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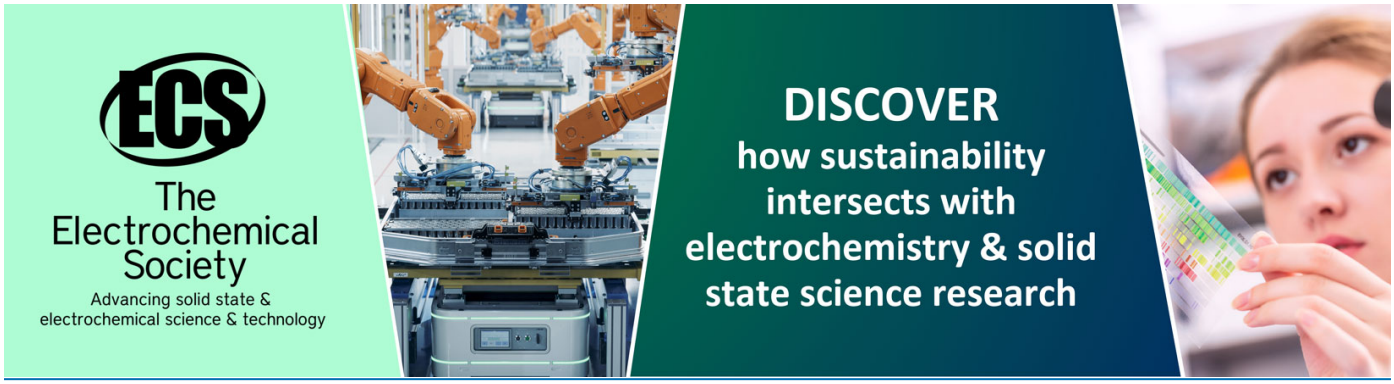

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Influence of extreme low temperature conditions on the dynamic mechanical properties of carbon fiber reinforced polymers

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Abstract. In the current study dynamic mechanical analysis (DMA) is performed in CFRPs that have been exposed for certain periods of time to extreme low temperatures. Through experimental data arising from respective DMA tests the influence of low temperature exposure (-40 ^{o}C) on the dynamic mechanical properties is studied. DMA tests were conducted in CFRP specimens in three point bending mode at both frequency and thermal scans in order to determine the viscoelastic response of the material in low temperatures. All experimental tests were run both for aged and pristine materials for comparison purposes. The results occurred reveal that there is deterioration both on transition temperature (Tg) and storage modulus values while there is also a moderate increase in the damping ability of the tested material as expressed by the factor tanδ as the period of exposure to low temperature increases.

1. Introduction

Carbon fiber reinforced polymer composites (CFRPs) mainly based on thermoset polymers are being increasingly used in a variety of modern engineering applications such as aviation and space industry or in offshore infrastructures because of their advantage to high stiffness and strength values over weight ratio, corrosion resistance and ease of fabrication.

However, as the use of composite materials expands into the field of oil pipeline networks and aircraft transportation, their components are subjected to more hostile environments, including exposure to extreme low or cryogenic temperature conditions.

In these conditions, such as very low temperatures and moisture environment, the performance of the CFRPs still remains under consideration and extensive study [1 - 4]. Thus, it is crucial to understand the response of advanced composite materials to such conditions in order to produce a well-designed, structurally efficient component using the minimum amount of material and understanding of how such materials behave at cryogenic temperatures.

Under service conditions, CFRP bars are subjected to a wide range of very low temperatures. The exposure to these temperatures may have radical effects on fiber-matrix bond deterioration and matrix cracking compared to any exposure. Sub-zero temperatures can cause changes in mechanical properties and create additional microcracks in CFRP materials.

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Additionally at lower temperatures microcracking can further result in the increase of water absorption at higher temperatures, leading to an increased matrix plasticization or delaminations. Frozen water present in cracks and voids may also lead in debonding and transverse microcrack growth resulting in material degradation, increased brittleness and changes in damage mechanisms. The effect of low-temperature thermal cycling on mechanical properties of carbon-vinylester composites and E-glass/vinyl ester composites has been investigated by a number of researchers [5-10]. In their studies they pointed out that freeze-thaw events could cause significant degradation of the mechanical properties and glass-transition temperatures of the FRP specimens immersed in an aqueous environment.

In order to predict the mechanical response of the material in these environments it is very important to know their performance for longer periods of time, thus the viscoelastic properties must be determined. To the author's knowledge limited or no data exist for the dynamic viscoelastic response of CFRPs under severe low temperature conditions.

The study aims to determine the effect of extreme low temperature in the dynamic viscoelastic properties of CFRPs and the change in their rheological behavior through the technique of dynamic mechanical analysis.

2. Theoretical background

Dynamic mechanical analysis has been used extensively in literature to study and characterize materials, mainly polymers and composites, that exhibit time dependent behaviour. Through a sinusoidal stress applied to the specimen, the strain of the material is measured, allowing thus to determine the storage modulus E', the loss modulus E'', tanδ and the glass transition temperature Tg of the material as well as to identify transitions corresponding to other molecular motions by varying the temperature or the frequency of the applied stress.

In a more extensive manner, by observing that the creep compliance vs. log (time) or log (frequency) curves for different temperatures retain the same shape, a technique of shifting these curves and superimposing them to a unique reference master curve can be applied [11-13]. This is the Time Temperature Superposition Principle (TTSP) that has been extensively used in literature [14-20] for polymers and composite materials.

In general, the steps followed when TTSP is applied so as to generate a creep master curve can be described by the methodology below.

