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Air Force Dynamic Mechanical Analysis of NATO Round Robin Propellant Testing for Development of AOP-4717

23 Sep 2015



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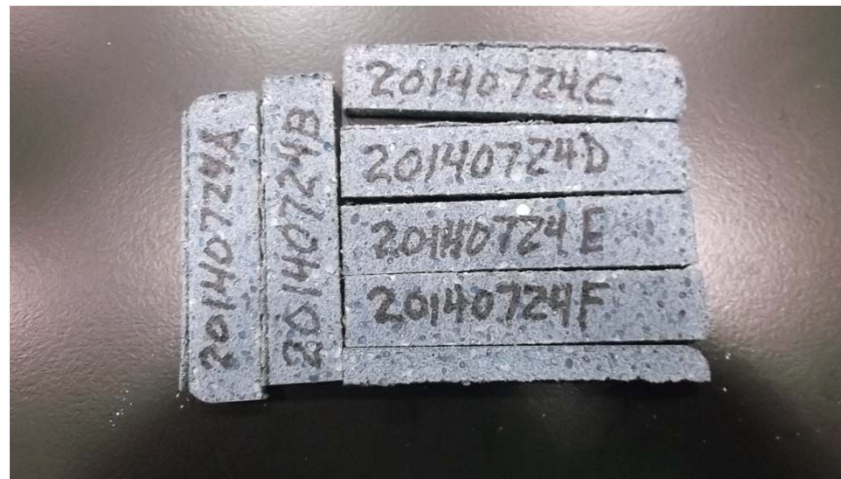
Introduction

Specimen name	Test type	Relative Humidity	Temperature (deg C)
20140724A	Strain sweep	53.5	18.1
20140724B			
20140724C			
20140724D	Frequency sweep	73.3	20.6
20140724E		61.8	21.5
20140724F		52.8	19.6

Specimen name	Width (mm)	Thickness (mm)	Length (mm)
20140724A	12.65	3.99	56.19
20140724B	12.79	4.11	56.37
20140724C	12.75	4.10	64.20
20140724D	12.19	4.12	63.57
20140724E	12.63	4.12	63.95
20140724F	12.98	4.16	63.73



Introduction





Introduction





Strain Sweep Tests

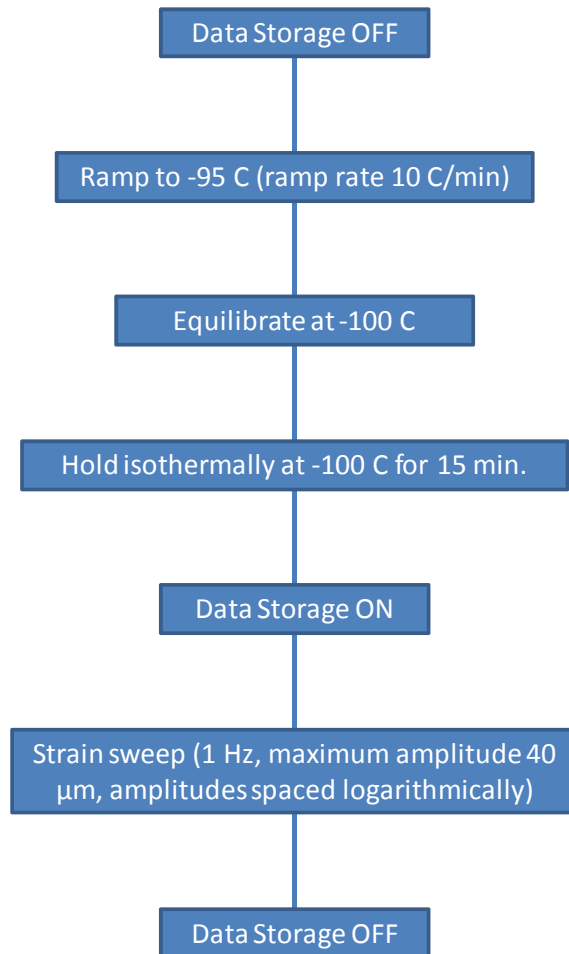
Specimen name	Test temperature (C)
20140724A	-100
20140724B	21
20140724C	70

