SAND2010-3720C

Improved Properties and Processing of Alumina Filled Nanocomposites

40th Annual PolyMAC Symposium

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Postdoctoral Appointee

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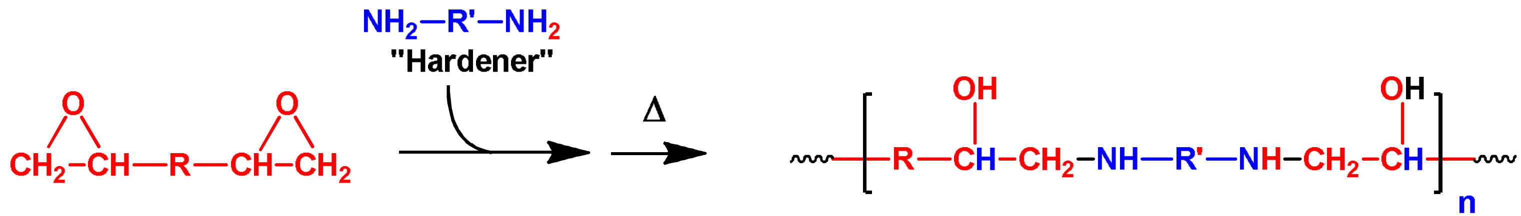
Overview/Problem

Sandia is carrying out research on encapsulants for the prevention of high voltage breakdown

The ideal material would…

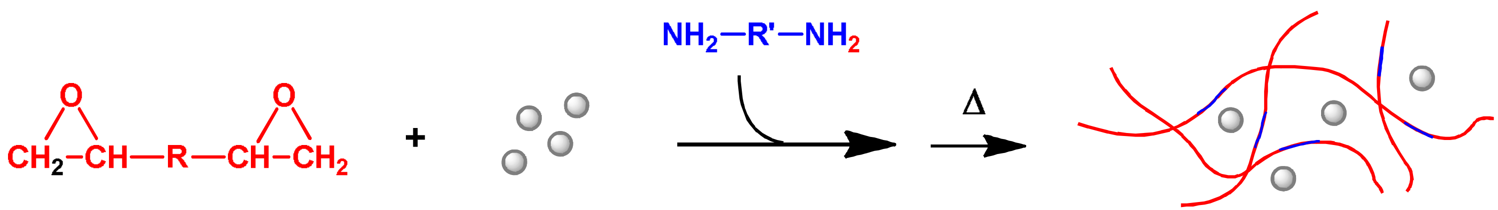
1) operate under shockwave conditions
2) survive thermal cycling
3) operate over a wide temperature range

Solution:
Alumina Filled Polymer Composites



"Epoxy Resin"

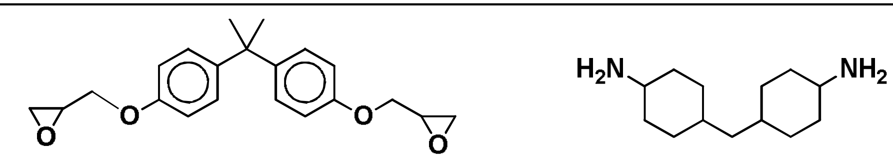
"Polymer"



Al_2O

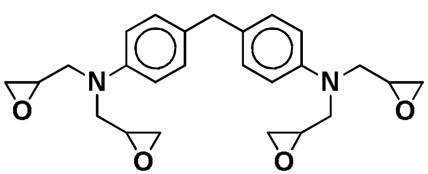
"Polymer Nanocomposite"

Pool of Epoxies and Hardeners

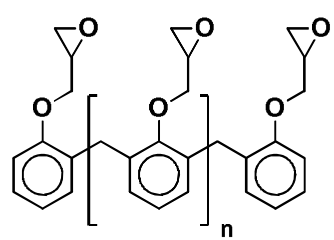


DGEBA

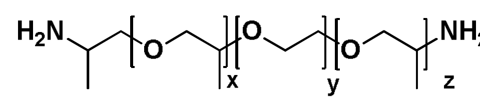
Cycloaliphatic diamines



Epoxide Monomers



Novolac Epoxies



Polyamines

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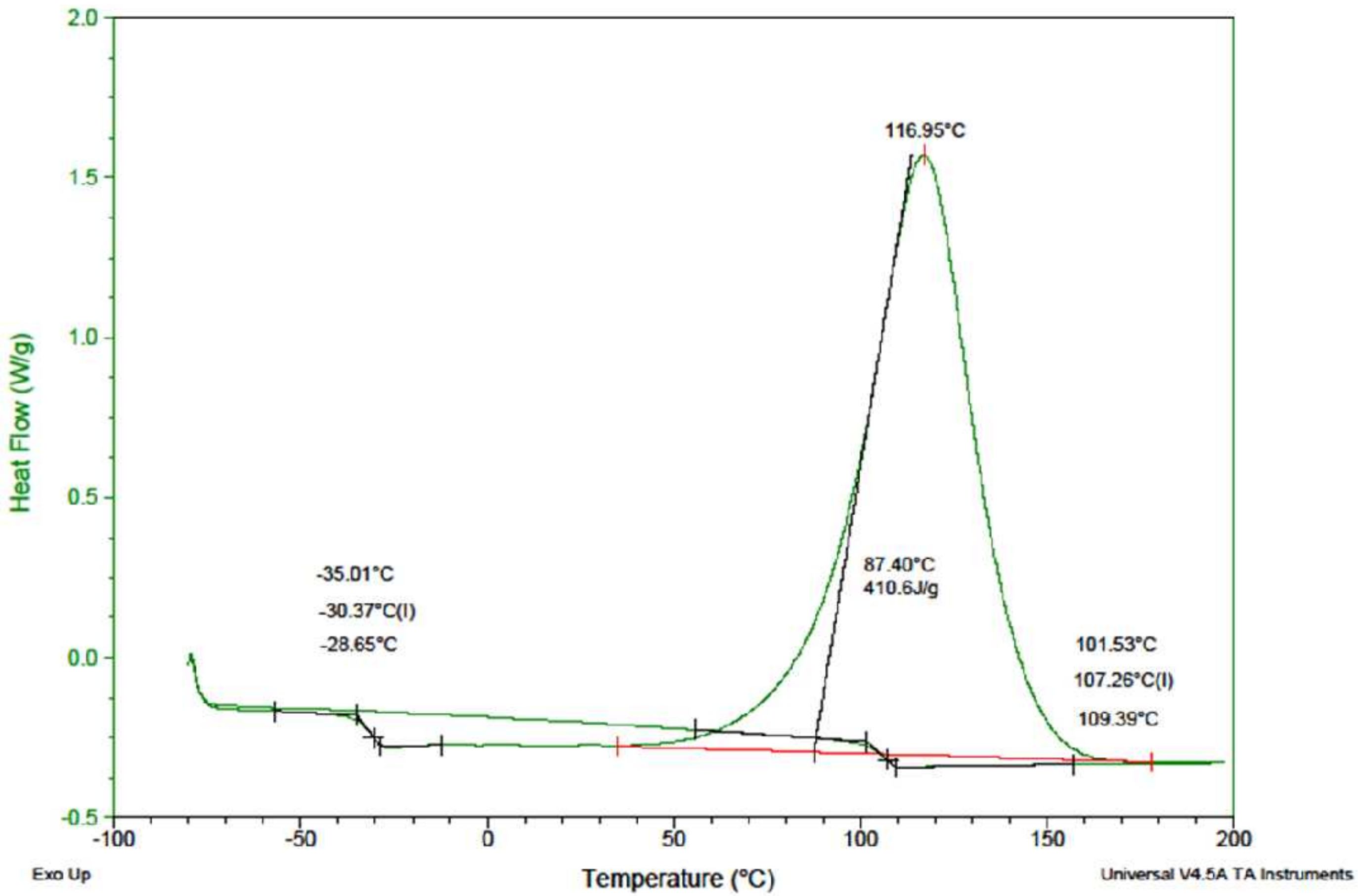
Curing Temperature

DSC Curve for Neat Polymer (1:1 Epoxy/Hardener)