- A material specimen is subjected to a constant load at a pre-set temperature level as in conventional creep testing, and the evolution of the creep strain of the specimen is recorded vs the log (time).
- Parallel experiments are performed for different specimens or even the same specimen, in modern DMA equipment, at different temperature levels and the relevant creep curves are obtained.
- A desired reference temperature is selected (TR).
- All the individual creep curves corresponding to different temperature levels are shifted along the log (time) scale to be superimposed to a master or mother curve.

If it is possible to generate a smooth master curve by applying a horizontal shift along the log (time or temperature) axis, the material can be then characterized as a thermorheologically simple material (TSM). However, for some materials a vertical shift factor may be needed to obtain a smooth master curve; they are then classified as thermorheologically complex materials (TCM).

TTSP has been used for polymeric materials, as it is governed by the solid rules of linearity and superposition that are applied in order to obtain the master curves. Usually, TTSP results exhibit the success of the method. Apart from polymer composites of various types [21], it has been successfully applied by researchers to investigate a variety of materials when time-related or temperature-activated procedures are considered. For example, it has been successfully employed for the characterization of asphalt concrete [22], viscous metallic liquids [23], solid rocket propellants [24] and wood [25].

3. Experimental procedure

3.1. Materials and manufacturing

Manufacturing of composite laminates was performed by Vacuum Assisted Resin Infusion Molding (VARIM) (Figure 1). Stacked woven carbon fiber layers, were cured for 12h at room temperature. The carbon fabric used was a C400P (TR50S) Plain Weave, (E= 240 GPa, σ_{ult} =4.9 GPa). Resin type was R2940/Infusion RTM (E=3.6 GPa, $\sigma_{ult} \approx$ 70-85 MPa, Tg ≈ 67-80 ^{o}C). Both room temperature curing resin and carbon fabric were supplied by FIBERMAX Composites Ltd. The fabric was hand laid in 6 layers of a stacking sequence $[(0/90)_2/\pm45]_s$ resulting in quasi isotropic Plates of 400x400x2.50 mm^3. The layers were vacuum-bagged and readily mixed resin/accelerator mixture infiltrated them by a flexible tube placed in one side of the vacuum bag. Specimens prior and after low temperature exposure were cut from these plaques by a diamond saw blade and polished with polishing paper in order to eliminate surface microcracks and local heterogeneities (Figure 2).

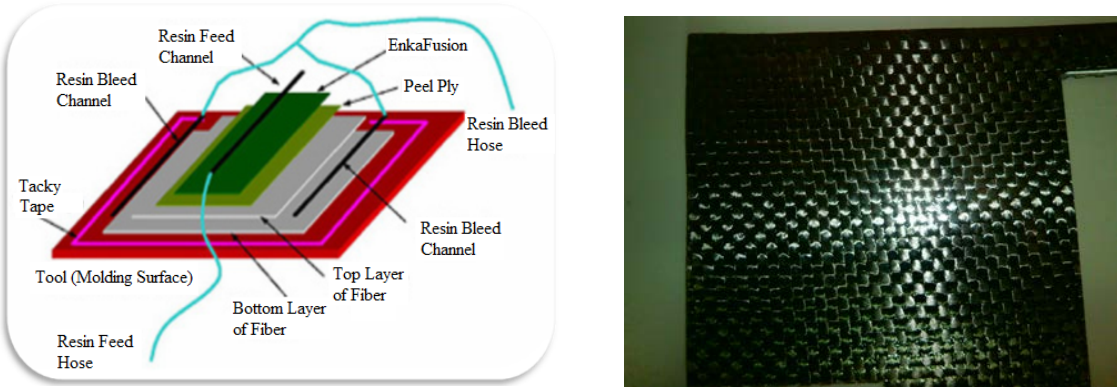


Figure 1. Schematic representation of the Vacuum Assisted Resin Infusion Molding technique used for the fabrication of the composite plates.

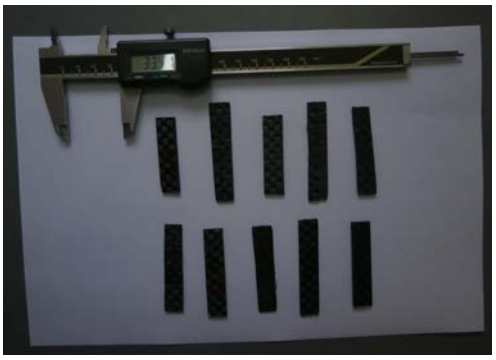


Figure 2. Specimens of CFRPs used for mechanical testing.

3.2. Composite plaques freezing procedure

In order to investigate the effect of low temperature on composites an experimental program applied included exposure of the composite plaques in a climatic chamber Instron mod.3119-409 (High Wycombe, UK) at -40 ^{o}C for three different periods of time, namely 30, 45 and 60 days. Composite plaques were then immediately afterwards removed and placed in a dry air freezer for 8 hours.

3.3. Testing

Three point bending testing (3PB) was performed on an Instron 3382 Universal Testing Machine as per EN ISO 178:1996, equipped with a specially manufactured high-stiffness bending rig (Figure 3). Specimens' nominal dimensions were Length = 50 mm, Width = 12 mm, Thickness = 2.50 mm and

crosshead speed of the three point bending test was set at 2 mm/min. Support span was 40mm. All specimens were set in isothermal conditions prior to testing in order to avoid discrepancies occurring from the variation of temperature. Five specimens per sample were used for property averaging.