One test per test temperature. Amplitude varied, frequency always 1 Hz

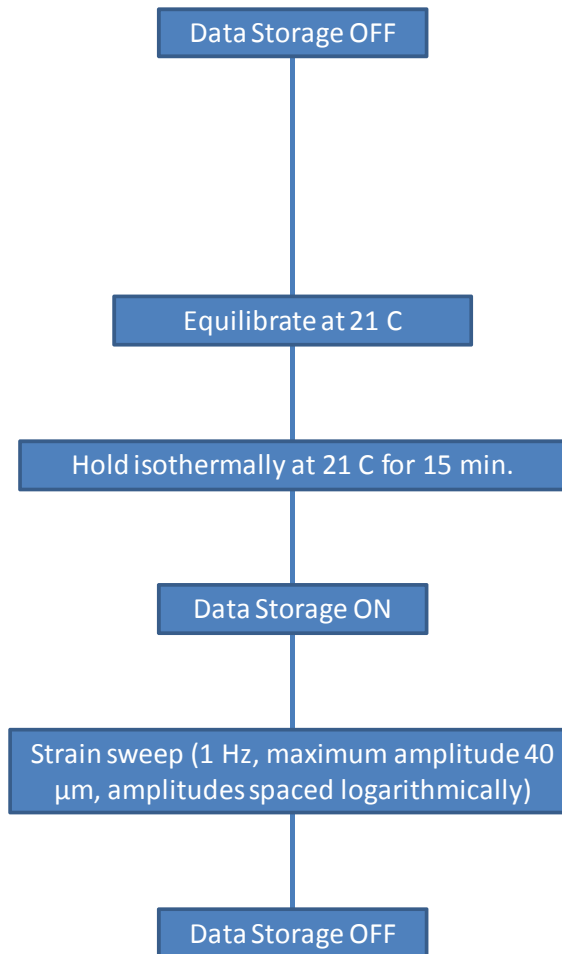


Strain Sweep Tests

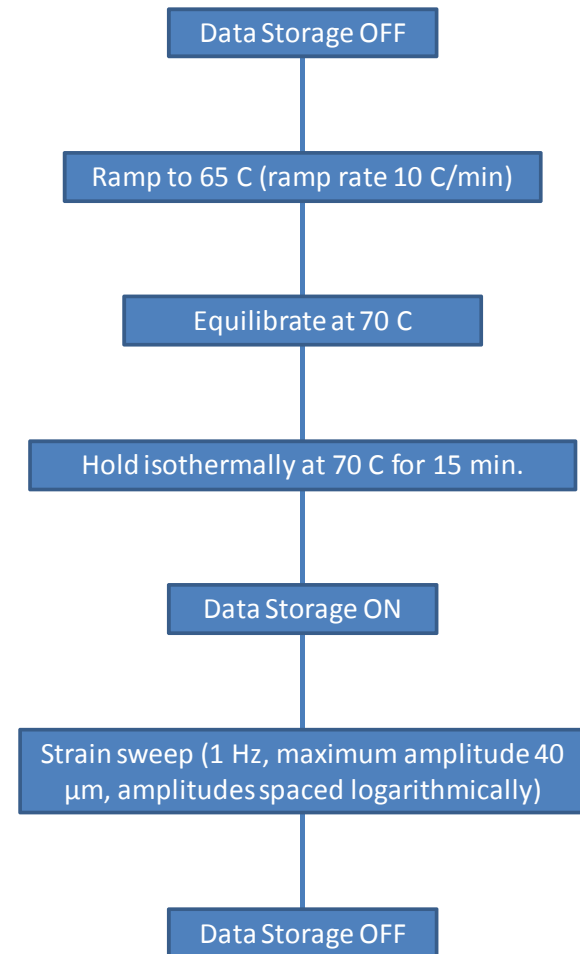
Strain sweep block diagram for -100 C



Strain sweep block diagram for 21 C



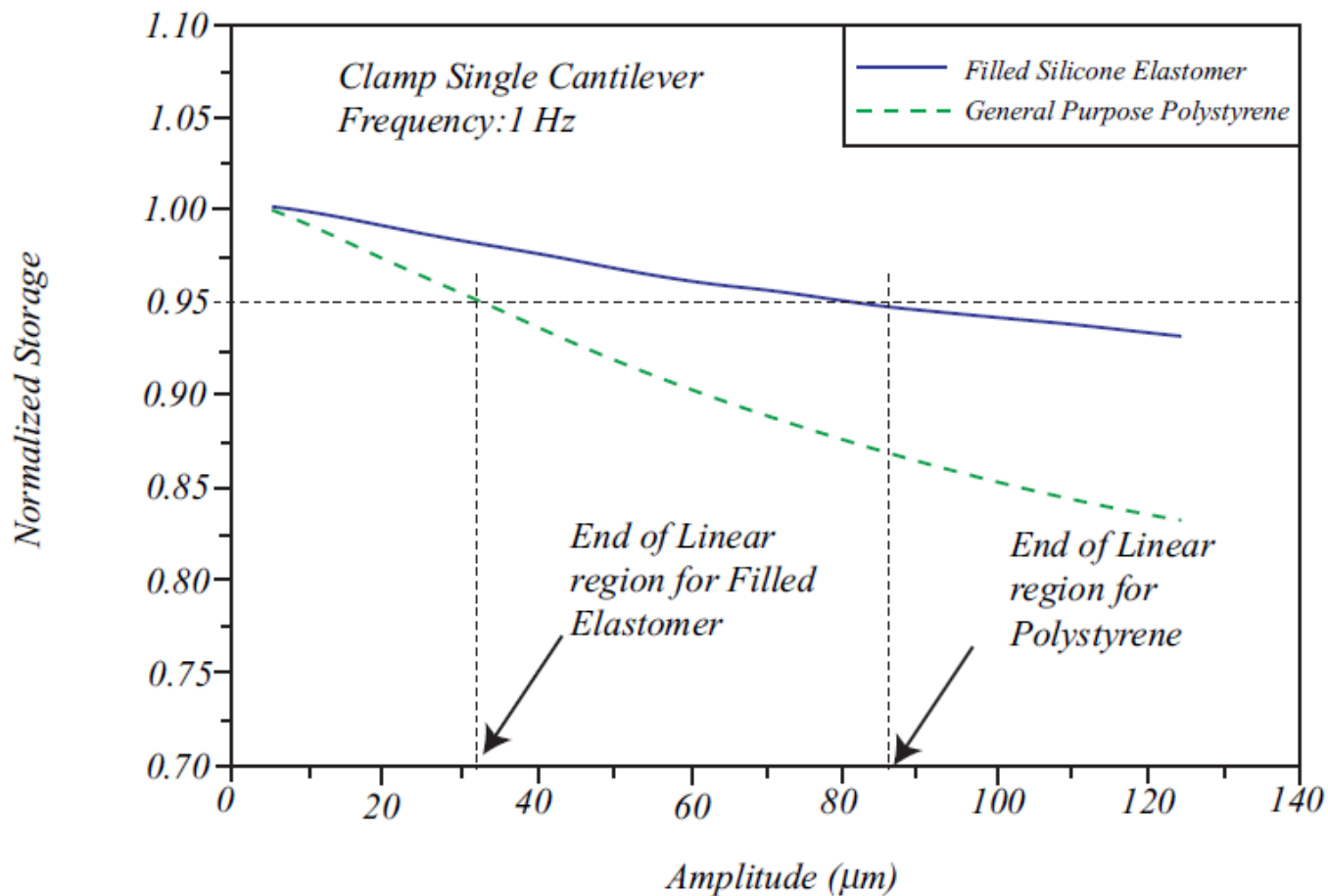
Strain sweep block diagram for 70 C





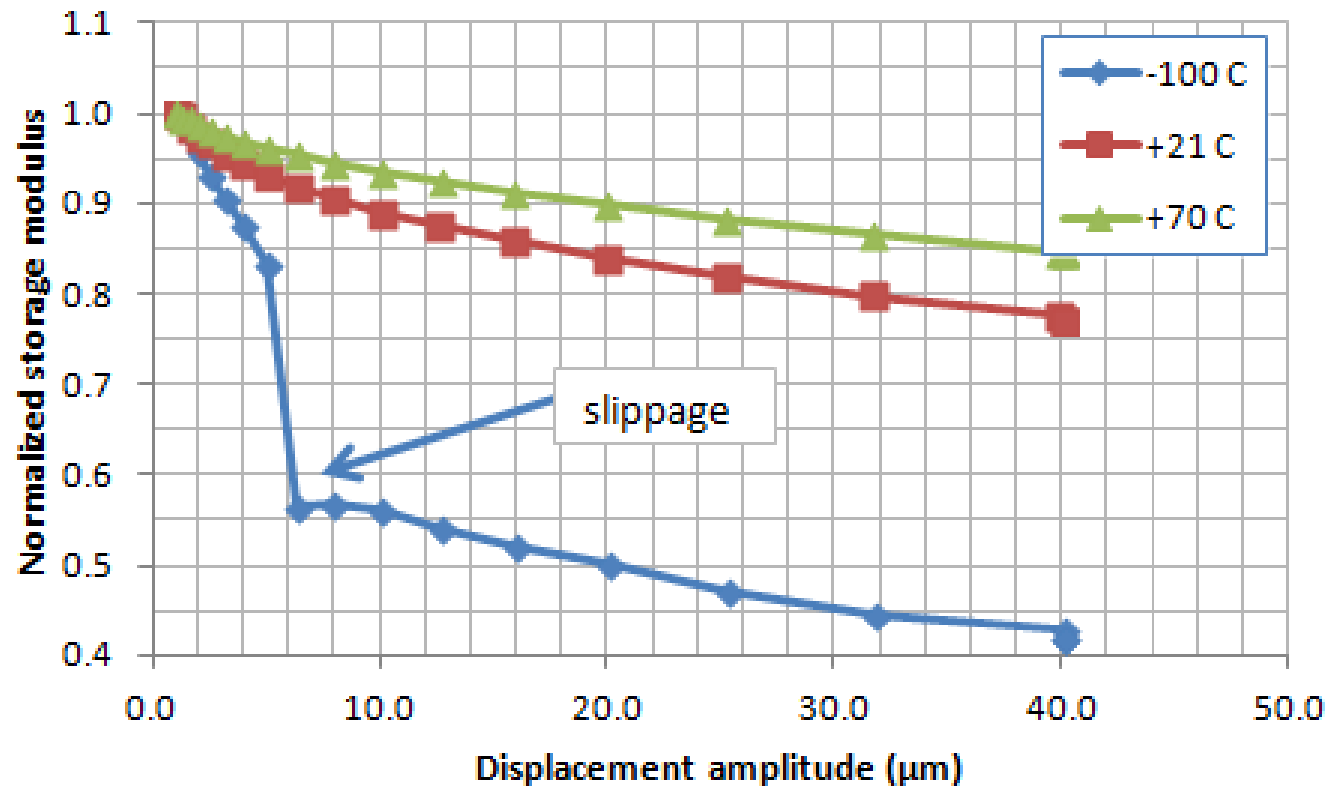
Strain Sweep Tests

Comparison of Normalized Storage Modulus for a Filled Silicone Elastomer and a General Purpose Polystyrene