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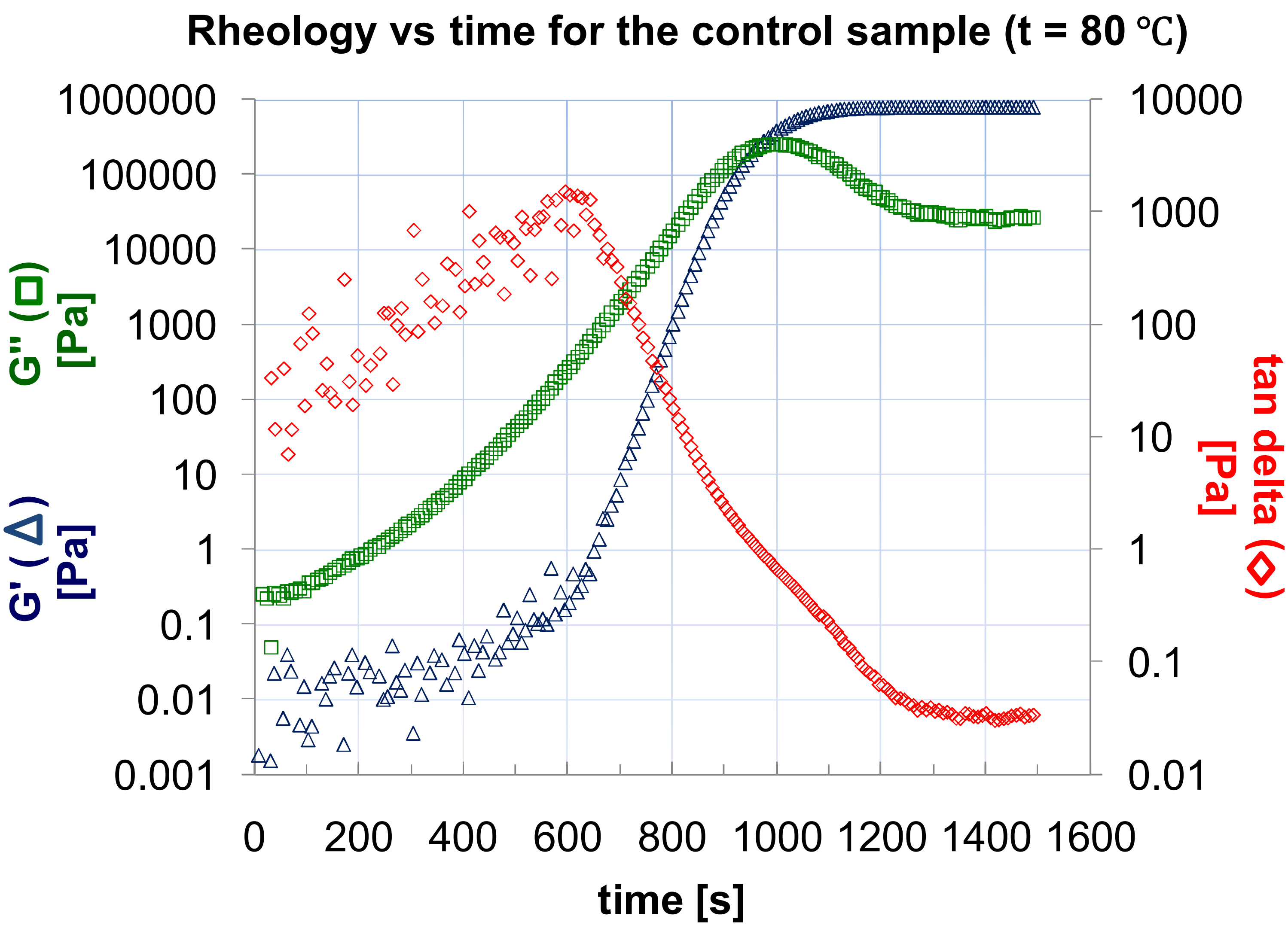
Rheology Measurement



(Measured at a frequency of 1 Hz.)

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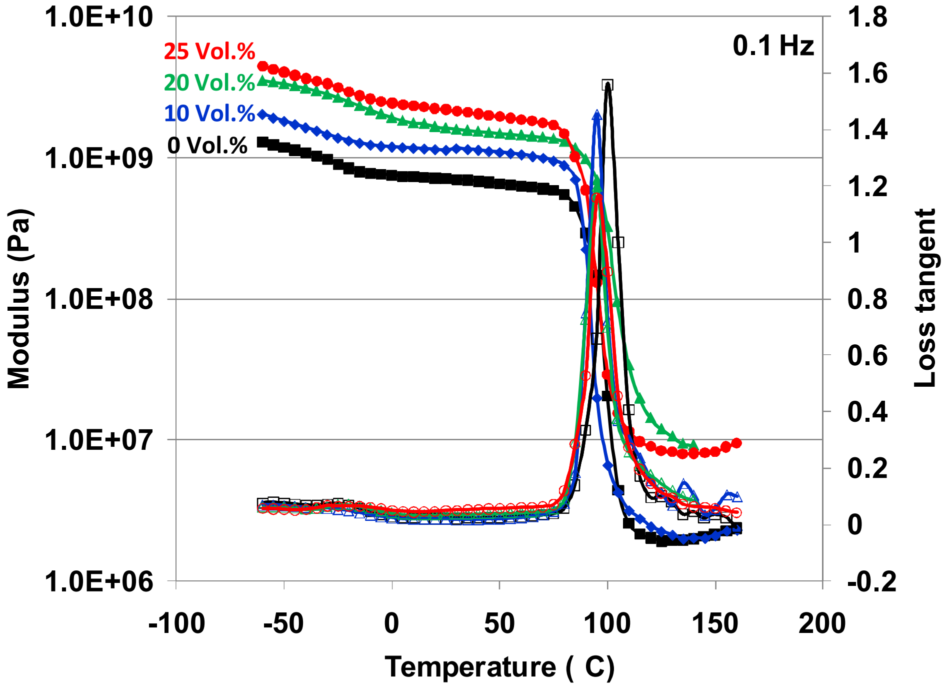
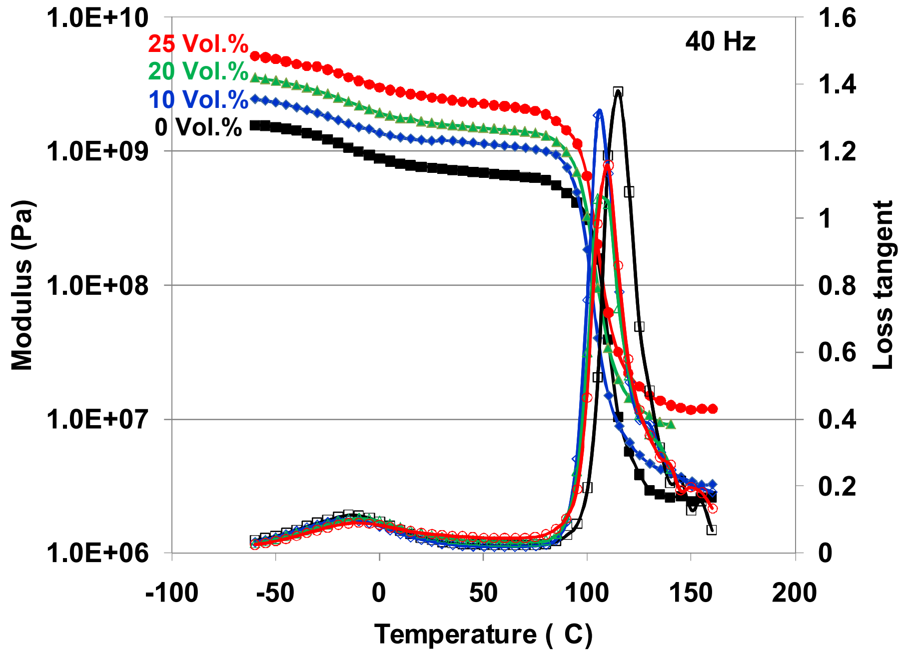
Dynamic Modulus and Tg Measurements

•The dynamic modulus of our nanocomposites increases as the amount of solid loading increases.

•The glass transition temperature of our nanocomposites is between 95°C and 114°C (depending on driving frequency).