A similar specimen (50 mmx12 mmx2.5 mm) was used for the dynamic mechanical analysis (DMA) in three point bending mode on a DMA Q800 system of TA Instruments (Figure 4). Temperature scans were performed at a frequency of 1 Hz while the temperature was varied from T = 25-150 ^{o}C and heat rate was set at dT/dt =3 ^{o}C/min. Multi-frequency tests were performed at a range of 1 to 200 Hz and a temperature that varied from T = 25-150 ^{o}C.

Figure 3. Three point bending tests procedure in Instron 3382 UTM.

Figure 4. The type of the three point bending clamp used in mechanical testing in TA Q800 DMA.

4. Results and discussion

4.1. Three point bending static tests

Three point bending strength and modulus values determined from respective three point bending tests deteriorate as freezing time increases as seen in Table 1. This is a result confirming the general trend that environmental factors such as alternating temperature and humidity, accounts for mechanical property loss.

Table 1. Flexural properties for all composite specimens

Specimen Type/ Aging Procedure	Bending Modulus [GPa]	Bending Strength [MPa]
Pristine Specimens	$21.32^{\pm 4.3}$	$693.15^{\pm 78}$
30 days at -40^{o}C	$18.16^{\pm 3.5}$	$657.31^{\pm 59}$
45 days at -40^{o}C	$14.20^{\pm 2.7.}$	$614.46^{\pm 61}$
60 days at -40^{o}C	$12.87^{\pm 2.1}$	$579.17^{\pm 69}$

4.2. Dynamic mechanical analysis tests-Temperature dependence

DMA spectra of dynamic mechanical properties i.e. storage modulus G', loss modulus G'' and tanδ parameter as a function of temperature are shown in Figures5-7 for the carbon fiber epoxy specimens. As it can be observed, the effect of the period of freezing temperature of -40^{o}C on the values of the dynamic properties is obvious. Dynamic storage modulus (Figure 5) tends to decrease with rising testing temperature while its value drops by almost 5000 MPa confirming the results from static three point bending testing. A typical sigmoidal curve is observed in all cases. So, the tested CFRP exhibits a common "viscoelastic" time-temperature dependent response. The glass transition temperature (T_g), as it may be determined from the onset of the slope of the Dynamic Storage Modulus E'-Temperature T curve, also deteriorates.

Damping coefficient as expressed by the tanδ parameter appears to be increased with the variation of temperature (Figure 7). At the same time, an increase of the period of freezing temperature shifts the values of Tg at a lower level verifying the trend in the plots of Dynamic Storage modulus E'.

This is an important finding implying that the material loses part of its ability for energy distortion however gains in capability in vibrational dumping, which is a positive effect in the design of composites structures.

The mean shift of Tg from 0 days to 60 days exposure at -40 ºC, is about -10 ºC (from ~84 to ~74 ºC) which is also an important part of restraint of the service temperature of a composite thus reducing his use capability.

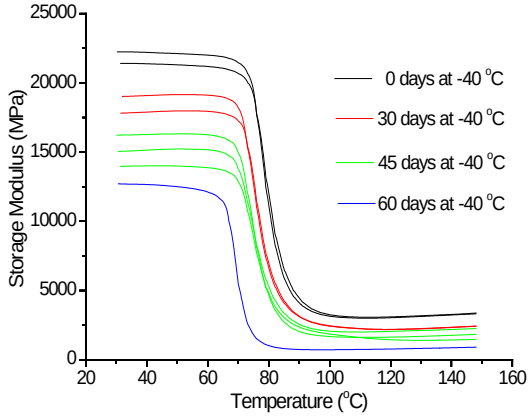


Figure 5. Storage modulus G' as a function of temperature and frequency.

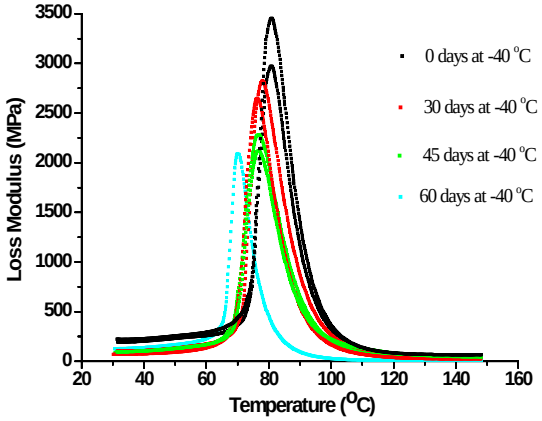


Figure 6. Loss modulus G'' as a function of temperature and frequency.

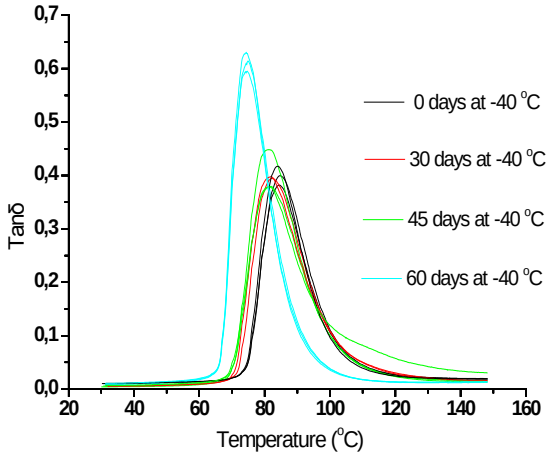


Figure 7. Tanδ as a function of temperature and frequency.