Strain Sweep Tests





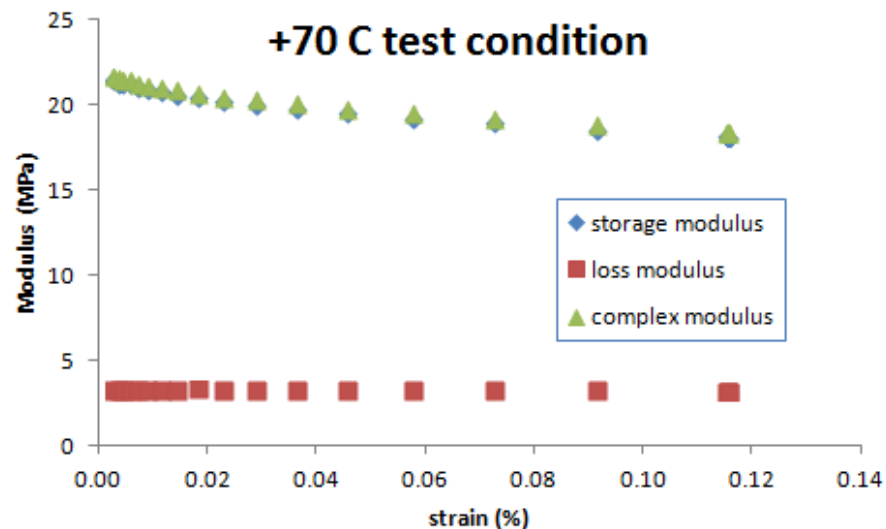
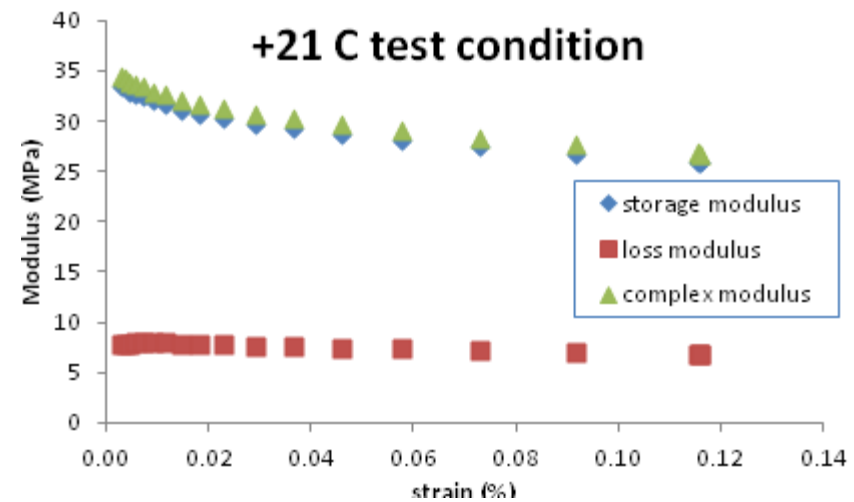
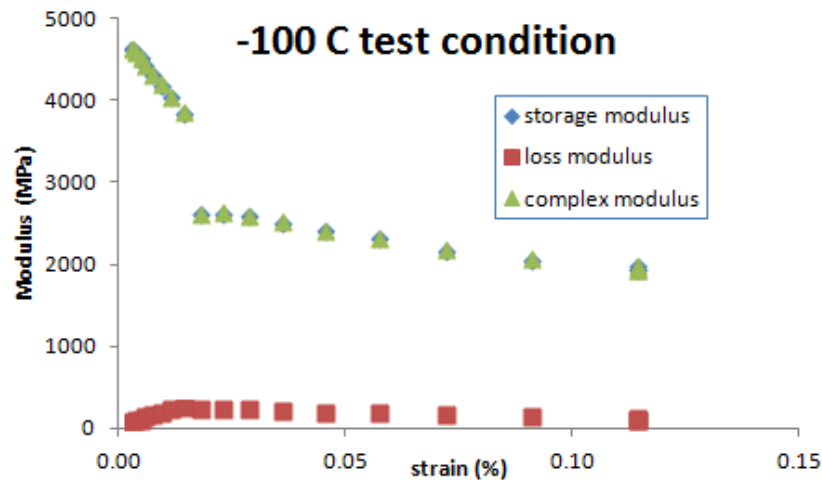
Strain Sweep Tests

Test condition (C)	Upper limit for amplitude for linear viscoelasticity (μm)
-100	2.15
+21	3.58
+70	7.06

This comes from the data on the previous slide. The frequency sweep tests all had the same amplitude, $2.10 \mu\text{m}$, so regardless of temperature linear viscoelasticity was ensured.