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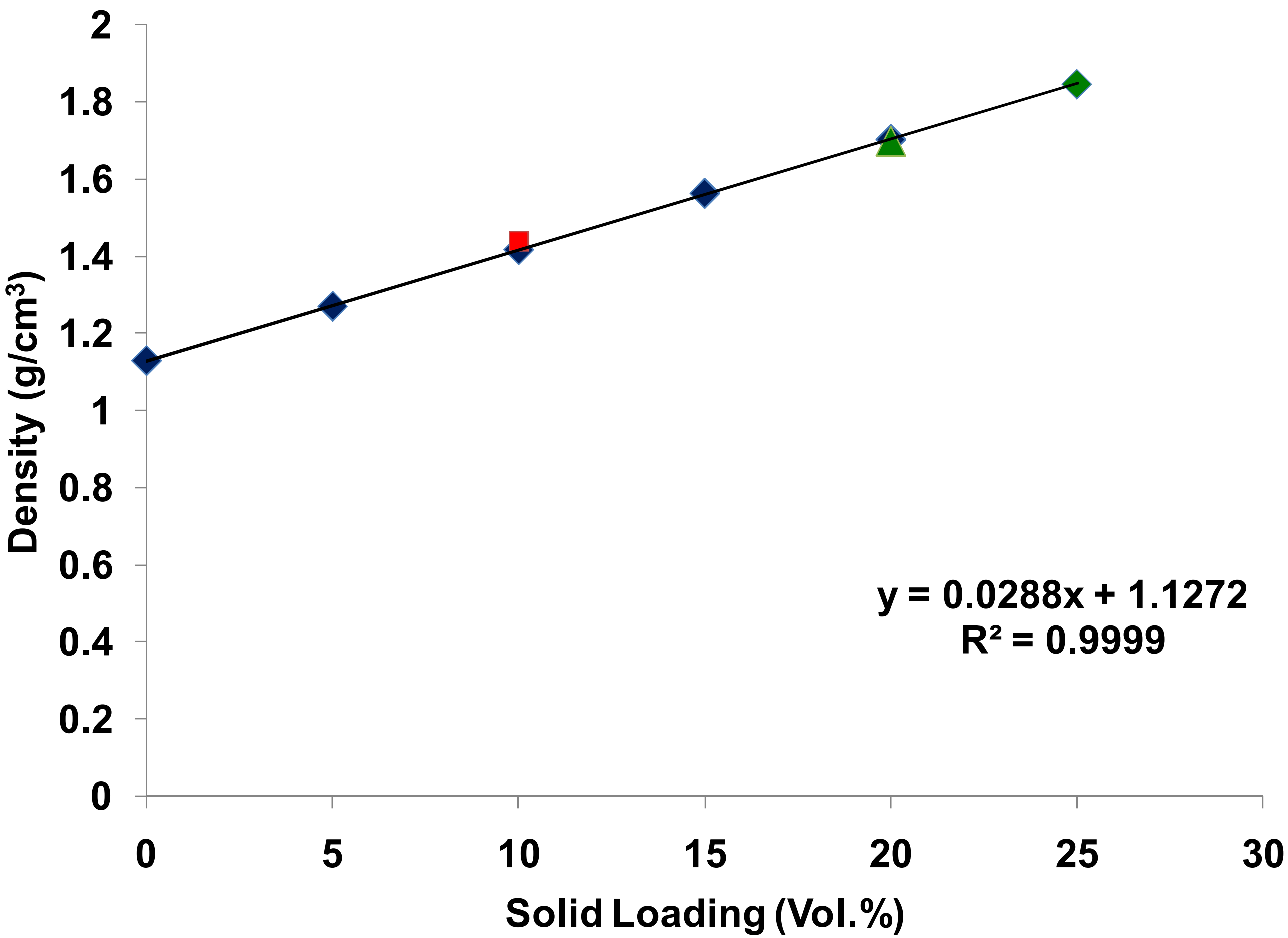
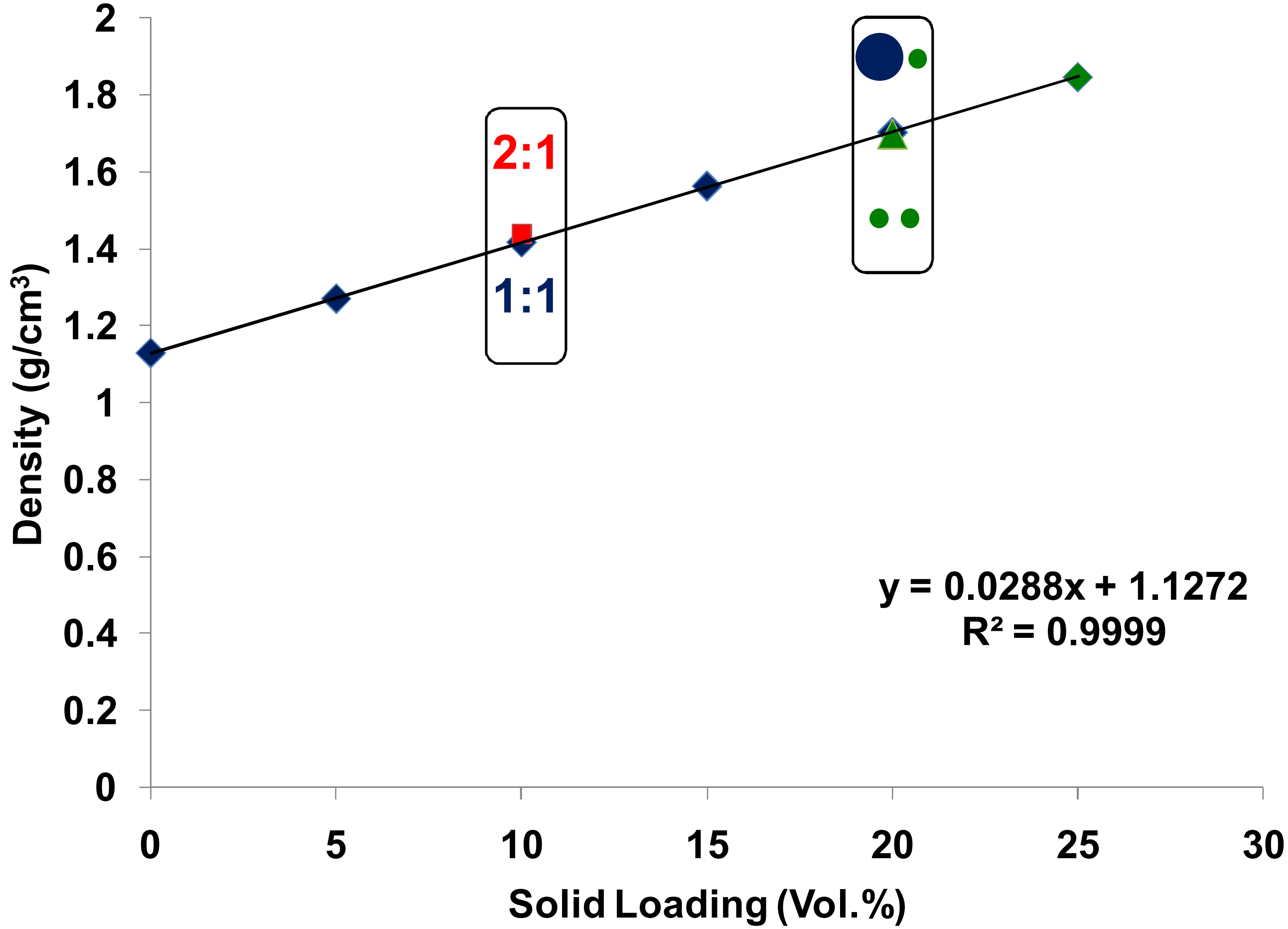
Density of the Composites



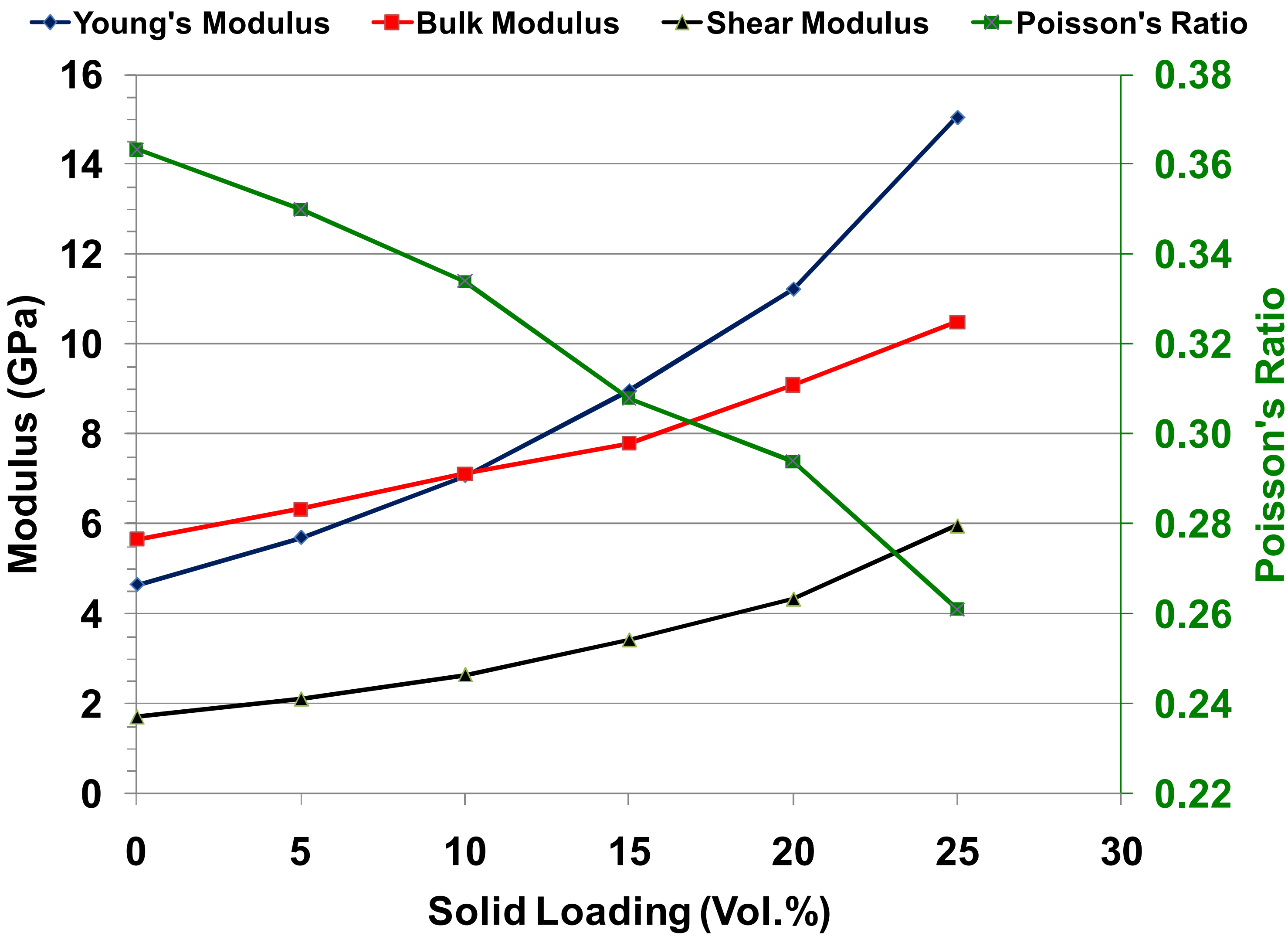
The bulk density increases linearly with the amount of solid loading. (Determined by the Archimedes method)

