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THE INFLUENCE OF THE THERMAL HISTORY ON THE MECHANICAL BEHAVIOR OF WAXY CRUDE OIL

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Abstract. *The pipelines employed during the production and transportation of crude oil are in contact with the seafloor. In this environment the temperature can be as low as 4 °C. At low temperatures, the wax solubility in the oil decreases and the precipitation of waxy crystals takes place. Wax crystallization changes the oil behavior from Newtonian to non-Newtonian. As a result, the viscosity of the crude oil increases and material gelation can take place. Rheometric tests have been extensively performed to analyze the influence of several parameters on the mechanical properties of waxy crudes oil at low temperature. The literature has shown that the thermal and shear histories have great influence on the rheological behavior of crude oils below the crystallization temperature. During the oil preparation for rheometric tests, the oil sample is usually heated above the wax appearance temperature to dissolve all wax in the oil. Then the sample oil is cooled to the test temperature and the experiment is performed. In the current work, rheometric tests are carried out to analyze the influence of the pre-test temperature. It can be anticipated that even above the crystallization temperature, the initial cooling temperature of the test has a great influence on the elastic modulus, gelation temperature and yield stress of wax crude oils.*

Keywords: *waxy crude oil; rheometry; thermal history; initial cooling temperature*

1. INTRODUCTION

At low temperatures wax crystallization can take place in crude oils. The precipitation of wax crystals provides a non-Newtonian behavior to the material. The crystals can form a structure, trapping the remaining oil in cages, gelling the material. Only 2wt% of crystals is enough to gel waxy crude oil (Aiyejina *et al*. 2011). Gelation of wax crude oil at low temperatures is an important issue in the petroleum industry. Gelation may take place in pipelines when the production or the transportation of the oil is interrupted for maintenance. High pressures – much higher than the usual work pressure – are required to break down the gel and start up the flow. The knowledge of the mechanical behavior of the wax crude oil at low temperature is fundamental to project the pipelines and pumps, and to plan the actions to resume the flow. High pressures can damage the pipelines and low pressures in some cases are not enough to break down the gel.

It is well known in the open literature that the thermal and the shear histories have a great influence on the gel strength of waxy crude oil. These influences are usually evaluated by means of five parameters: the initial cooling temperature, T_i, the temperature of the test, T_f, the cooling rate, $\dot{T}$, the shear rate during the cooling, $\dot{\gamma}_h$ and the aging time at the test temperature, t_{ag}. Beyond the history of the waxy crude oil, the test itself used to determine the yield stress of the material influence on the measured value. Some recent works stated that although the yield stress is test condition dependent the yield strain would be constant (Hou, 2012; Tarcha *et al*. 2015).

The influence of the initial cooling temperature was not fully investigated in the open literature. All the works that evaluated the effect of this parameter (Smith and Ramsden, 1978; Rønningsen *et al*. 1991; Marchesini *et al*., 2012, Jemmett *et al*., 2013; Andrade *et al*. 2015) studied the influence of temperatures close to the wax appearance temperature (WAT). In the current work, it is investigated if high values of initial cooling temperature are able to "erase" the thermal history of the material and eliminate the influence of this parameter of the test. Besides, the influence on the yield strain of the test condition and of the temperature itself is also evaluated.

2. EXPERIMENTAL TESTS

A sample of waxy crude oil obtained from Brazilian oil field and provided by Petrobras was used to conduct the investigation. The pretreatment at high temperatures to evaporate light ends, proposed in a recent work by Marchesini *et* *al*. (2012), was not performed. As concluded elsewhere (Andrade *et al*. 2015) the pretreatment not only affect the properties of the material but also do not improve the repeatability of the tests. Then, the material was investigated as received.