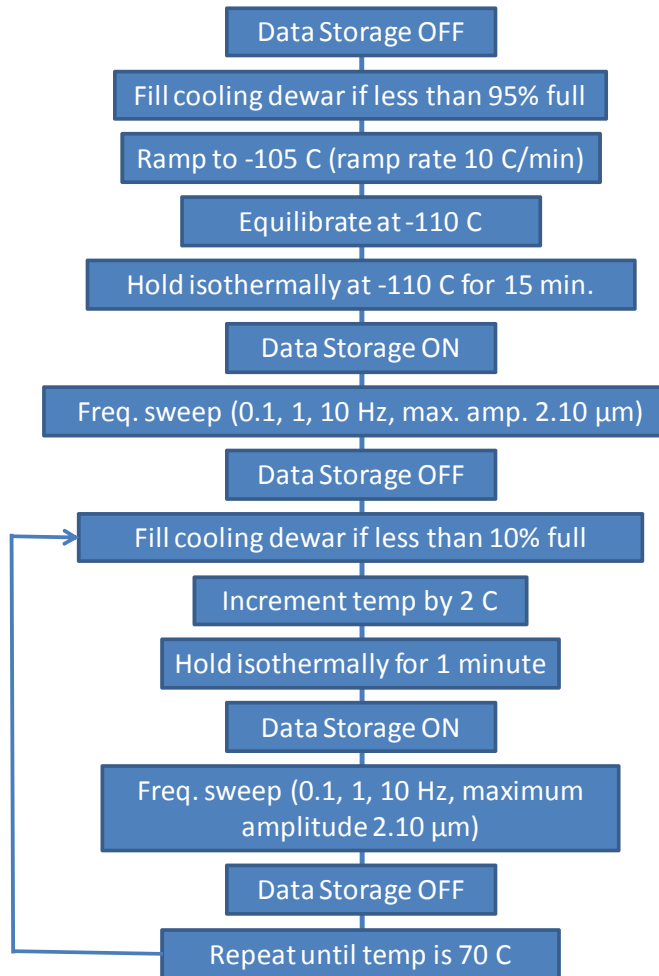
Strain Sweep Tests





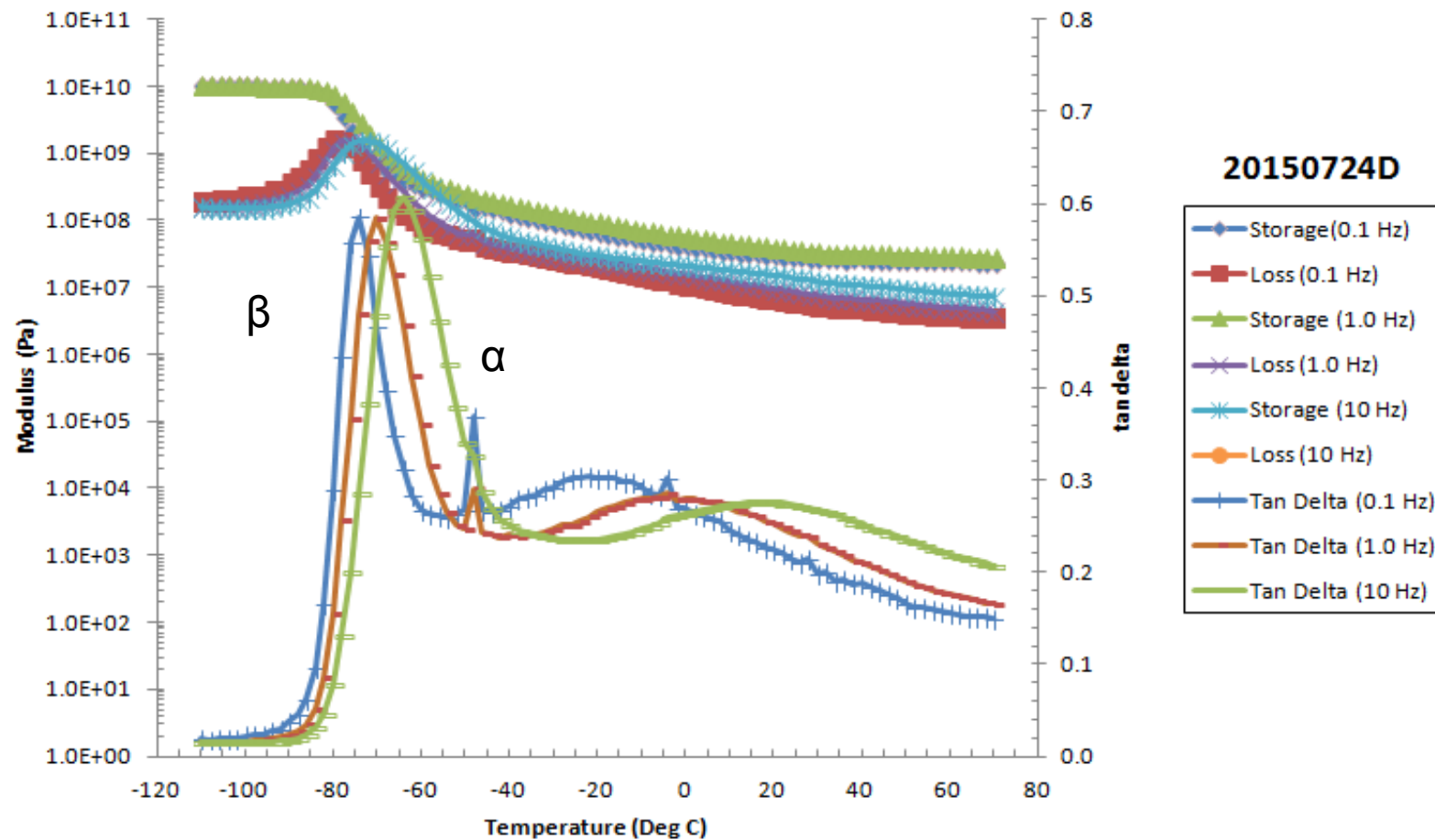
Frequency Sweep Tests

Frequency sweep block diagram



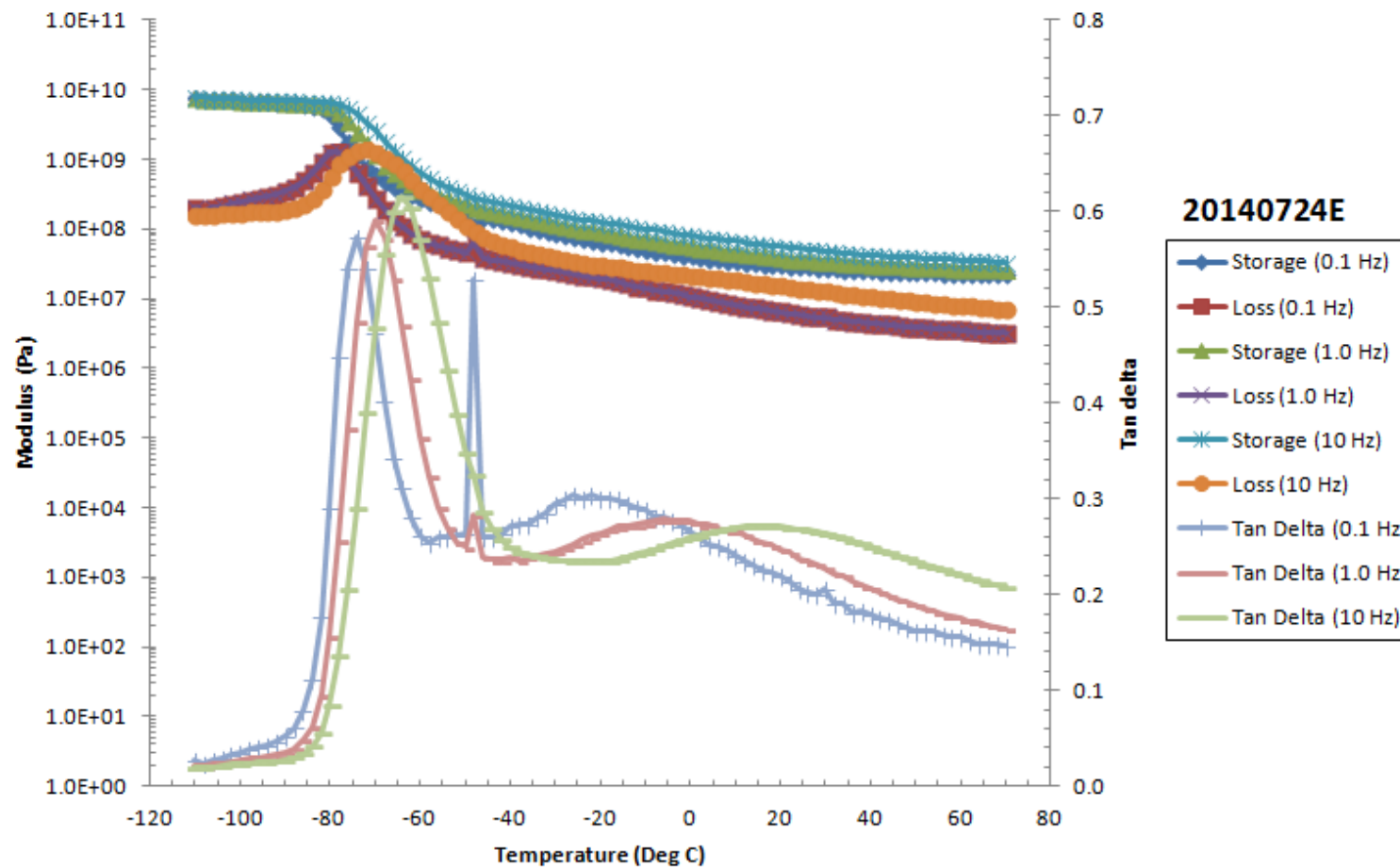


Frequency Sweep Tests



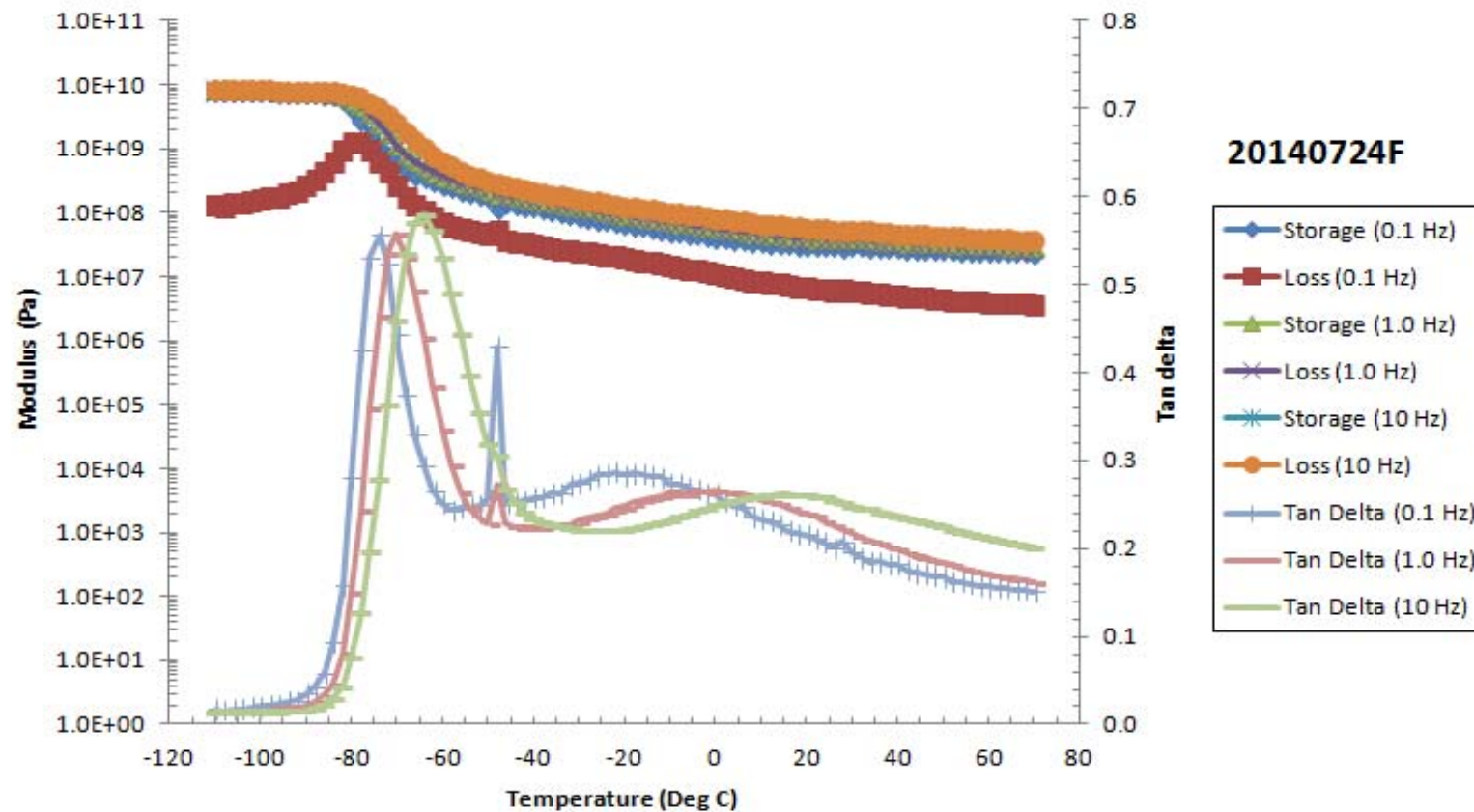