Density of the Composites



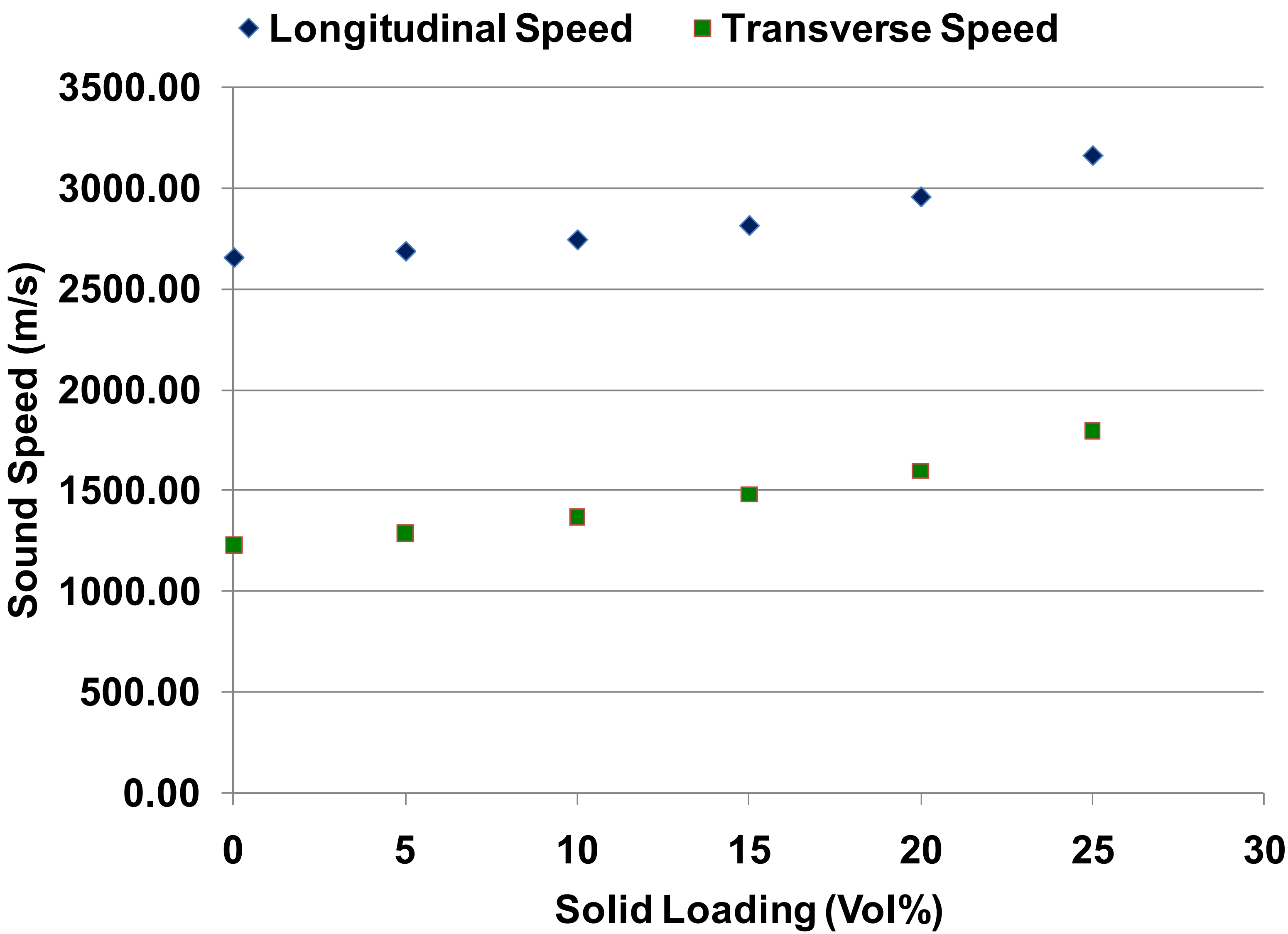
The bulk density increases linearly with the amount of solid loading.
(Determined by the Archimedes method)

Elastic Modulus Measurements



(Moduli were determined by pulse/echo technique at 2.25 MHz)

Acoustic Measurements



(Sound speed was determined pulse/echo technique at 2.25 MHz)

Summary – Density, Elastic, and Acoustic Measurements

Solid loading (vol.%)	Thickness (mm)	Density (g/cc)	TOF (μsec) Longit.	TOF (μsec) Shear	Speed (m/sec) Longit.	Speed (m/sec) Shear	Youngs Modulus (GPa)	Shear Modulus (GPa)	Bulk Modulus (GPa)	Poission's Ratio
0	6.6751	1.128	2.516	5.430	2653.28	1229.36	4.65	1.70	5.67	0.363
5	6.6777	1.269	2.487	5.178	2684.78	1289.56	5.70	2.11	6.33	0.350
10	6.6192	1.416	2.414	4.838	2742.44	1368.04	7.07	2.65	7.12	0.334
15	6.6243	1.562	2.355	4.473	2813.30	1481.07	8.97	3.43	7.79	0.308
20	6.6675	1.702	2.255	4.177	2957.07	1596.39	11.23	4.34	9.10	0.294
25	6.6662	1.846	2.108	3.707	3162.55	1798.07	15.05	5.97	10.50	0.261
20	6.624	1.700	2.199	4.029	3007.61	1641.60	11.80	4.58	9.27	0.288
10	6.607	1.440	2.436	4.863	2711.70	1358.65	7.08	2.66	7.04	0.332

- A linear correlation between solid loading and density is observed – suggesting a well behaved and controlled mixing process.
- It appears that the only thing which can significantly change the elastic properties of these nanocomposites is the amount of solid loading. (at least determined by ultrasonic measurements)
  - The amount of cross-linking makes no difference
  - Alumina particle size makes no difference

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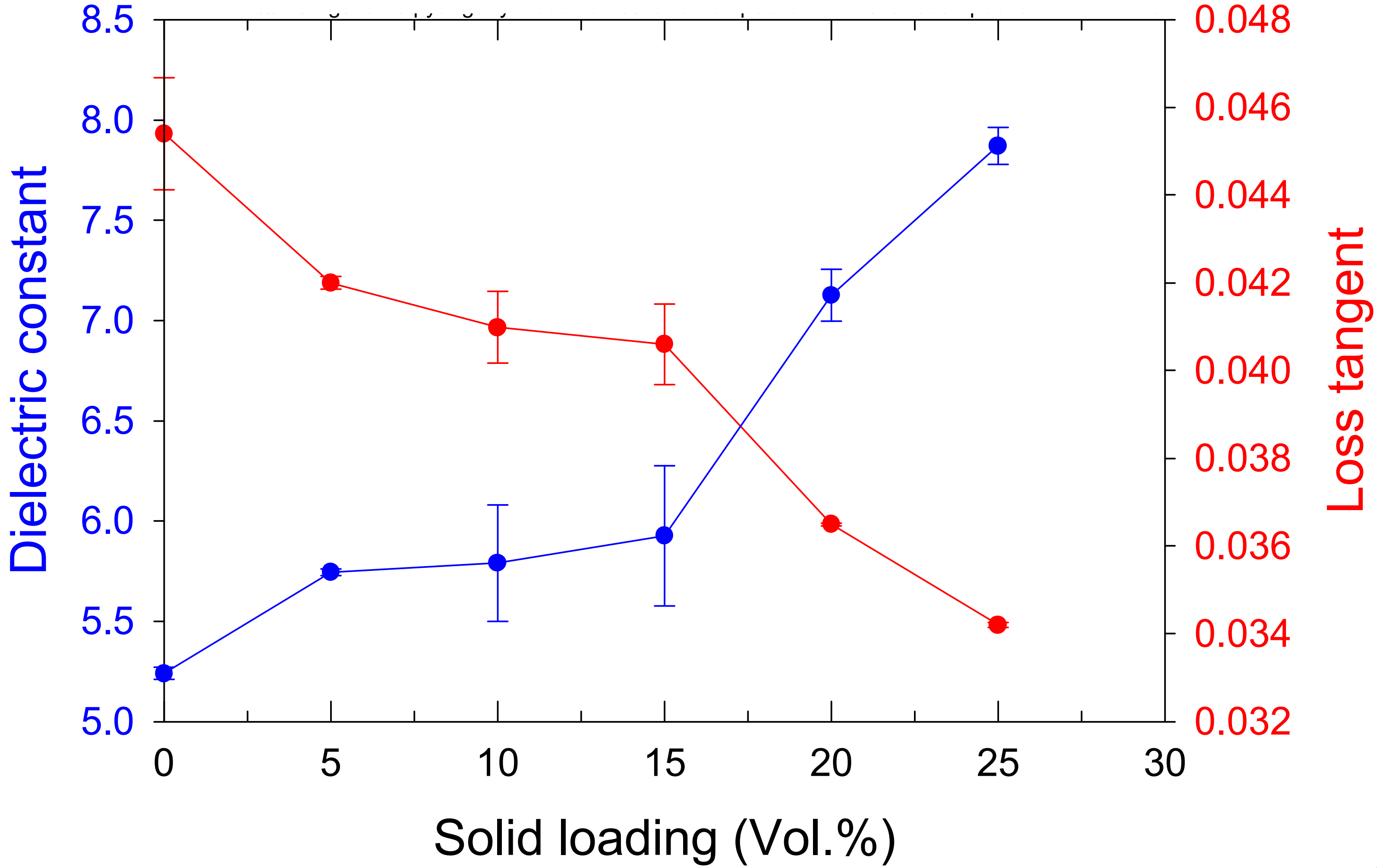
Dielectric Constant Measurements