4.3. Dynamic mechanical analysis tests-Frequency dependence

Plots of storage modulus, loss modulus and tanδ for the three tested loading scenarios versus frequency occurring from scans of 35 ºC up to 150 ºC temperature range are appended in Figures 8-10. The curves that are to be superimposed are depicted in Figures 8a, 9a, 10a and 11a (in Appendix A), while the respective master curves are shown in Figures 8b, 9b, 10b, and 11b. It should be noted that all shifts in the curves that were to be superimposed in Figures 9a-11a were performed with respect to a reference curve that was experimentally determined at a temperature of 150 ºC.

A comparison of the evolution of storage modulus versus frequency for specimens that have been exposed at -40 ºC for three different periods of time is shown in Figure 12. As expected, an increase of the storage modulus as frequency per decade increases is observed, however, a tendency for decrease in the values of Storage Modulus as the exposure time to freeze increases is also obvious.

Similar behavior for loss modulus versus frequency is also depicted in Figure 13. The values of Loss Modulus tend to lower values as the composite remains for longer times in the reference freezing temperature. Finally, a view of the tanδ values is shown in Figure 14 confirming the previous trend of decrease versus freezing temperature. An important point should be also noted concerning the transition range of the stiffening of the material at around 2×10^5 rad/sec.

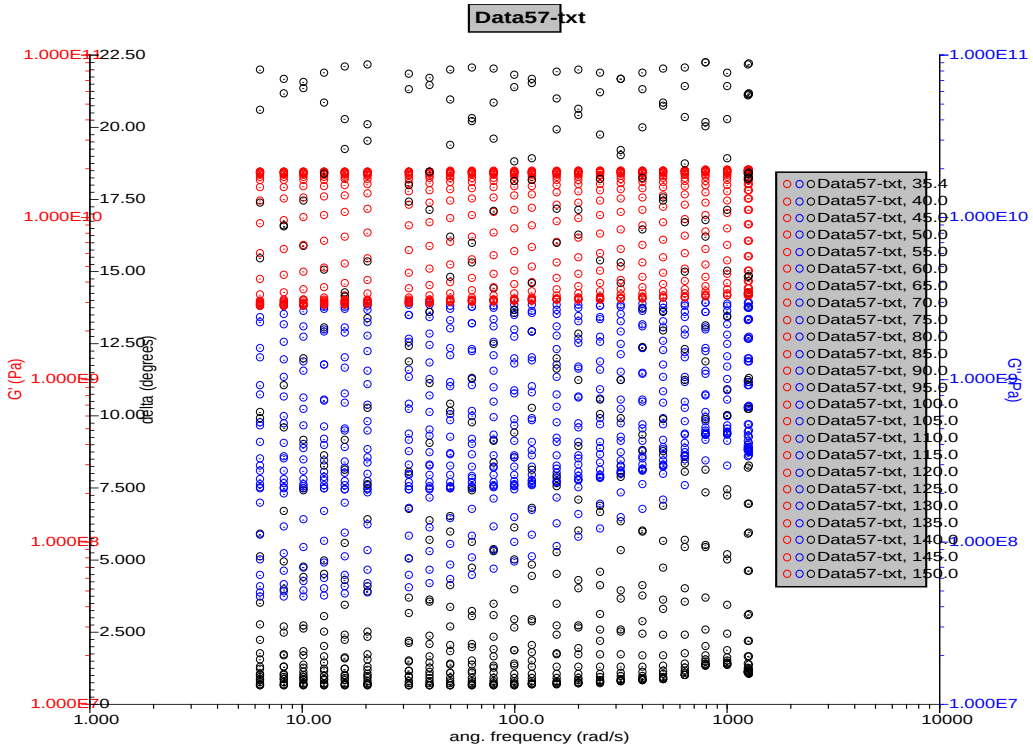
5. Conclusions

The study of the static and dynamic mechanical behavior of a fiber reinforced polymer composite exposed at a temperature of -40 ºC proved the effect of freezing period on the mechanical properties of the material.

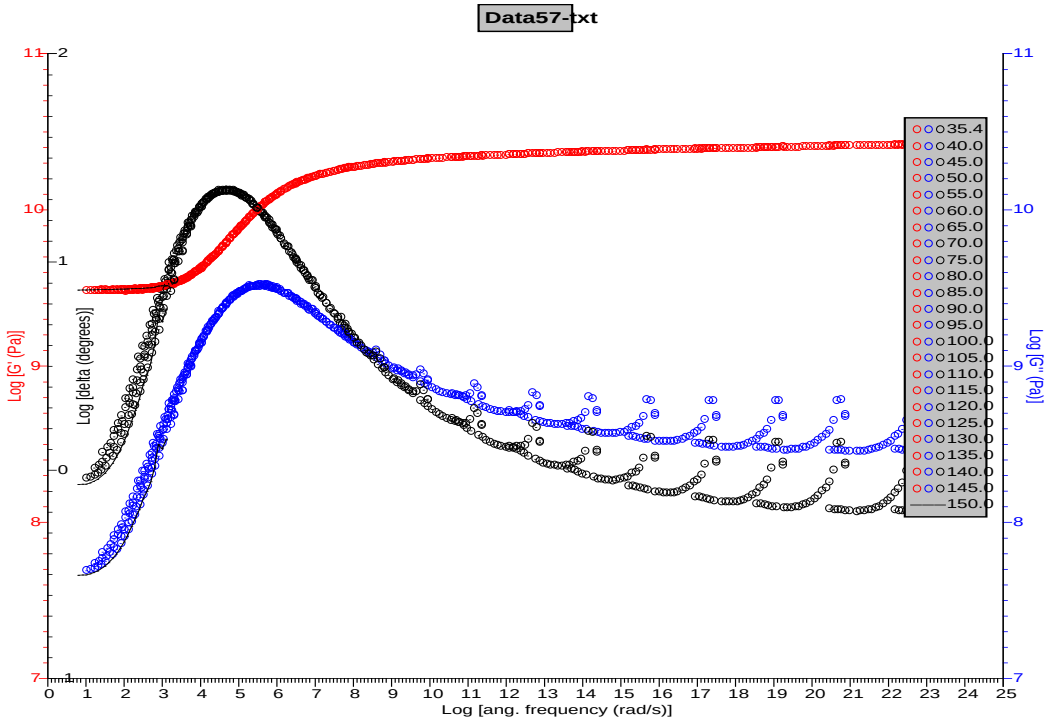
More precisely, deterioration of the values of static bending modulus and bending strength was observed while dynamic storage modulus was also reduced as the time of exposure at the tested low temperature was increased. On the contrary, an increase in the loss modulus versus time of exposure was noted. The above experimental observations were followed as expected by an increase of the dumping factor tanδ.