Frequency Sweep Tests





Frequency Sweep Tests





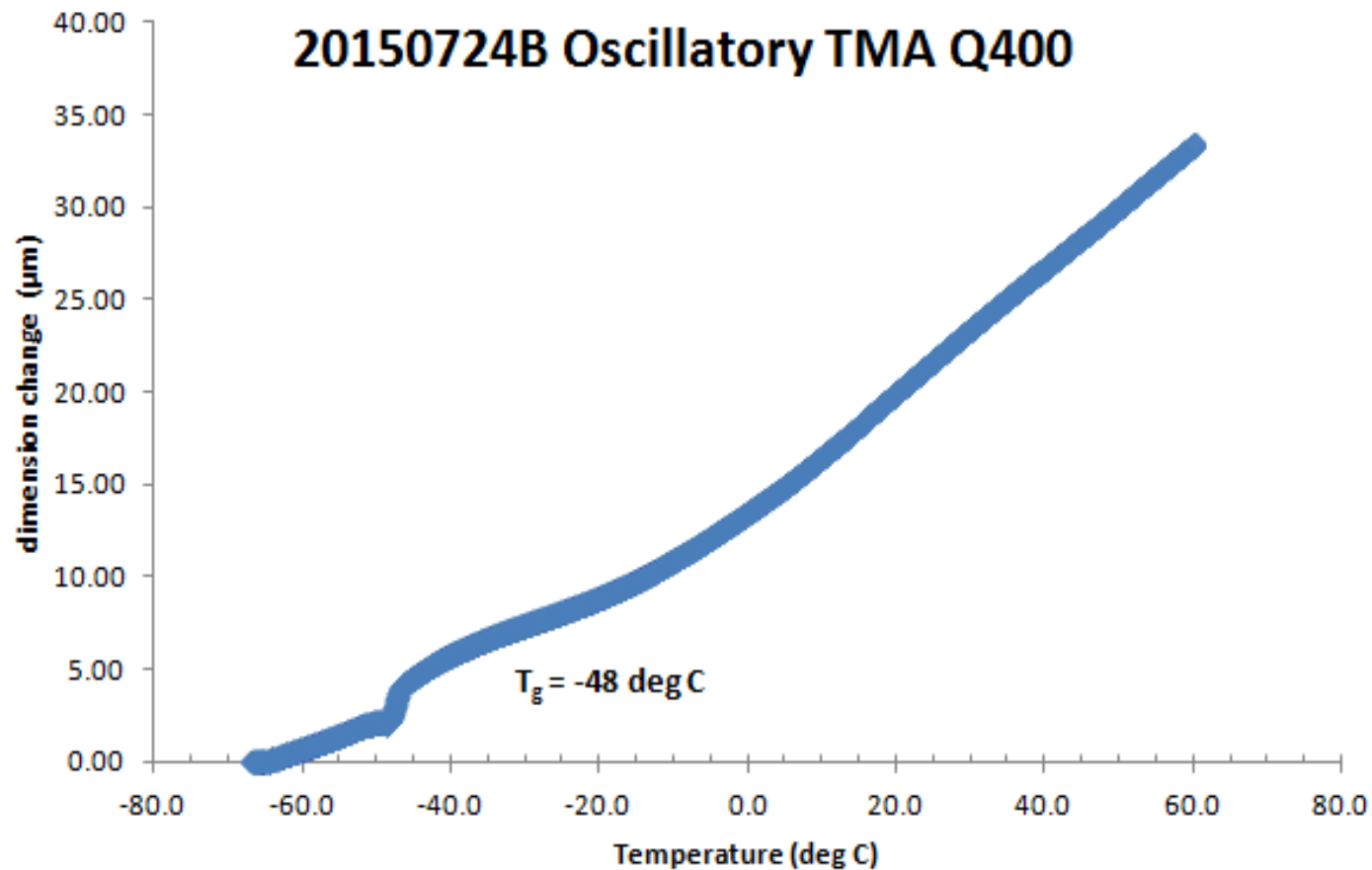
Frequency Sweep Tests

Specimen name	α peaks (deg C)			β peaks (deg C)		
	0.1 Hz	1.0 Hz	10 Hz	0.1 Hz	1.0 Hz	10 Hz
20140724D	-48.0	-48.0	-48.0	-74.0	-70.0	-64.0
20140724E	-48.0	-48.0	-48.0	-74.0	-70.0	-64.0