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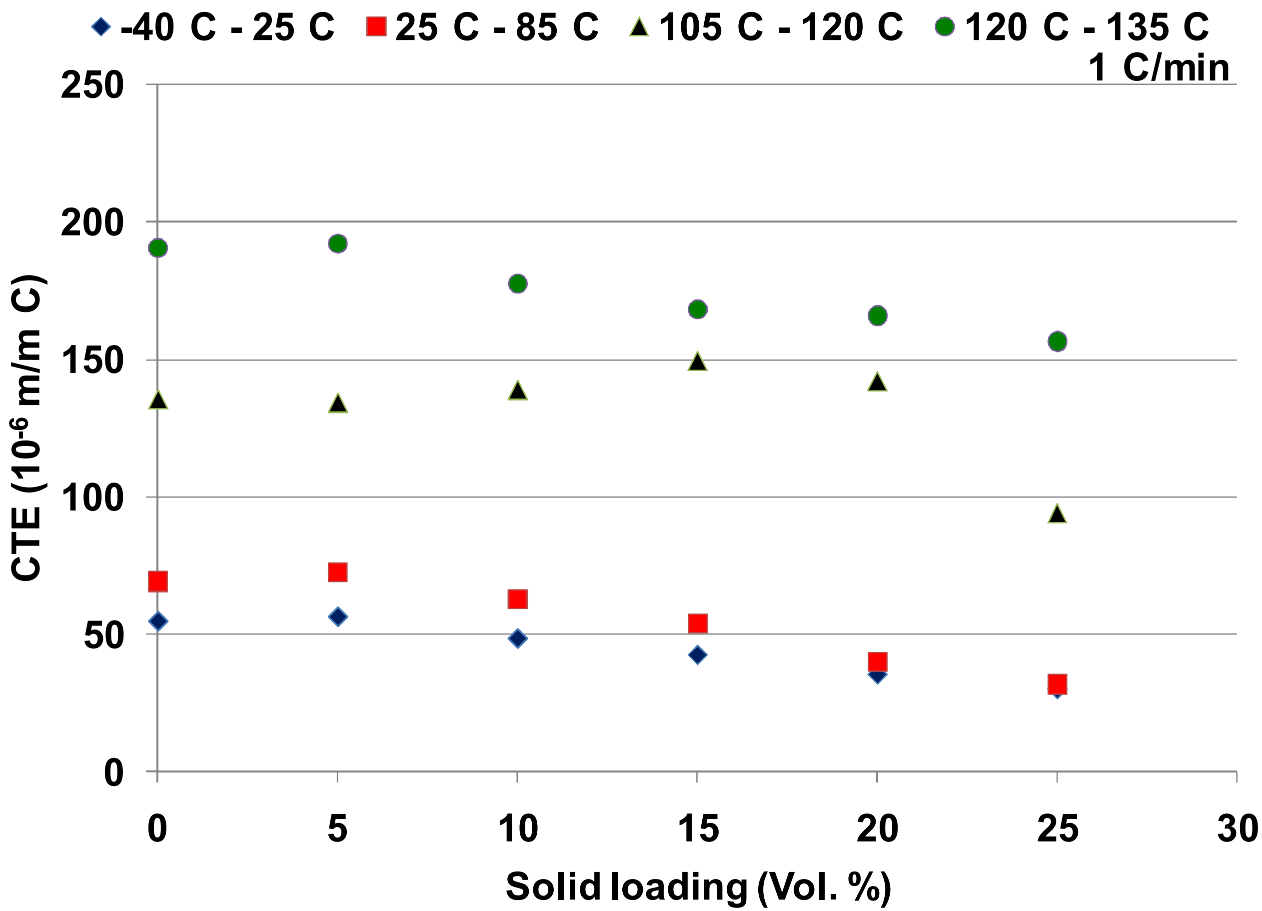
Coefficient of Thermal Expansion Measurements



- In general, the CTE value decreases as the amount of filler increases.
- The difference in CTE values between two sections below the glass transition (T_g ~ 80-90°C)temperature decreases as the amount of solid increases.

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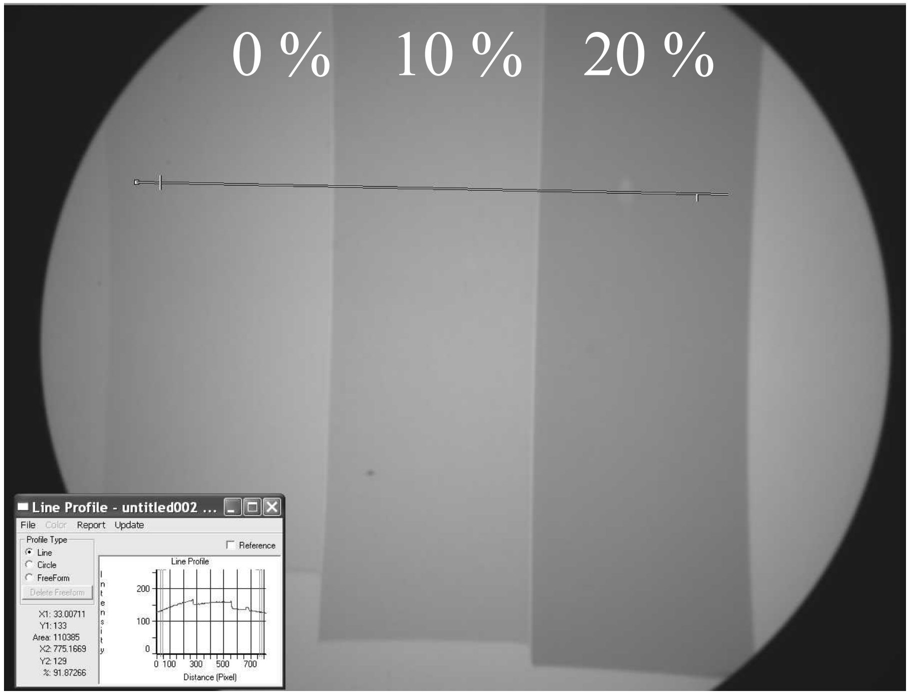
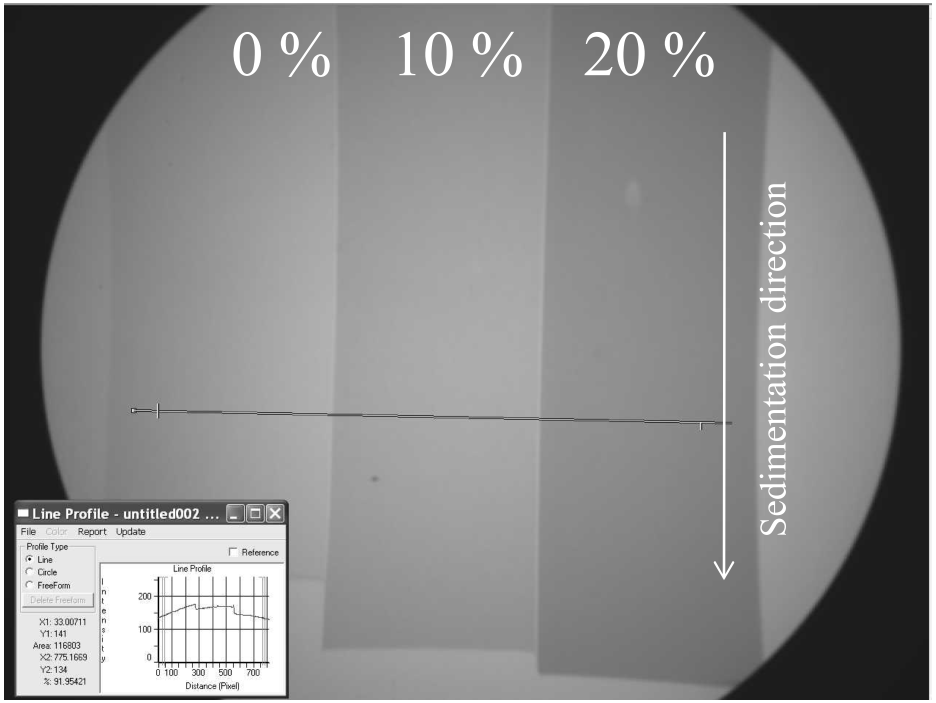
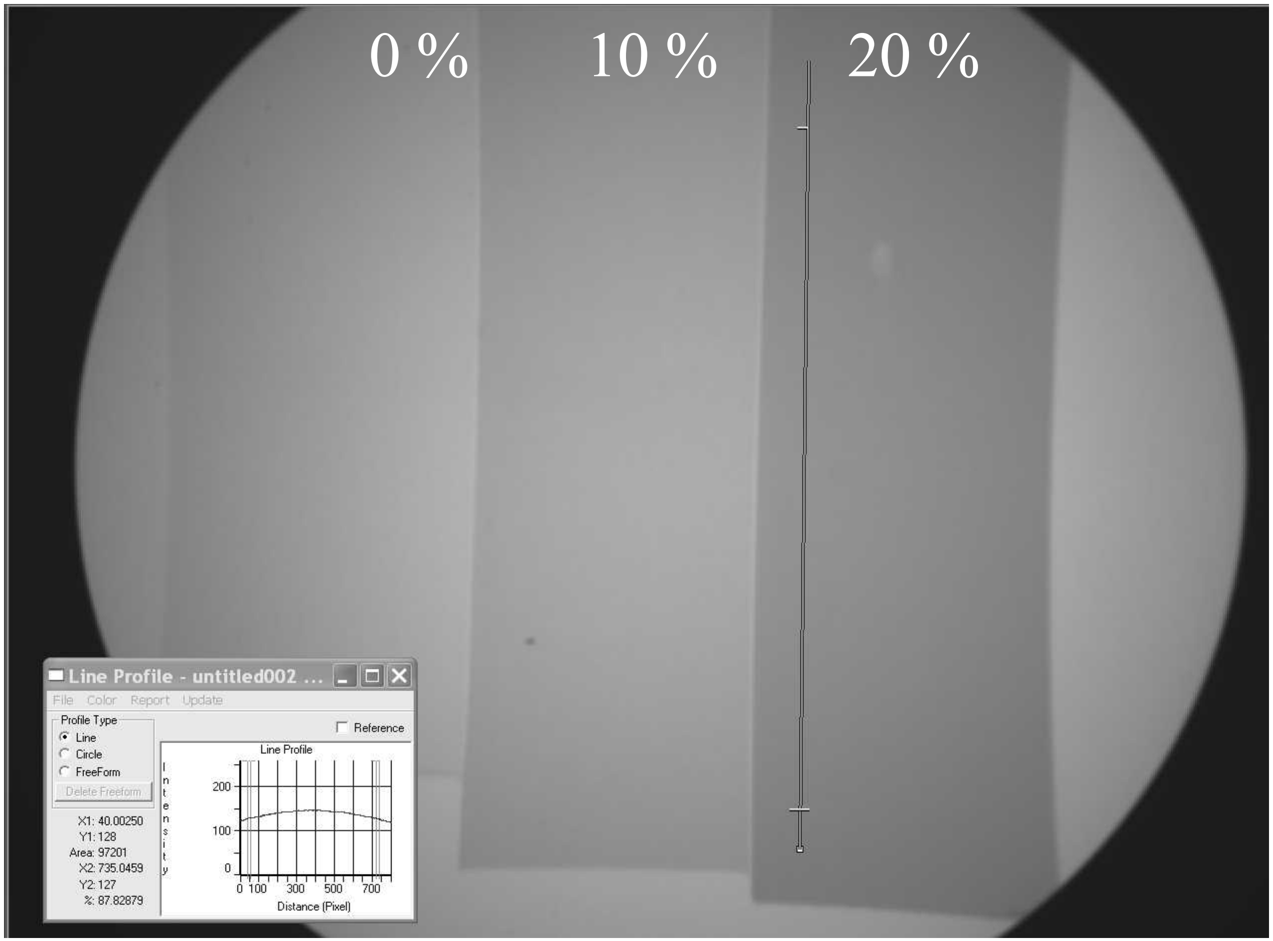
Sedimentation Measurements