The rheometric tests were carried out by employing the TA DHR3 rotational rheometer that can measure a minimum torque of 5.10^{-9} Nm. All tests were performed by using a 40 mm diameter parallel-plate geometry with a gap of 1 mm and serrated surface to avoid wall slip (Dimitriou *et al*. 2011). The temperature in the rheometer was controlled by a Peltier-thermostatic bath system. Before any test, the sample was mixed and a small amount of material was collected and placed in the rheometer plate at 25 °C by using a syringe. The rotor was then lowered to its measuring position and the oil specimen was heated to the initial test temperature, T_i. The temperature was then kept at T_i for 30 minutes to assure thermal homogenization of the specimen. In all rheometric tests, a sample hood was used to minimize evaporation of light ends.

After holding the temperature at T_i for 30 minutes, the specimen was cooled from T_i to a final temperature T_f and then maintained quiescently for a specific aging time, t_{ag}. Finally, an oscillatory stress amplitude sweep or a stress loading rate was applied to determine the yield stress. The experimental procedure applied to the crude oil is depicted in Tab. 1. One can observe that it was evaluated the influence of the initial cooling temperature, test temperature, cooling rate, aging time and the test condition to break down the gel.

Table 1 Experimental procedure

Rotor type	Serrated parallel-plates, 40 mm diameter, gap of 1 mm
Initial cooling temperature, T_i, [°C]	30, 45, 60, 75, 90
Test temperature, T_f, [°C]	4, 10
Cooling rate, $\dot{T}$, [K/min]	0.1, 1.0
Static cooling	Small Amplitude Oscillatory Test ω= 0.5 Hz Strain amplitude= 10^{-3}
Aging time, t_{ag}, at test temperature [min]	0, 1, 10, 60, 120, 180
Test for evaluation of the yield stress	
Oscillatory Stress Amplitude Sweep	ω= 0.5 Hz τ_i= 10^{-1} Pa; τ_f= 10^4 Pa (100 points per decade)
Stress loading rate, $\dot{\tau}$, [Pa/min]	50, 500, 5000

3. RESULTS AND DISCUSSION

The DSC curve of the waxy crude oil analyzed in the current work is presented in Fig. 1. The DSC test was performed in an external institute (LACTEC, 2015) by employing the Netzch DSC 204 F1 differential calorimeter. In the DSC test the cooling rate was kept constant at 1 K/min, and the heat flow to maintain this cooling rate was measured. It is possible to conclude that at 29.9 °C an exothermic event – the precipitation of the first wax crystals – was observed, as shown by the dashed red line. This temperature is the WAT (Wax Appearance Temperature) of the crude oil analyzed in the current work.

3.1. Effect of Initial Cooling Temperature

All works in the open literature reported that the oil was firstly heated to an initial high temperature in order to eliminate any thermal history that exist in the material and then cooled down to the test temperature. In a recent work Andrade *et al.* (2015) concluded that the initial cooling temperature has a great influence on the gel strength of the material. In that work with exception to initial cooling temperature, all test parameters were maintained constant. As presented in Fig. 2 the authors observed that the yield stress varied from approximately hundreds to thousands after static cooling within the tested range of the initial cooling temperatures. One can observe that there was a critical initial cooling condition to this evaluated oil. In that case the critical range was between 45 and 50 °C. In other words, the highest values of yield stress were observed when the cooling started within this temperature range.

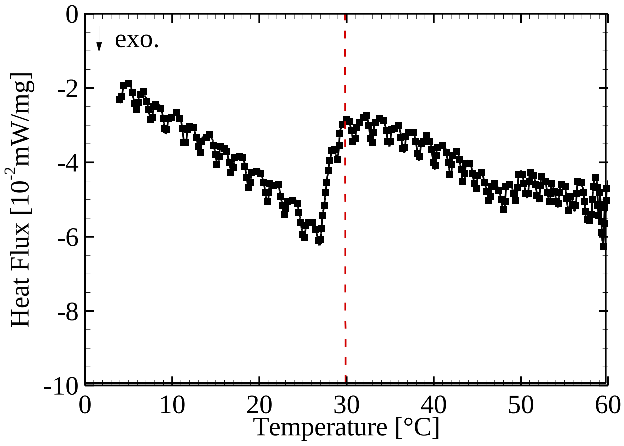

Figure 1 Differential scanning calorimetry (DSC) curve of the waxy crude oil of the current work.