Glass transition temperature (Tg) was also affected by freezing time, moving to lower values thus limiting the service temperature of the material. Time temperature Superposition principle applied in CFRPs specimens denoted a moderate decrease in storage modulus, loss modulus and tanδ values versus freezing time at longer times, however, glass transition temperature seems not to be affected by this variation.

Appendix A

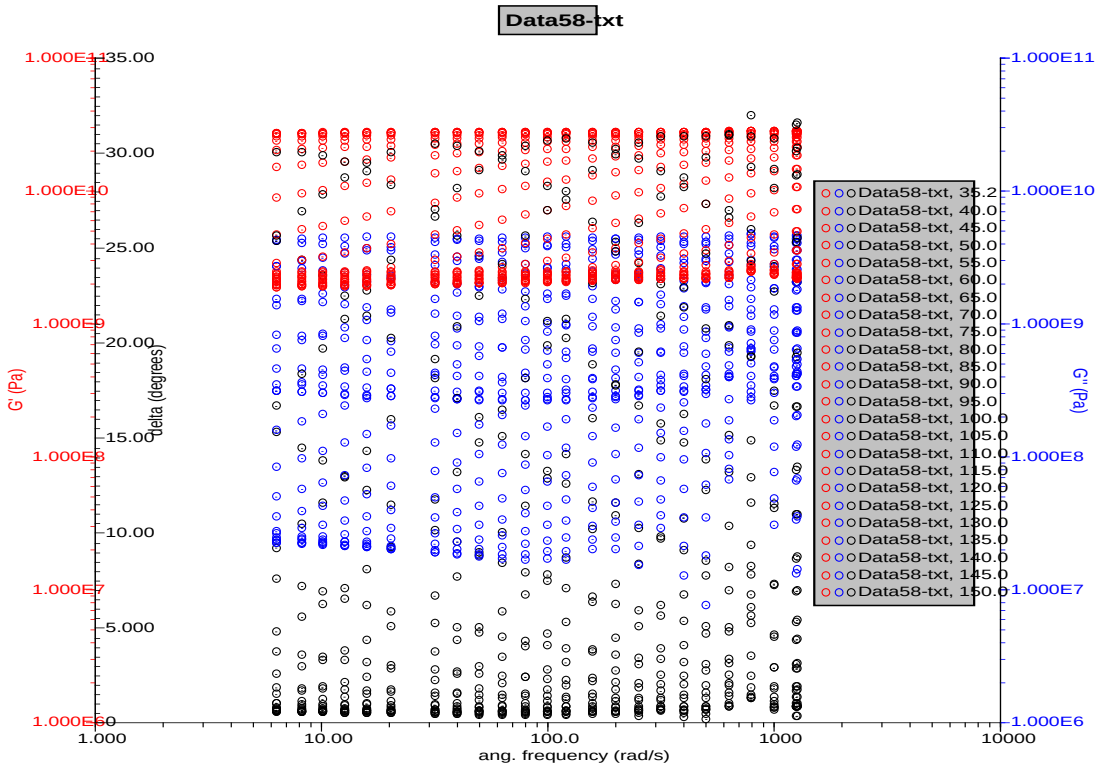


a)



b)

Figure 8. a) G', G'' and tanδ curves versus fequency at different temperatures ranging from 35 to 150 ºC that are to be superimposed and b) Master curves for G', G'' and tanδ as a function of frequency, for neat specimens.