(Measured X-ray radiography technique)

Sedimenation Measurements (cont'd)

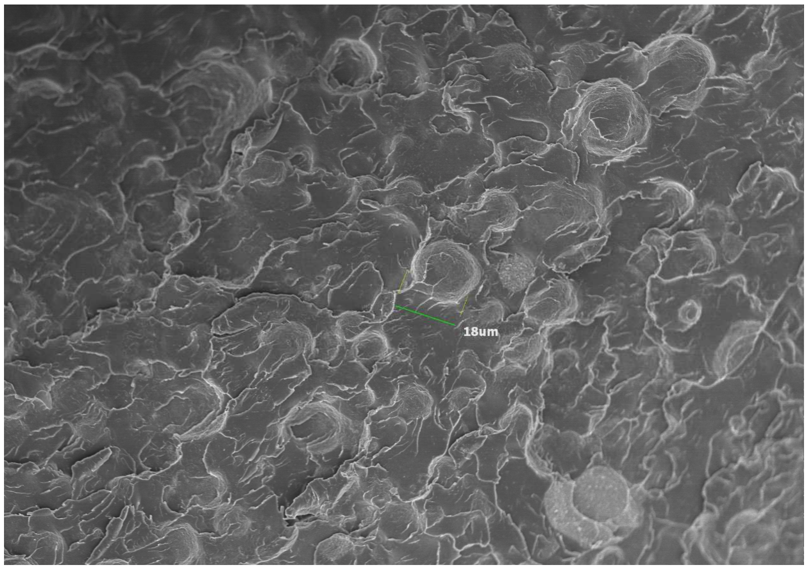


(Measured X-ray radiography technique)

Summary – X-ray radiography inspection

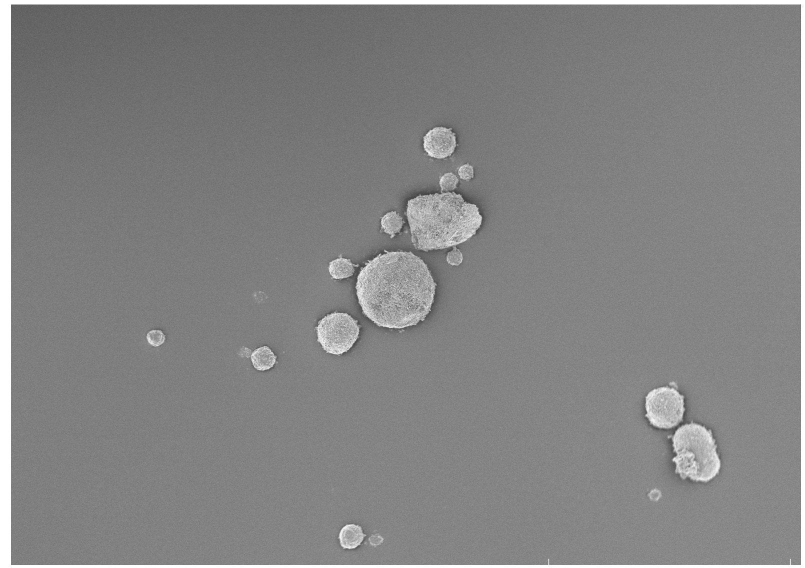
- The mixture of alumina and epoxy resin can be easily poured into a mold.
- Within the detection limitations of our instrument, two samples show very homogenous distribution of alumina powder in the nanocomposites (i.e., no evidence of sedimentation).

SEM Images



50 µm

20% v/v sample prepared using 30-40 nm alumina

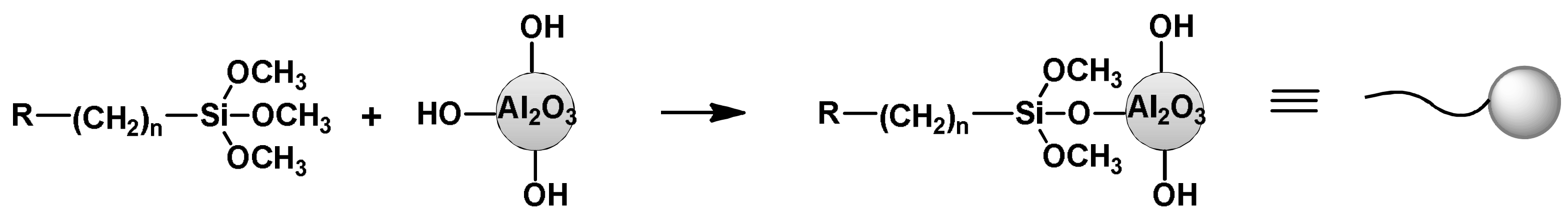


50 µm

30-40 nm alumina powder

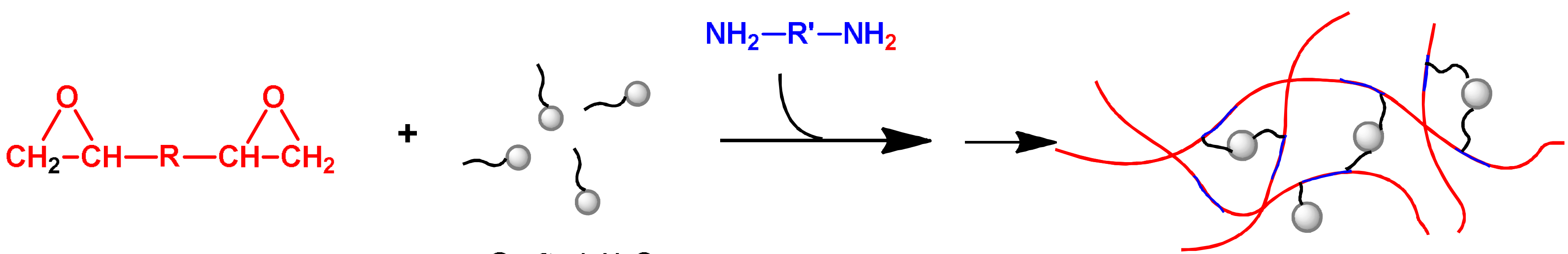
Alumina Surface Treatment

Surface Modification

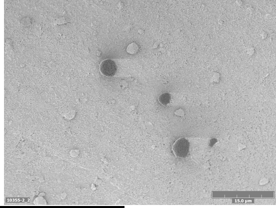


"Silane Coupling Agent" "Nanoparticle" "Grafted Al_2O_3"