20140724F	-48.0	-48.0	-46.0	-74.0	-70.0	-64.0

Very consistent peak values for temperature among specimens. T_g not affected by frequency, apparently.

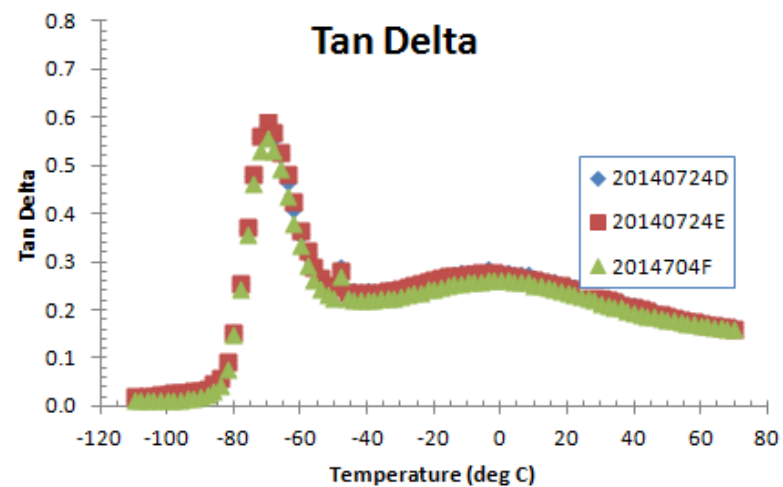
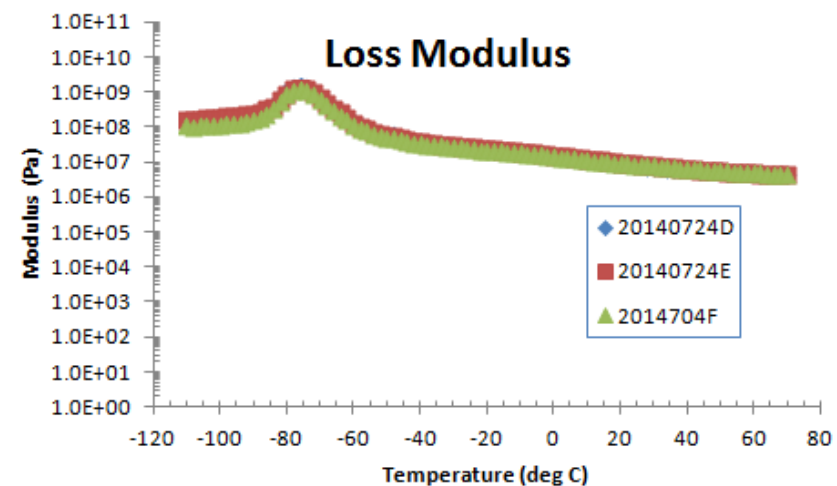
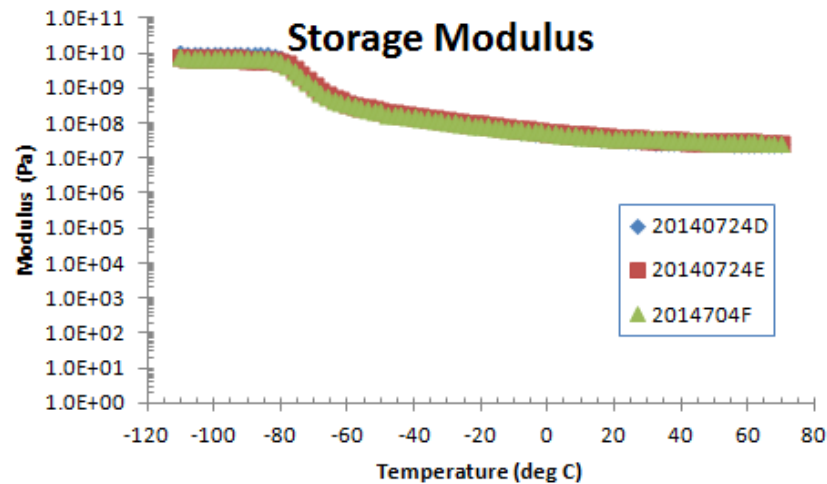