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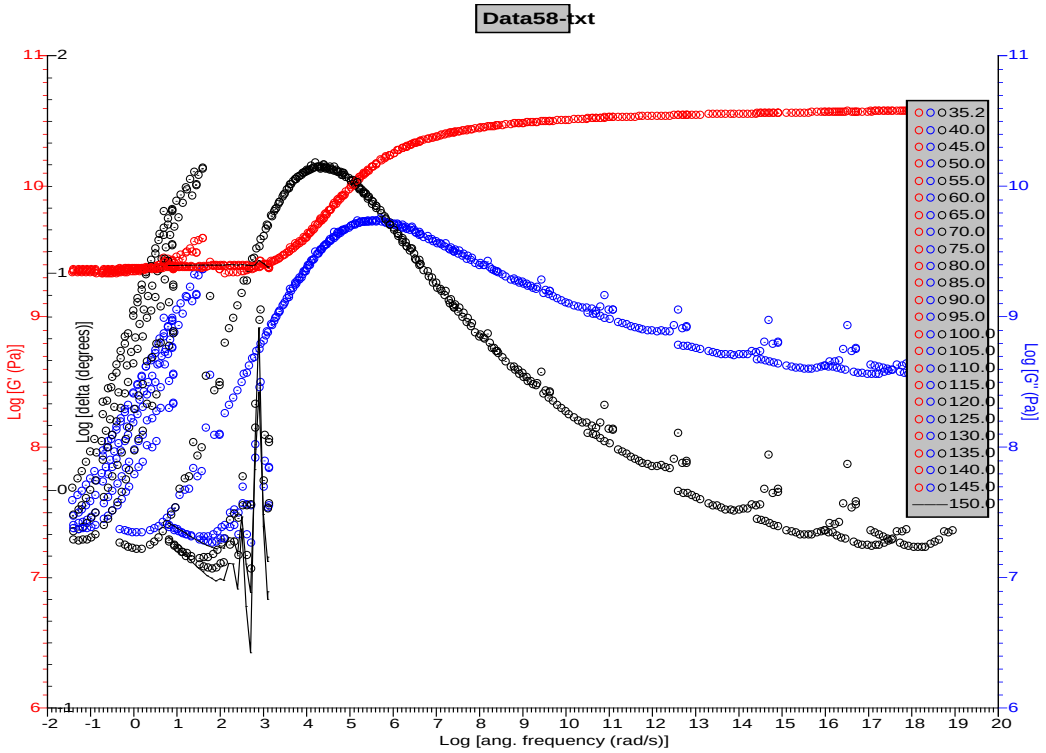
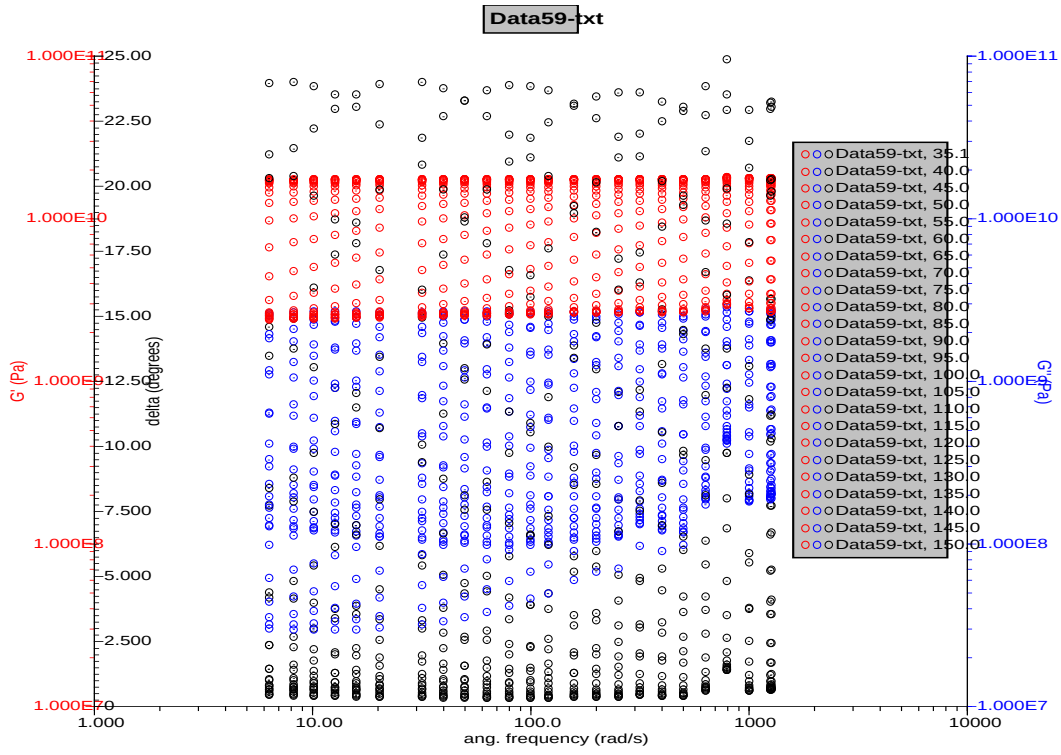
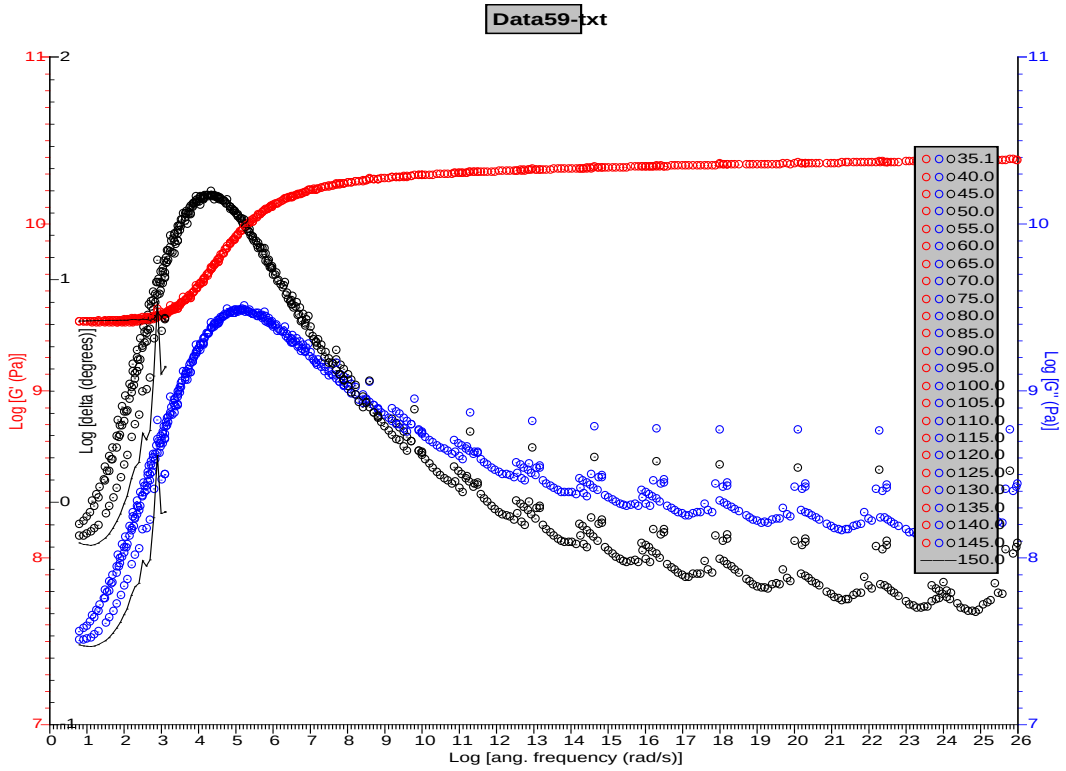