3



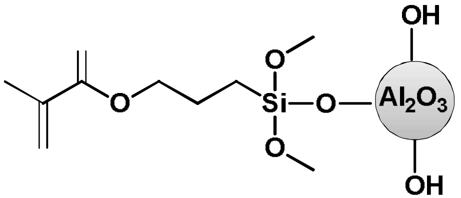
Grafted Al_2O_3

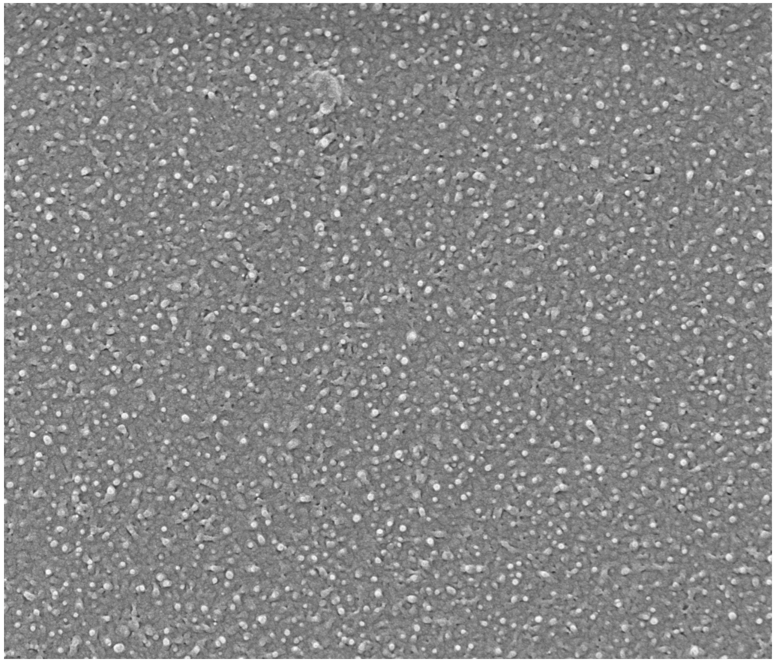


Functionalized Powder

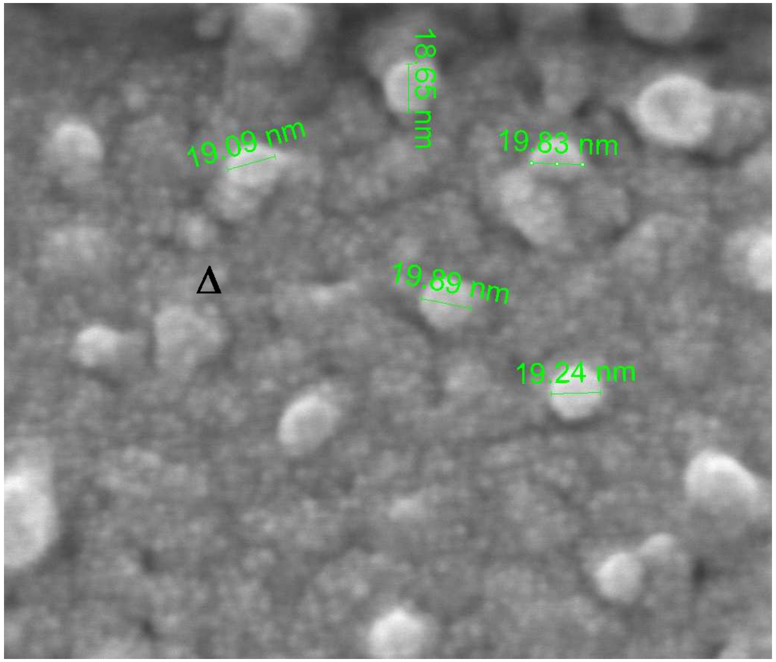
SEM Images

Images are from a 5% v/v nanocomposite containing alumina capped with MPTMS



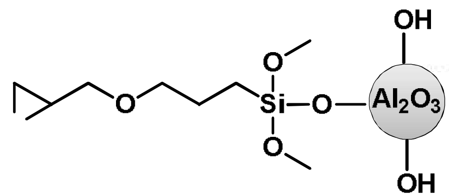


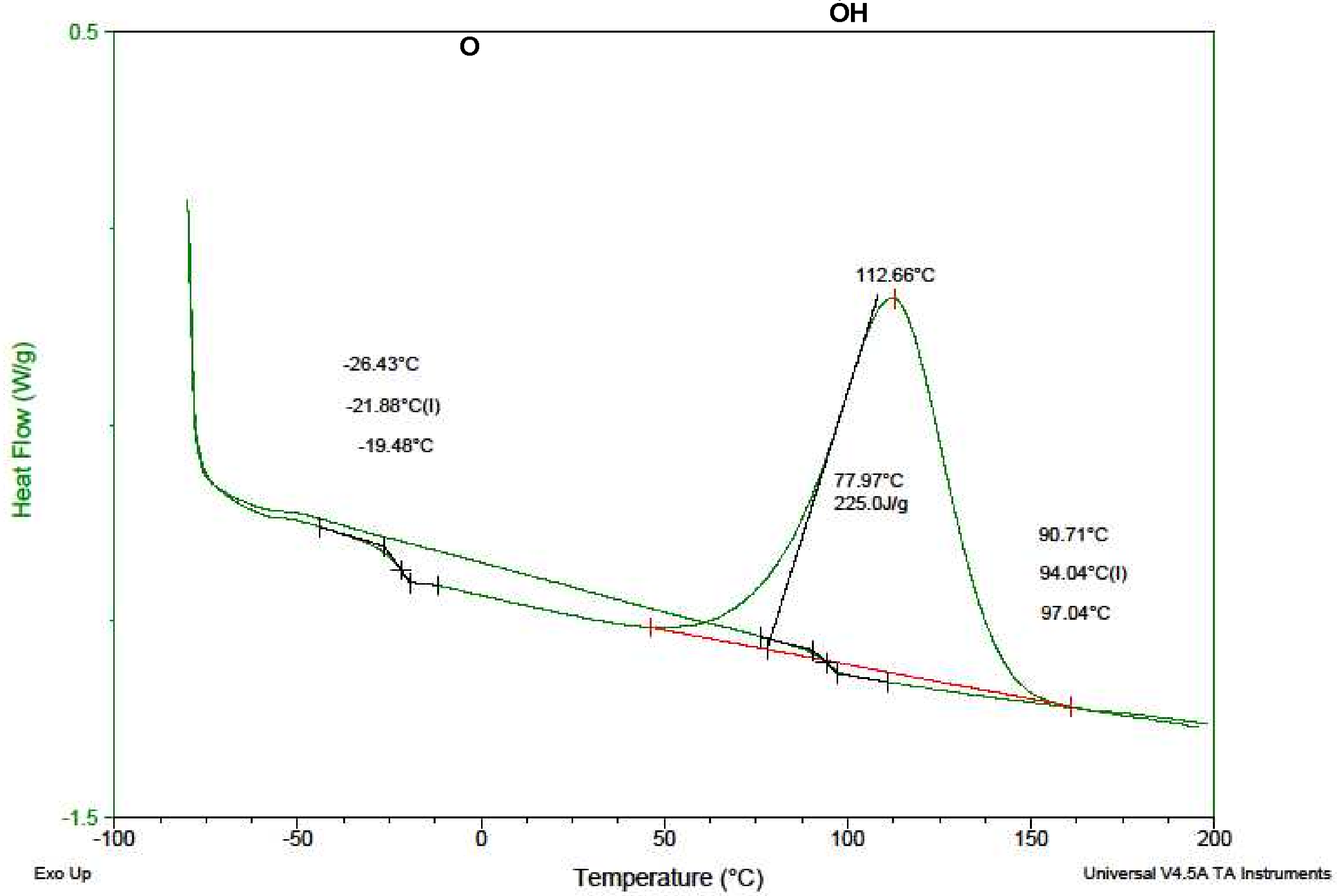
1 µm



100 nm

DSC (10% v/v Functionalized Alumina)





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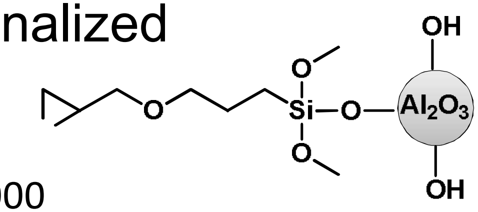
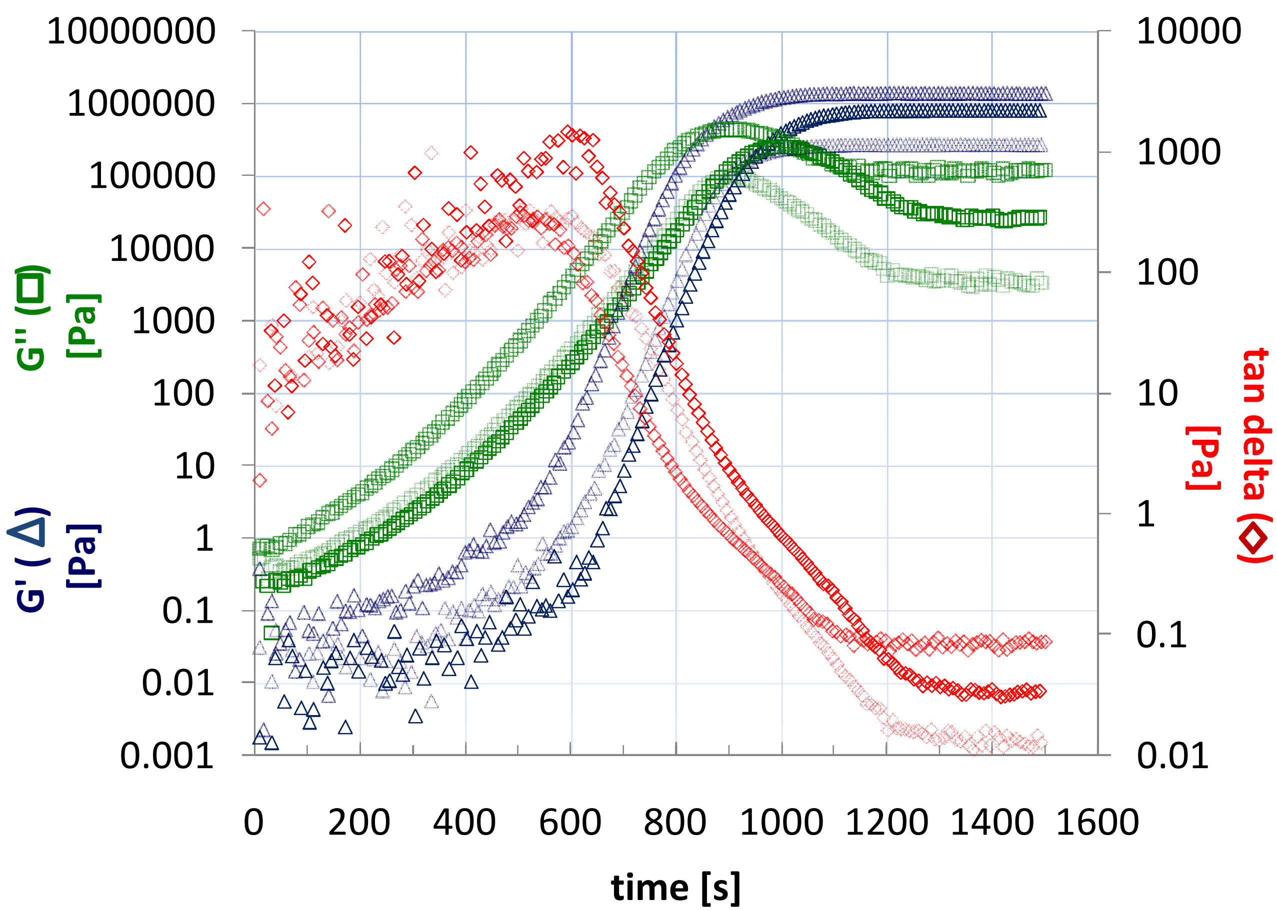
Rheology Measurements