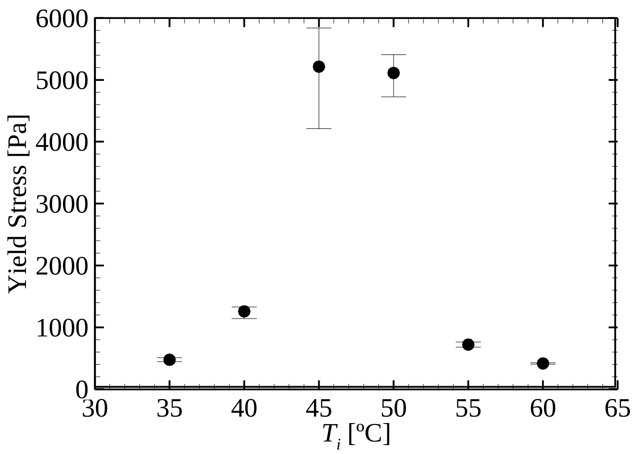

Figure 2 Yield stress of a Brazilian Crude Oil at 4 °C as a function of the initial cooling temperature (Andrade *et al*. 2015)

Smith and Ramsden (1978) reported that resins and asphaltenes in wax crude oils affect the dependency of the material strength on the initial cooling temperature. Resins and asphaltenes are called natural pour point depressants (PPD) (Al-Shafey *et al*., 2011). Some authors (Rønningsen *et al*., 1991, Venkatesan *et al*. 2005) state that the presence of such components changes the interaction between wax crystals, delaying the gelation process. If the oil is heated to a sufficiently high temperature, all wax crystals are dissolved and the PPDs that were linked to these crystals are free again to join new wax crystals that will be formed during a next cooling. On the other hand, if the oil is heated to an intermediate temperature, only a portion of crystals is dissolved and the PPDs mobility is refrained by the remaining wax crystals. In this case, wax crystallization takes place without PPDs interference as the sample is cooled down, resulting in higher gelation temperatures[#] (Rønningsen *et al*., 1991). According to Rønningsen *et al*. (1991) the maximum gelation temperature is obtained when the initial cooling temperature is high enough to dissolve all wax

[#] Temperature in which the amount of precipitated wax crystals changes the oil behavior from viscous to viscoelastic during a static cooling (Li et al., 2009) crystals but not so high to dissolve or dissociate the resins joined to the wax crystals. According to Marchesini *et al.* (2012) wax crystals are not completely dissolved when the oil is heated to a temperature lower than the critical initial value, but the remaining crystals are not large enough to modify the oil viscosity above the crystallization temperature. However, when the crystal chains are formed by the new precipitated crystals they are weaker because of the small amount of dissolved wax at the beginning of cooling.

According to the hypothesis of the pour point depressants influence, if one heats the crude oil to a high enough temperature, the initial cooling temperature would not interfere in the mechanical properties of the oil. In other words, above a determined temperature the variation of the initial cooling condition would not have influence on the gel strength of the oil at 4 °C. The influence of T_i higher than 60 °C is now investigated.

With exception to initial cooling temperature, the same test conditions proposed elsewhere (Andrade *et al.* 2015) was used in the current work (i.e., T_f= 4 °C, $\dot{T}$ = 1 K/min, t_{ag}= 10 minutes and an oscillatory stress amplitude sweep to determine the yield stress). The initial cooling temperatures evaluated were 30, 45, 60, 75 and 90 °C. One can note that the initial cooling temperatures that have been chosen were the value of the WAT (30 °C) and temperatures much higher than the WAT.

