effect will be avoided. The reason for this is the fact that in SAOS test the measuring head oscillates with a given frequency and does not make a complete turnaround. Thus, normal force does not generate that pushes the fluid up along the rod.

3.2 Appearance of Thixotropy

Figure 7 (left) shows the initial viscosity of each oil sample at 100 °C which are below the initial value of 600 Pas at 25 °C. The highest deviation is 34 Pas between A0 and B0 curves at 135 s that means 5.89% relative difference from the average viscosity (577 Pas) of B0 and C0 curves calculated for 135 s. Figure 7 (right) proves the fact, that the rheological results can be reproduced with high accuracy (provided that each measurement is done with a new oil sample). The highest deviation in measured viscosities before the Weissenberg effect (occurs on 40 1/s) is 80 Pas between A1 and C1 curves on 21 1/s shear rate. This means a 17.78 % relative difference from the average viscosity of A1, B1 and C1 curves calculated for 21 1/s shear rate.

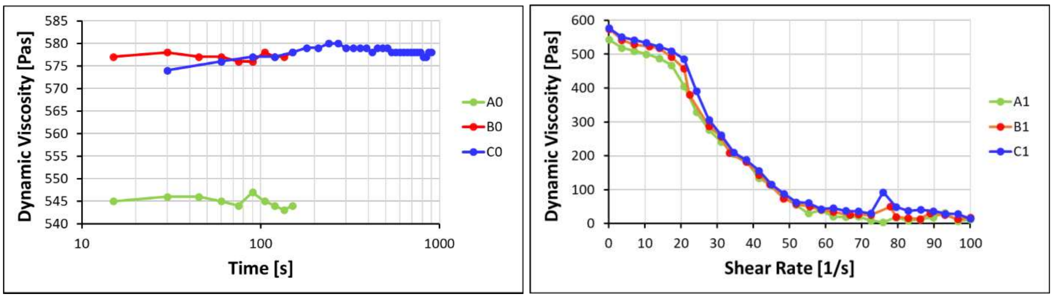
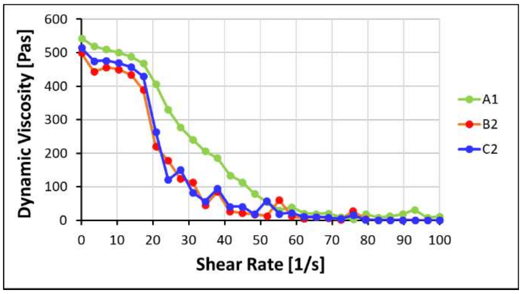


Fig. 7 Measured initial viscosities (left) and reproduced viscosity results (right)

The effect of shearing time on the recorded viscosity results can be clearly seen in Fig. 8 (left). In case the shearing lasts longer (B2 and C2 curves), a steeper decrease with lower viscosity values can be observed. The reason for this phenomenon is that longer shearing allows more time for the molecule chains to destructurize. The A1 curve with shorter shearing time reaches the destructurized state only on around 55 1/s while B2 and C2 curves already reached this equilibrium state on 40 1/s. The regeneration process of silicone oil can be well studied in Fig. 8 (right). The A1 curve represents the viscous behaviour of the previously original oil sample, which mechanical load started from the beginning of the measurement. The A3 curve shows the characteristics of a used oil, which mechanical load started from a previously loaded state. The initial viscosity growth on A3 curve in the small shear rate range (0.1 – 8 1/s) occurs due to the fact that the oil starts to regenerate after the previous load but above 10 1/s the current load overcomes the regeneration process and the curve reaches its minimum on 45 1/s. By waiting almost 3 hours to apply mechanical load on the oil again (A2 curve), the regeneration process had enough time to bring back the oil close to its initial state (A1 curve). It turned out from the thixotropy measurements that AK 600 000 STAB silicone oil suffers steeper viscosity loss with longer shearing time and requires more than 3 hours to regenerate to its initial state after a linearly increasing mechanical load valid in 0.1 – 100 1/s shear rate range.



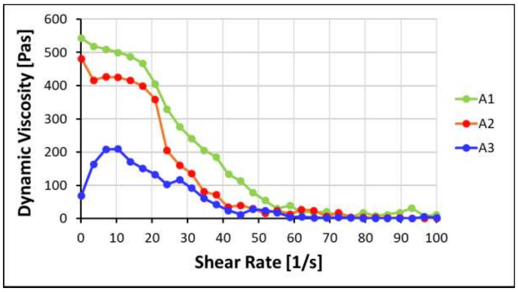


Fig. 8 Influence of shearing time (left) and resting time on the viscosity results (right)

4. CONCLUDING REMARKS

Following the introduction of degradation process, rotational rheometer measurements have been performed on AK 600 000 STAB high viscosity silicone oil used in torsional vibration dampers to investigate its thixotropic behaviour and identify the Weissenberg effect enhancing conditions. The oil samples were analysed with simple shear tests in 25 – 200 °C temperature and 0.1 – 100 1/s shear rate ranges.

In case of increasing the temperature, the rod-climbing effect appears on higher shear rates and the phenomenon is delayed. Above 125 °C no Weissenberg effect has been observed. Concerning the amount of oil sample, by increasing the amount of the investigated oil, the rod-climbing effect appears on higher shear rates and the phenomenon can be delayed. Moreover, either the application of parallel plate measuring head instead of concentric cylinder [15] and using SAOS measurement technique [16], the up-climbing phenomenon will not appear.

The thixotropic behaviour of silicone oil has been revealed by isothermal tests where the effect of resting time and shearing time on the viscosity results have been demonstrated. As far as the correct interpretation of the thixotropic behaviour in viscosity results is concerned, it is necessary to record each oil sample's initial viscosity before the rheological analyses, perform analysis always on a new oil sample being in initial state or rest the a previously loaded oil sample longer than 3 hours before a new analysis to give enough time for the oil molecular structure to regenerate, select and record an appropriate shearing time that allows the oil to reach its equilibrium state on each investigated shear rate. As the described phenomenon is the inherent characteristics of the silicon oils, one should consider this effect in the material model in the continuation of the present investigation.

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