Dark Shaded line: Control (no alumina)
Medium Shade: 10% v/v alumina loading non-functionalized
Light Shade: 10% v/v alumina loading functionalized





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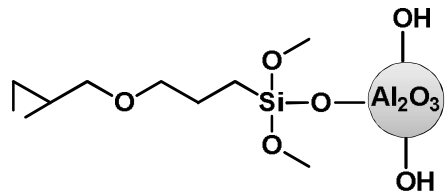
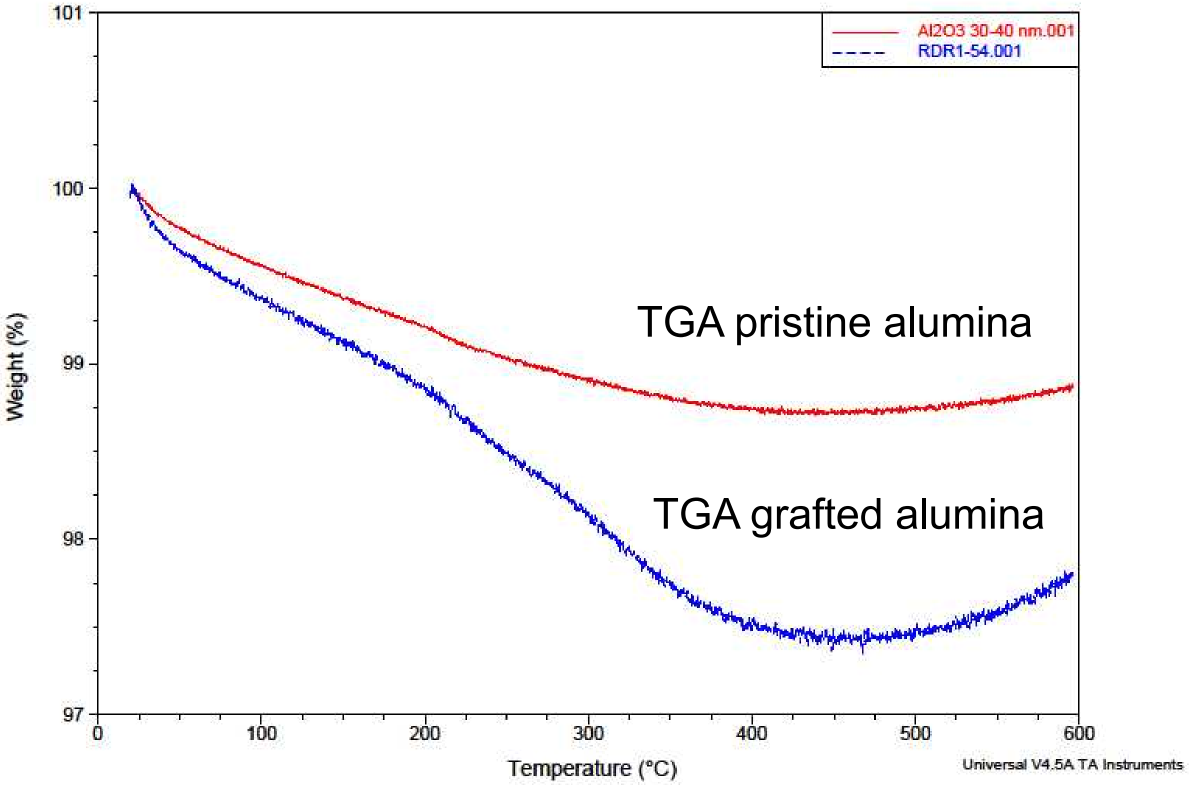
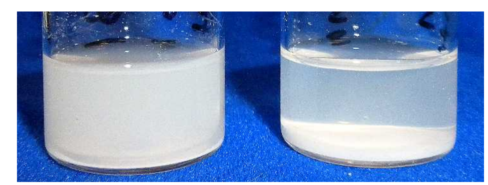
Quantification/Dispersion





O

Settling of alumina in ethanol after 24 hours



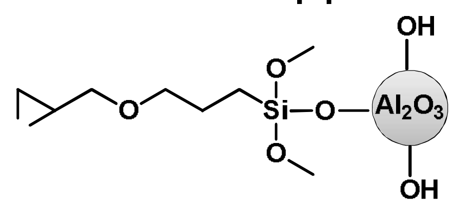
Treated Aumina

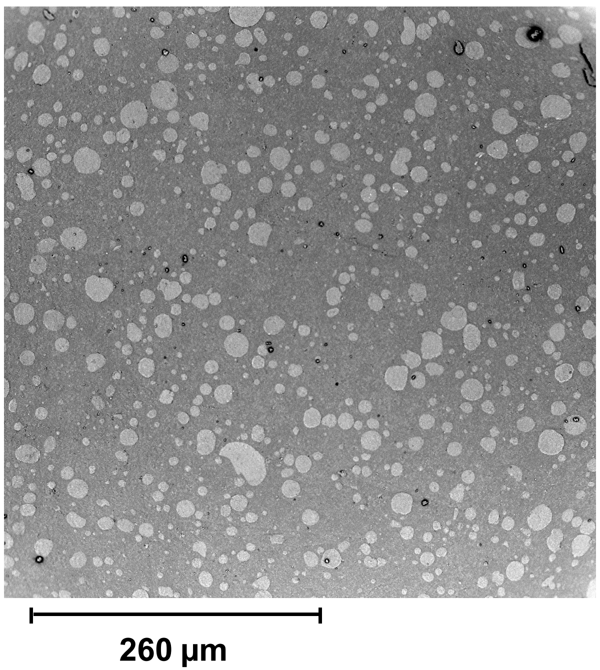
Pristine Alumina

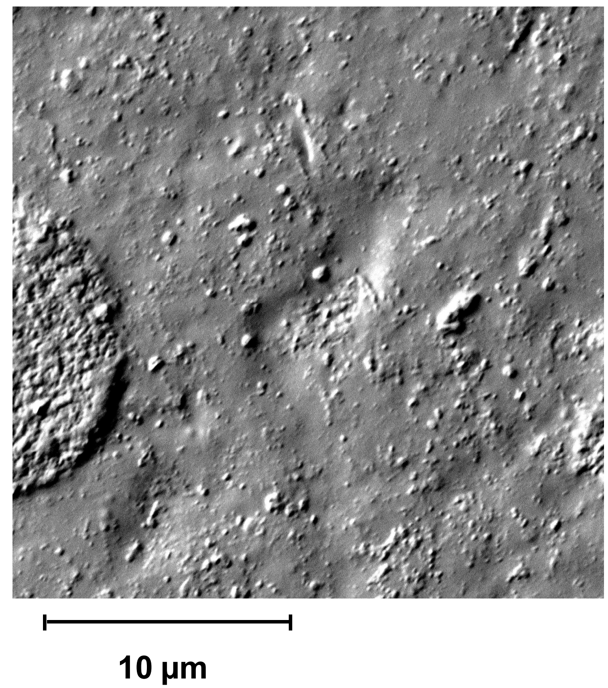
Grafted amt. silane = 1.2 μmol/g

SEM Images

10% v/v Nanocomposite made with alumina capped with 3-glycidoxypropyl)trimethoxysilane







Conclusions

- Alumina enhances the thermal, acoustic, and dielectric properties of an epoxy based polymer matrix.
- Alumina increases the viscosity of the polymer matrix prior to curing but remains pourable at high loading (25%).
- The high specific surface area of nanoalumina filler 44 m^2/g leads to aggregation outside of the nano-range.
- Alumina treated with a silane coupling agent leads to improved dispersion into the polymer matrix.