b)

Figure 9. a) G', G'' and tanδ curves versus fequency at different temperatures ranging from 35 to 150 ºC that are to be superimposed and b) Master curves for G', G'' and tanδ as a function of frequency, for specimens for 30 days at -40 ºC.

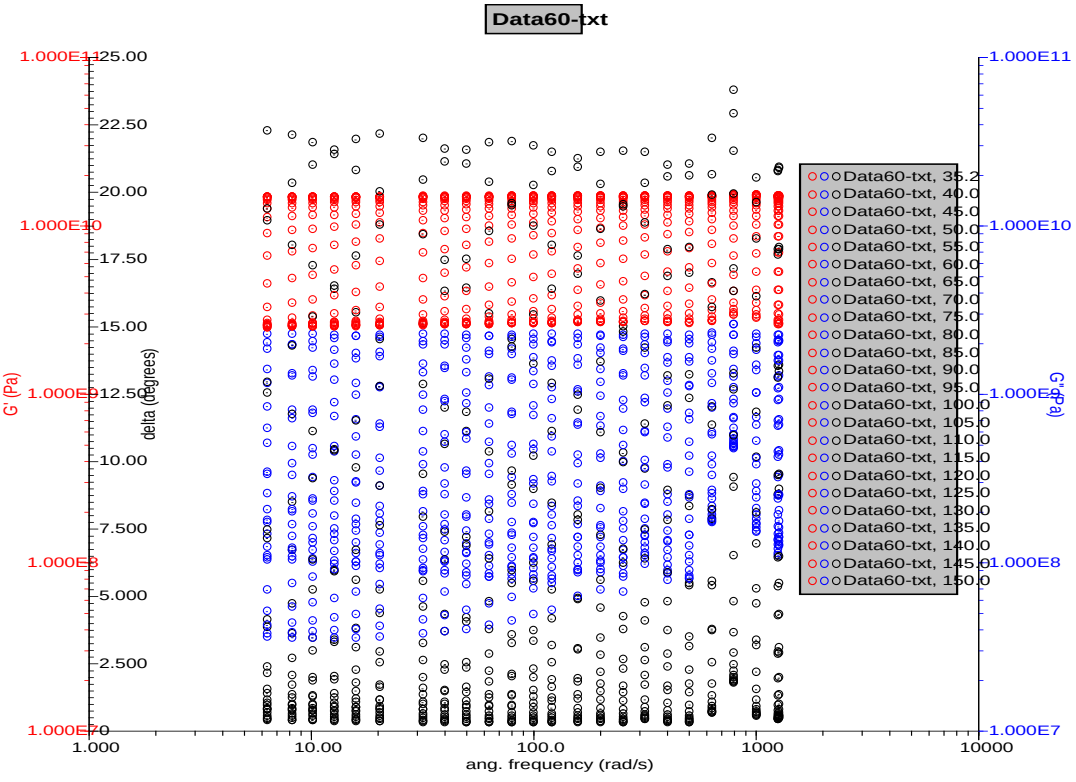


a)