The influence of T_i on the gelation temperature, T_g, are shown in Fig. 3(a). The points shown are average values obtained from test results measured three times and the error bars represent the maximum and minimum values in each test condition. In the current work the gelation temperature was determined by using oscillatory tests during the cooling. At high temperatures the material presents dominant viscous behavior – in this case the loss modulus, G'', is higher than the storage modulus, G'. Both moduli increase with the temperature reduction and at a certain temperature, G' starts growing faster than G''. When G' exceeds G'' at a lower temperature the material behavior changes from viscous to predominantly elastic. The temperature where the G'-G'' crossover takes place is assumed to be the gelation temperature.

One can see in Fig. 3(a) that to the case where a cooling rate of 1 K/min was used, there is a critical value, 60 °C, for the initial cooling temperature in which the T_g reaches the maximum value. It can be noted that even for T_i much higher than the WAT the initial cooling temperature affects the gelation of the oil. For instance, for T_i= 60 °C the gelation temperature is 22 °C, whereas for T_i= 90 °C the material gels at 10 °C. The influence of the initial cooling temperature is even more emphasized when the value of the storage modulus, G', in the end of the cooling, at 4 °C, is evaluated (Fig. 3(b)). It can be observed that for T_i higher or lower than 45 °C the G' is lower than the value measured for T_i= 45 °C. Besides, G' obtained for T_i= 90 °C is three orders of magnitude smaller than that observed for T_i= 45 °C.

Figure 4 shows the influence of the initial cooling temperature on the yield stress of the waxy crude oil at 4 °C. As observed for the storage modulus, the critical temperature when τ_0 is analyzed was also 45 °C. One can observe that the yield stress varies from 1000 Pa for *Ti*= 45 °C to 50 Pa for *Ti*= 90 °C. The most interesting result is observed when the tendency of the results presented for $\dot{T}$ = 1 K/min in Fig. 3 (a), Fig. 3(b) and Fig. 4 are compared. It is possible to conclude that to this waxy crude oil the materials properties at low temperature are always dependent of the initial cooling temperature. In other words, there is not a high enough temperature that "erases" the thermal history of the material.

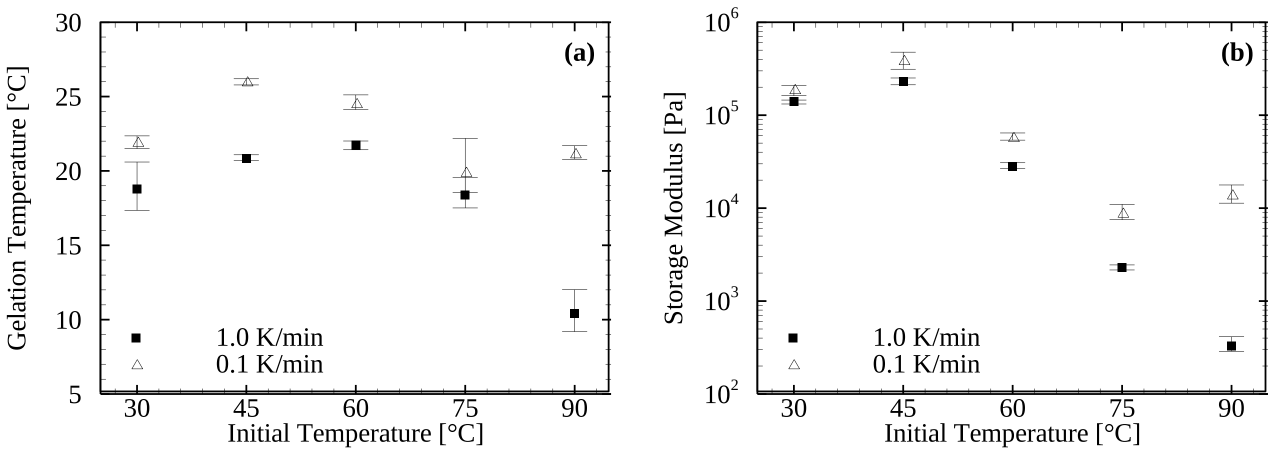

Figure 3 Effect of the initial cooling temperature on the (a) Gelation temperature and (b) Storage Modulus in the end of the cooling, at 4 °C, of the waxy crude oil.