Frequency Sweep Tests





Frequency Sweep Tests



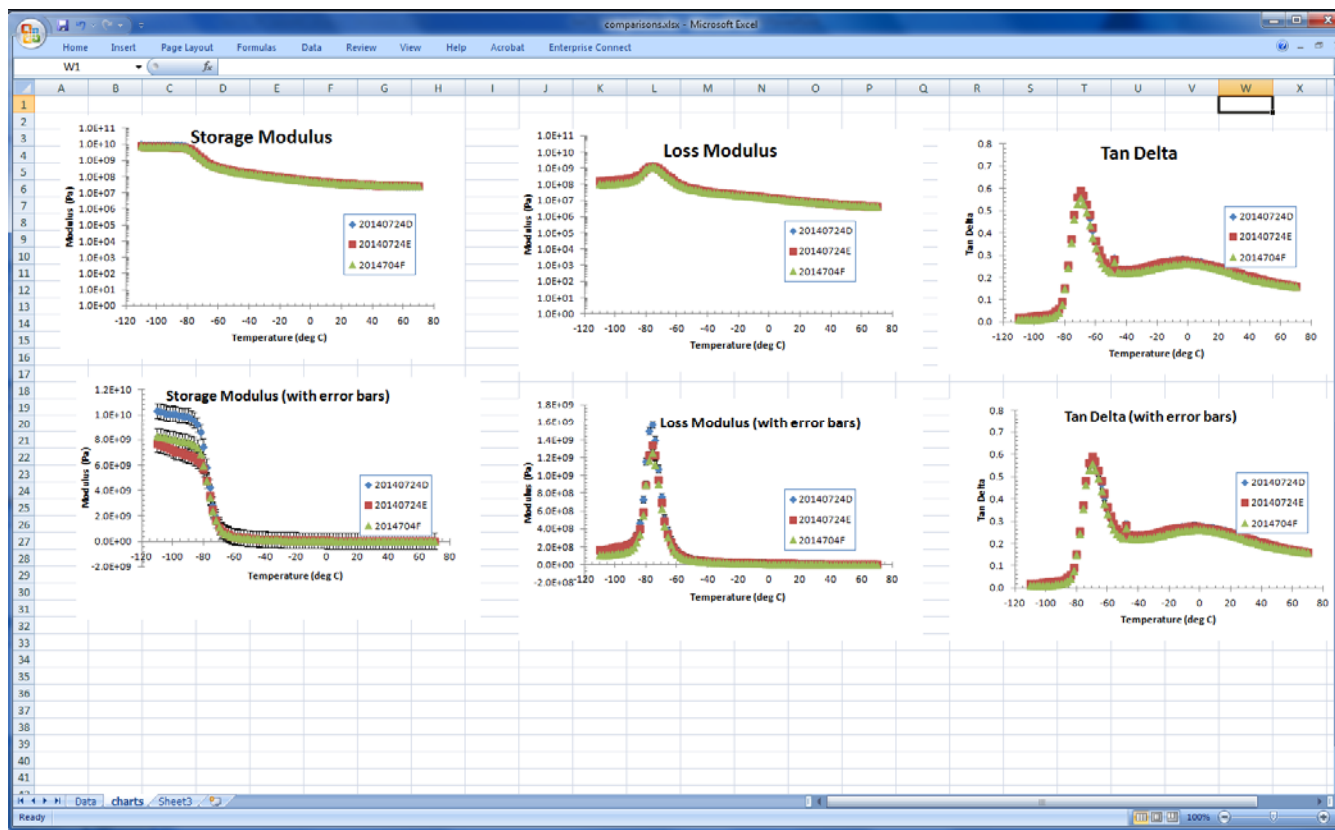


Freq. Sweep Tests (Bonus Information)



$$s_p^2 = \frac{\sum_{i=1}^k (n_i - 1) s_i^2}{\sum_{i=1}^k (n_i - 1)} = \frac{(n_1 - 1) s_1^2 + (n_2 - 1) s_2^2 + \cdots + (n_k - 1) s_k^2}{n_1 + n_2 + \cdots + n_k - k}$$

wikipedia “pooled variances” (for more detail, see book by John Mandel on experiments and statistics)





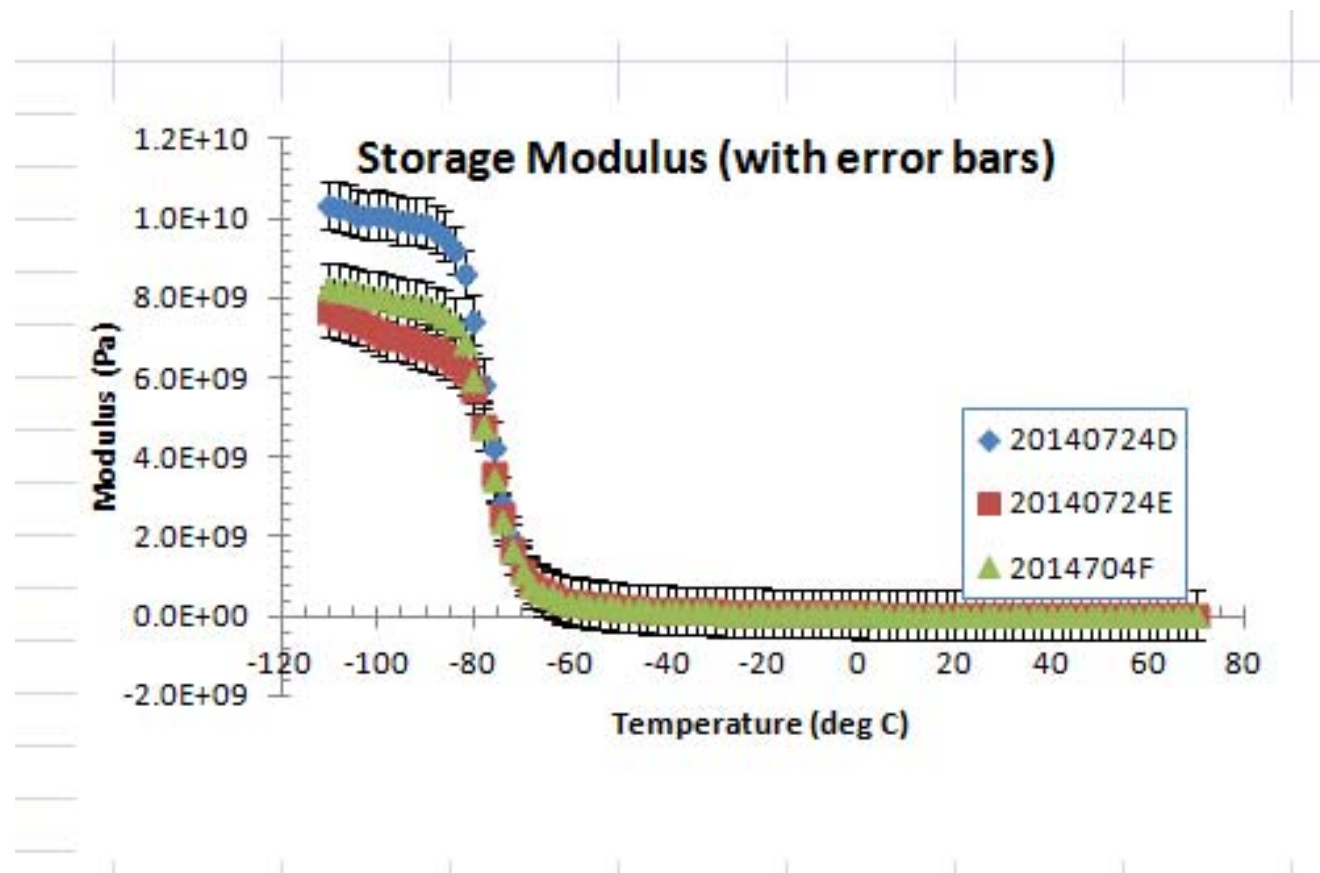
Freq. Sweep Tests (Bonus Information)