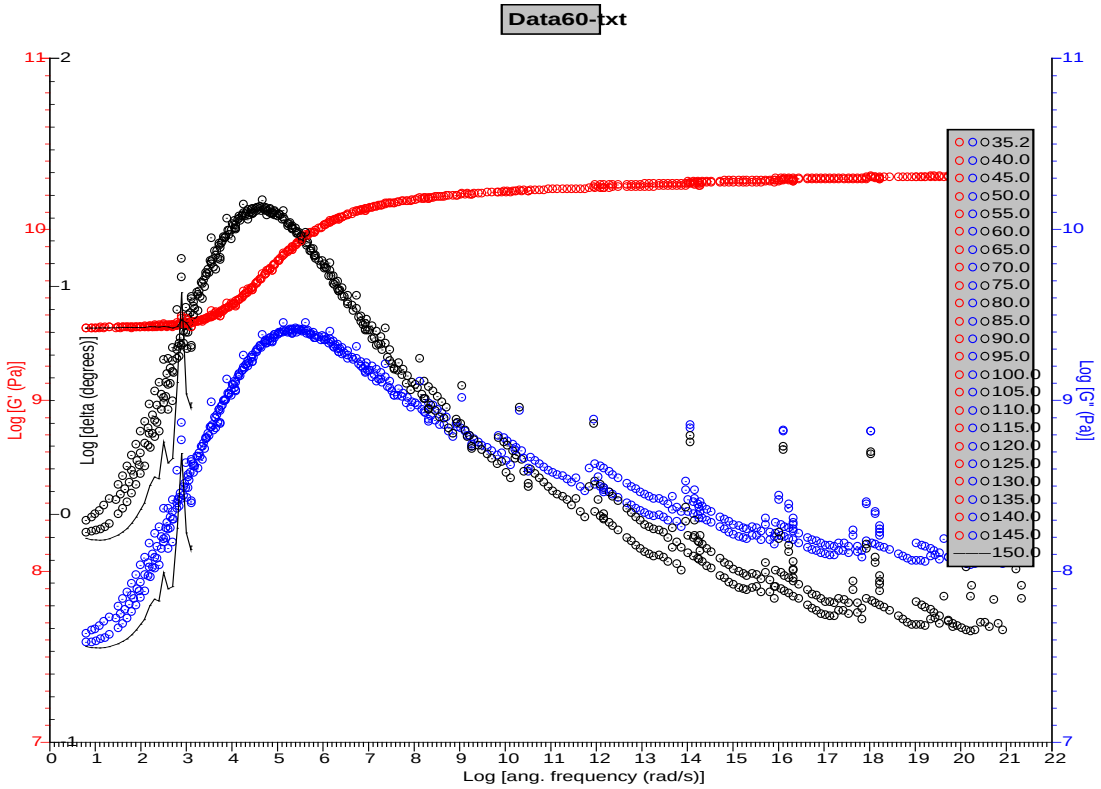


b)

Figure 10. a) G', G'' and tanδ curves versus fequency at different temperatures ranging from 35 to 150 ºC that are to be superimposed and b) Master curves for G', G'' and tanδ as a function of frequency, for specimens for 45 days at -40 ºC.



a)



b)

Figure 11. a) G', G'' and tanδ curves versus fequency at different temperatures ranging from 35 to 150 ºC that are to be superimposed and b) Master curves for G', G'' and tanδ as a function of frequency, for specimens for 60 days at -40 ºC.

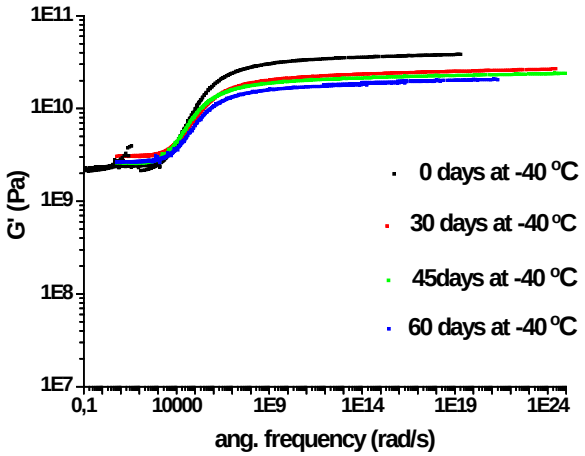


Figure 12. Comparison of superimposed storage modulus as a function of frequency for different exposure periods at -40 ºC.

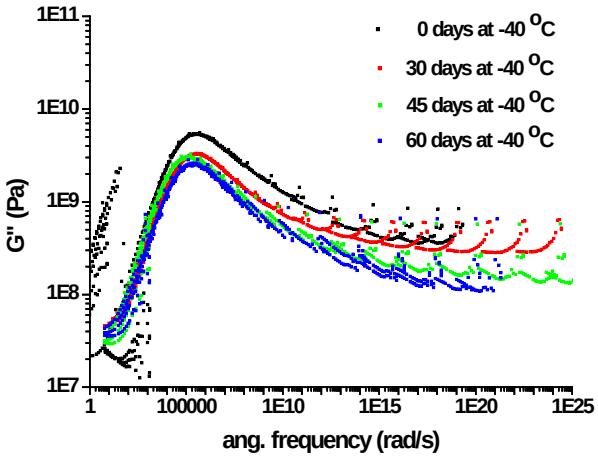


Figure 13. Comparison of superimposed loss modulus as a function of frequency for different exposure periods at -40 °C.

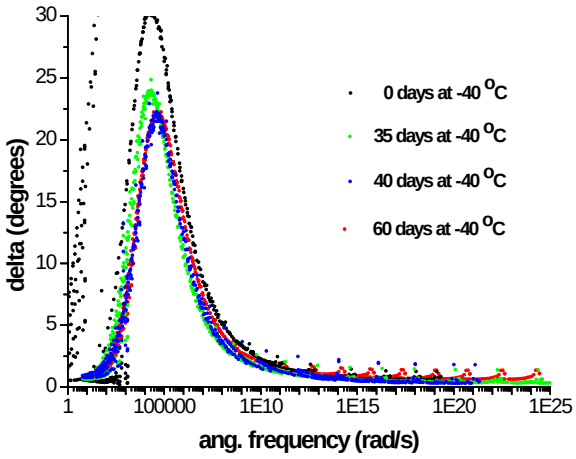


Figure 14. Comparison of superimposed tanδ as a function of frequency for different exposure periods at -40 °C.

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