In order to determine if the magnitude of the cooling rate affects the influence of T_i on the gel strength of the material, a slower cooling rate was used, $\dot{T}$ = 0.1 K/min. The idea was to cool the material so slowly that in every temperature during the cooling the specimen would be close to the thermodynamic equilibrium. When the results obtained for $\dot{T}$ = 0.1 K/min are compared, hollowed symbols in Fig. 3 (a), Fig. 3(b) and Fig. 4, one can observe that the influence of T_i on the properties of the crude oil is reduced. But even for slower rate of cooling, T_i affects the mechanical behavior of the crude oil at low temperature.

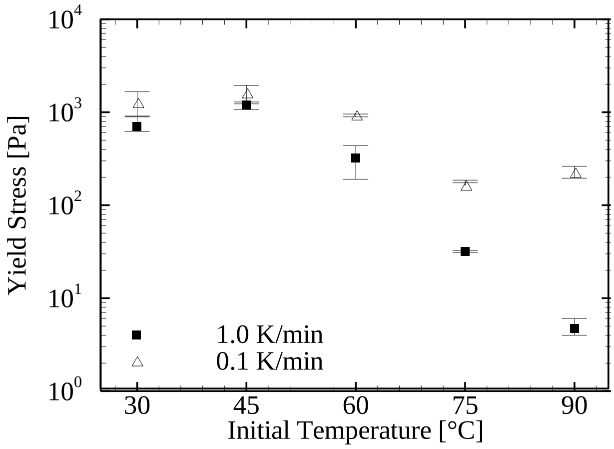

Figure 4 Yield stress at 4 °C as a function of the initial cooling temperature

3.2. Effect of Aging Time

In the previous section a fixed aging time, t_{ag}= 10 minutes, was used to keep the same tests conditions used elsewhere (Andrade *et al*. 2015). In that work, some tests were performed with different aging times, but its influence on the gel strength was not deep investigated. In this current section the evaluation of the influence of aging time on the gel strength of the wax crude oil is performed.

Figure 5 presents the variation of G' and G'' along the time of aging at 4 °C. The result observed in Fig. 5 was obtained for T_i= 45 °C, $\dot{T}$ = 1 K/min and T_f= 4 °C. One can note that in the first hour of rest both G' and G'' increase much faster than in the second hour. After three hours of aging time both moduli practically reach the final steady state value. Table 2 shows the yield stress measured after different aging times. The pretest was the same of the used in the test presented in Fig. 5, and a stress loading rate, $\dot{\tau}$ =5000 Pa/min, was performed to evaluate the yield stress.

One can note in Tab. 2 that the yield stress increases substantially with the aging time until t_{ag}=120 min. Table 3 shows the variation of G', G'' and τ_0 in some evaluated period of time. As can be seen the variation of τ_0 between t_{ag}= 2 and 3 hour was less than 5%. The same was observed for G' and G''. It is worth of noting that the variations of both moduli in the first hour were much higher than the variation of the yield stress in the same period of time. On the other hand, not only the storage and loss moduli reached the steady state value after two hours of aging but also the yield stress. Because of that, from now on, the aging time used in the current work will be t_{ag}= 120 minutes.