Nominal Temperature (deg C)	20140724D (0.1 Hz)			20140724E (0.1 Hz)			20140724F (0.1 Hz)			20140724D (1.0 Hz)			20140724G (1.0 Hz)
	Storage Modulus (Pa)	Loss Modulus (Pa)	Tan Delta	Storage Modulus (Pa)	Loss Modulus (Pa)	Tan Delta	Storage Modulus (Pa)	Loss Modulus (Pa)	Tan Delta	Storage Modulus (Pa)	Loss Modulus (Pa)	Tan Delta	
-110	1.020E+10	1.883E+08	0.0185	7.554E+09	2.053E+08	0.0272	8.142E+09	1.334E+08	0.0164	1.033E+10	1.589E+08	0.0154	7.664E+09
-108	1.014E+10	1.843E+08	0.0182	7.470E+09	1.777E+08	0.0238	8.134E+09	1.235E+08	0.0152	1.029E+10	1.597E+08	0.0155	7.592E+09
-106	1.008E+10	1.976E+08	0.0196	7.356E+09	2.007E+08	0.0273	8.086E+09	1.401E+08	0.0173	1.025E+10	1.636E+08	0.0160	7.479E+09
-104	9.967E+09	1.874E+08	0.0188	7.286E+09	2.104E+08	0.0289	8.024E+09	1.374E+08	0.0171	1.013E+10	1.629E+08	0.0161	7.416E+09
-102	9.914E+09	1.962E+08	0.0198	7.185E+09	2.336E+08	0.0325	7.956E+09	1.474E+08	0.0185	1.008E+10	1.636E+08	0.0162	7.329E+09
-100	9.913E+09	2.222E+08	0.0224	7.037E+09	2.406E+08	0.0342	7.911E+09	1.581E+08	0.0200	1.010E+10	1.626E+08	0.0161	7.179E+09
-98	9.889E+09	2.341E+08	0.0237	6.934E+09	2.639E+08	0.0381	7.868E+09	1.710E+08	0.0217	1.008E+10	1.786E+08	0.0177	7.067E+09
-96	9.798E+09	2.299E+08	0.0235	6.845E+09	2.802E+08	0.0409	7.781E+09	1.739E+08	0.0223	9.982E+09	1.805E+08	0.0181	7.014E+09
-94	9.737E+09	2.740E+08	0.0281	6.779E+09	2.898E+08	0.0427	7.711E+09	2.019E+08	0.0262	9.943E+09	1.908E+08	0.0192	6.945E+09
-92	9.695E+09	2.853E+08	0.0294	6.648E+09	3.087E+08	0.0464	7.677E+09	2.087E+08	0.0272	9.936E+09	2.076E+08	0.0209	6.842E+09
-90	9.608E+09	3.672E+08	0.0382	6.557E+09	3.461E+08	0.0528	7.602E+09	2.671E+08	0.0351	9.889E+09	2.358E+08	0.0238	6.767E+09
-88	9.409E+09	4.346E+08	0.0462	6.433E+09	3.912E+08	0.0608	7.422E+09	3.163E+08	0.0426	9.722E+09	2.654E+08	0.0273	6.706E+09
-86	9.090E+09	5.597E+08	0.0616	6.250E+09	4.924E+08	0.0788	7.214E+09	4.219E+08	0.0585	9.555E+09	3.264E+08	0.0342	6.601E+09
-84	8.457E+09	8.251E+08	0.0976	5.872E+09	6.529E+08	0.1112	6.827E+09	6.356E+08	0.0931	9.210E+09	4.694E+08	0.0510	6.396E+09
-82	7.386E+09	1.224E+09	0.1657	5.270E+09	9.337E+08	0.1772	5.930E+09	9.329E+08	0.1573	8.622E+09	7.330E+08	0.0850	6.148E+09
-80	5.433E+09	1.575E+09	0.2898	4.297E+09	1.248E+09	0.2904	4.439E+09	1.248E+09	0.2812	7.459E+09	1.160E+09	0.1555	5.711E+09
-78	3.459E+09	1.505E+09	0.4351	2.896E+09	1.298E+09	0.4481	2.914E+09	1.235E+09	0.4240	5.853E+09	1.504E+09	0.2569	4.771E+09
-76	2.114E+09	1.182E+09	0.5593	1.782E+09	9.642E+08	0.5411	1.769E+09	9.383E+08	0.5303	4.281E+09	1.573E+09	0.3674	3.564E+09
-74	1.325E+09	7.760E+08	0.5857	1.154E+09	6.621E+08	0.5736	1.139E+09	6.326E+08	0.5554	2.916E+09	1.399E+09	0.4800	2.517E+09
-72	9.019E+08	4.910E+08	0.5444	7.915E+08	4.282E+08	0.5410	8.183E+08	4.288E+08	0.5241	1.931E+09	1.080E+09	0.5591	1.680E+09
-70	6.626E+08	3.097E+08	0.4675	5.928E+08	2.803E+08	0.4728	5.992E+08	2.656E+08	0.4432	1.305E+09	7.624E+08	0.5842	1.171E+09
-68	5.284E+08	2.104E+08	0.3981	4.726E+08	1.904E+08	0.4029	4.738E+08	1.776E+08	0.3748	9.347E+08	5.221E+08	0.5586	8.457E+08
-66	4.485E+08	1.567E+08	0.3494	3.965E+08	1.356E+08	0.3421	4.058E+08	1.333E+08	0.3284	7.326E+08	3.839E+08	0.5240	6.489E+08
-64	3.868E+08	1.205E+08	0.3116	3.475E+08	1.085E+08	0.3124	3.539E+08	1.038E+08	0.2932	5.872E+08	2.745E+08	0.4674	5.297E+08
-62	3.389E+08	9.636E+07	0.2844	3.094E+08	8.681E+07	0.2806	3.124E+08	8.293E+07	0.2655	4.870E+08	2.009E+08	0.4125	4.426E+08
-60	3.030E+08	8.099E+07	0.2673	2.715E+08	7.087E+07	0.2610	2.814E+08	7.048E+07	0.2505	4.165E+08	1.502E+08	0.3606	3.731E+08
-58	2.709E+08	7.148E+07	0.2639	2.469E+08	6.289E+07	0.2547	2.544E+08	6.179E+07	0.2429	3.661E+08	1.157E+08	0.3161	3.320E+08
-56	2.453E+08	6.425E+07	0.2619	2.232E+08	5.806E+07	0.2601	2.309E+08	5.674E+07	0.2457	3.277E+08	9.359E+07	0.2856	2.995E+08



Freq. Sweep Tests (Bonus Information)





Summary and Conclusions

- When making the specimens for dual cantilever beam fixtures, one important consideration is uniform thickness – machining to a uniform thickness before cutting the specimens from a common slab can enhance reproducibility.
- The linear viscoelasticity regime can be discerned by comparing normalized storage moduli values over a range of deformations and finding the threshold at which the normalized value drops to 95% of the starting value.
- Clamping tension is not guaranteed when testing using frequency sweeps over a wide range of temperatures unless a procedure is used that ensures the clamps are tight at the coldest temperature.
- Long tests such as the frequency sweep sequences prescribed in this round robin may be problematic for the Q800 due to lack of sufficient reserve of liquid nitrogen in the gas cooling accessory.



Summary and Conclusions

- **Determination of T_g by locating the peak of the tan delta function was very repeatable and consistent with thermomechanical analysis results from another device.**
- **The β peaks varied with frequency but were repeatable for a given frequency. For the α peaks, there was no significant frequency effect.**
- **Of the three pertinent parameters (storage modulus, loss modulus, and tan delta), only the storage modulus showed variability issues, and that only took place on the “upper shelf” (below about -80 C).**

The End