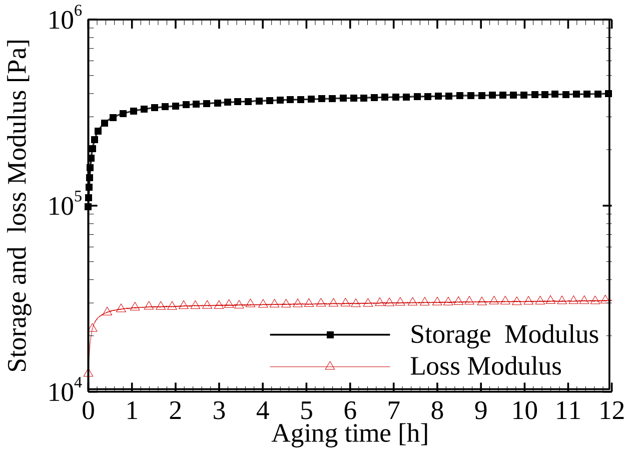

Figure 5 G′ and G″ at 4 °C as a function of aging time after a static cooling

Table 2. Influence of the aging time on the yield stress

Aging time [min]	Yield Stress [Pa]	Standard Deviation [Pa]
0	3325	84.7
1	3510	218.7
10	4036	219.3
60	4185	65.8
120	4717	818.9
180	4913	740.5

Table 3. Variation of the storage modulus (G'), loss modulus (G'') and yield stress (τ_0) along the aging time

Evaluated Period of Time	$\Delta G'$ [%]	$\Delta G''$ [%]	$\Delta\tau_0$ [%]
0 and 1h	226.5	125.5	25.9
1h and 2h	7.0	2.5	12.7
2h and 3h	4.0	1.3	4.2
3h and 4h	2.5	0.5	-
4h and 5h	1.7	0.4	-

3.3. Effect of Shear Loading Stress

Despite the different methods employed to evaluate the yield stress (Balmforth *et al*. 2014, Bonn *et al.* 2015) in the current section a stress loading rate was used to determine τ_0. It was evaluated the influence of the stress loading rate used to break down the gel. Three different values of $\dot{\tau}$ were evaluated, 50, 500 and 5000 Pa/min. The pretest performed for these tests was: T_i= 45 °C, $\dot{T}$ = 1 K/min, T_f= 4 °C and t_{ag}= 120 minutes. Figure 6 presents the shear strain as a function of shear stress for the three stress loading rate tests. One can observe that small strains were noted in the predominantly elastic region, to low values of shear stress, and at a certain shear stress value the gel was broken – in that point the strain increased abruptly.

It can be noted on Fig. 6 that the higher the $\dot{\tau}$ the higher the measured yield stress. For instance, τ_0=3480 Pa for $\dot{\tau}$ =50 Pa/min and 4717 Pa for 5000 Pa/min. It is worth of noting that, as observed by Hou (2012) and Tarcha *et al.* (2015), although the conditions of the tests, as $\dot{\tau}$, affects the measured yield stress, the yield strain, γ_c, was almost the same for all the cases, for these tests, γ_c= 0.2 as shown by the red dashed line.

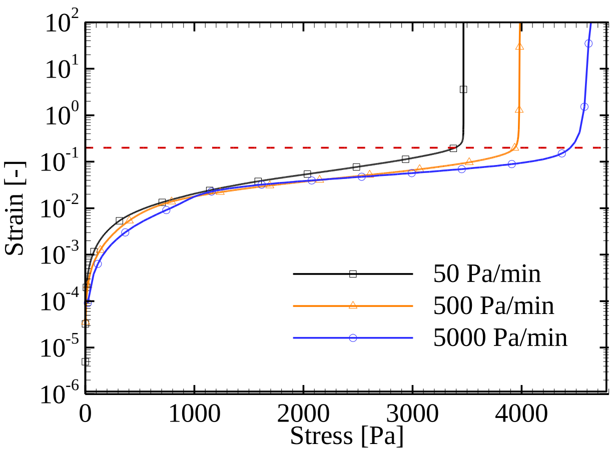

Figure 6 Shear strain as a function of shear stress for stress loading rate tests performed at 4 C.

3.4. Effect of Final Temperature

It is well known in the literature that the lower the temperature the higher the viscosity, the storage and loss moduli, and the yield stress of the wax crude oil (Hou and Zhang, 2010; Dimitriou et al. 2011; Hou, 2012; Lin *et al.* 2012; Zhao *et al.* 2012; Andrade *et al*. 2013; Teng and Zhang 2013; Mendes *et al*. 2015). The rise of such properties was attributed to wax solubility reduction in oil as the temperature decreases (Huang *et al*., 2011; Coto *et al*., 2011). In the current section the influence of the initial and final cooling temperature mainly on the yield strain was evaluated. Eight tests conditions were evaluated, four T_i=45, 60, 75 and 90 °C, and two final temperatures, 4 and 10 °C. Other conditions of the test were kept constant: i.e., $\dot{T}$ = 1 K/min, t_{ag}= 120 minutes and $\dot{\tau}$ =50 Pa/min.

Figure 7 presents the shear strains as a function of the shear stress for stress loading rate tests performed. First of all, it is important to note that even for a high value of aging time (i.e., t_{ag}= 120 minutes) the initial cooling temperature has a great influence on the yield stress. As expected the lower the final temperature of the cooling the higher the yield stress of the material. For example, for T_i=45 °C the yield stress increased from 2000 to 3480 Pa when the final temperature was decrease from 10 to 4 °C. On the other hand, Hou (2012) stated that the lower the final temperature the lower the yield strain for a Daqing waxy crude oil. For instance, the author noted that the yield strain increased from 0.1 to 1 increasing just 4 °C in the final temperature. One can observe in Fig. 7, as shown by the red dashed line, that in the current work the measured yield strain was kept almost constant, γ_c= 0.2, independently of the final temperature.

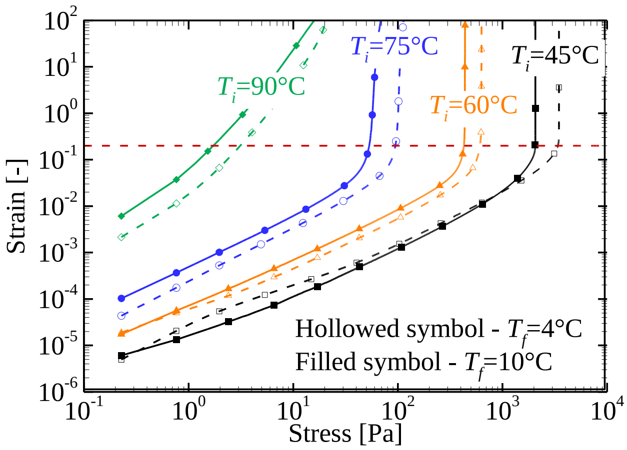

Figure 7 Shear strain as a function of shear stress for stress loading rate tests performed at 4 and 10°C, four T_i different were evaluated.

4. CONCLUSIONS

Thermal and shear histories have a great influence on the gel strength of waxy crude oil at low temperatures. In the current work it was analyzed the influence of different test conditions on the gel strength of the material. The main results are summarized as:

i) The initial cooling temperature affects the mechanical behavior of the material. It was noted that even temperatures much higher than the Wax Appearance Temperature (WAT) still affect the gel strength of the material when it was cooled to the final temperature. In order words, it was not possible to “erase” the thermal history of the material.

ii) The aging time at the final temperature affects the yield stress of the material until a certain value of time at rest. After two hour of aging time the yield stress, storage and loss moduli reached a steady state value. Then above two hour of aging the gel strength of the material do not change.

iii) The condition of the stress loading rate test affects the measure yield stress of the material. The higher the stress loading rate the higher the measure yield stress. On the other hand, the yield strain was kept constant independently of the test condition.

iv) The lower the final temperature the higher the yield stress of the material. The yield strain is kept constant even for different values of final temperature.

5. ACKNOWLEDGEMENTS

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