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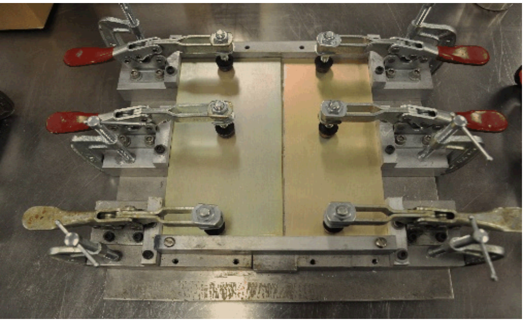

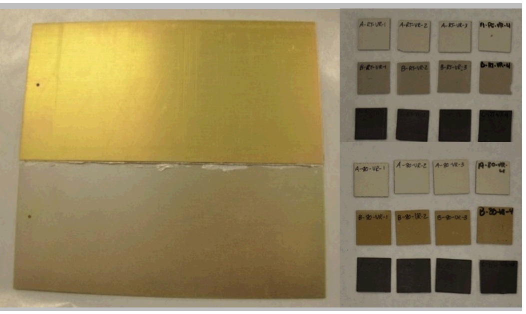

Aging of Conductively Filled Adhesives

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U.S. DEPARTMENT OF ENERGY

NNSA National Nuclear Security Administration

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Motivation

- Electrically conductive adhesives are commonly used for electronic component bonding and sealing applications that require a combination of mechanical and electrical properties.
- Uses for conductive adhesives include:
  - Cold solder for heat-sensitive components where hot soldering is impractical
  - Creation of a continuous EMR seal
  - Assembly and repair of electrical modules
  - Printed circuits
  - Wave guides
  - Flat cables
  - High frequency shields
- The adhesive must retain its mechanical strength, adhesion, and characteristic conductivity properties throughout the lifetime of the component in which they are used.
- For some applications, these conductive adhesives can be in use for decades.
- Oxidation and surface coatings of the conductive fillers in addition to degradation or oxidation of the polymer binder can lead to loss of electrical conductivity and/or mechanical properties.
- To ensure that these prerequisites are being met, accelerated aging studies are in progress with representative adhesives.

Approach

- Three representative conductive adhesives were chosen for the accelerated aging study:
  - Loctite Ablestik 2902 (Ag-filled epoxy)
  - Loctite Hysol KS0004 (Ag-filled epoxy)
  - Parker Chomerics Tecknit 72-00035 Con/RTV-Ni (Ni-filled silicone)
- Single overlap shear, volume resistivity, and dynamic mechanical thermal analysis (DMTA) test samples were prepared to monitor changes in:
  - mass
  - adhesion
  - conductivity
  - glass transition temperature
  - shear modulus

Approach- Accelerated Aging

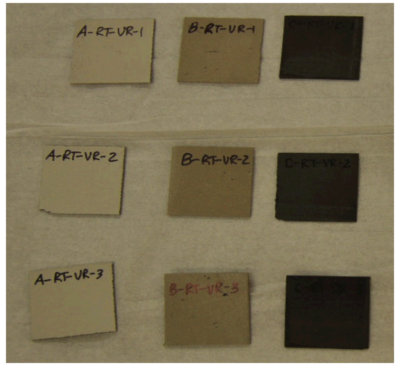
- Thermal accelerated aging studies at 80° C constituted a major part of this program.
  - The samples were aged in an air circulating oven in uncovered trays.
  - The accelerated aged samples were removed from the oven at pre-determine intervals for various tests.
  - Control samples were aged in laboratory conditions (22-24° C) in covered trays.

- The information gained from this study will provide the technical basis evidence that operational life and service requirements are being met.

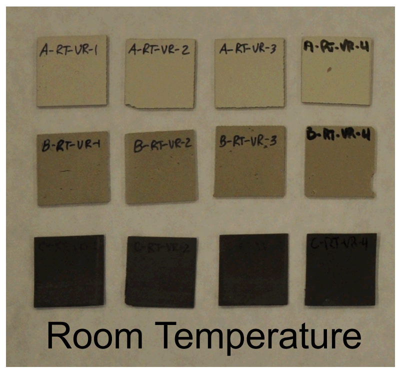
Visual Changes Due to Accelerated Aging

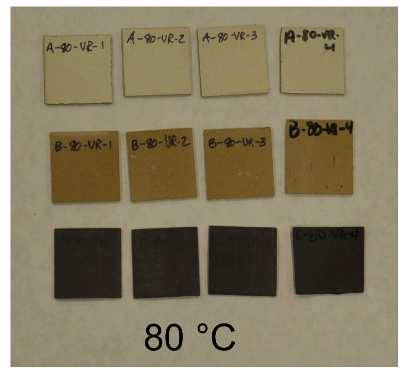
A = Ablestik 2902
B = Hysol KS0004
C = Tecknit 00035 RTV-Ni

0 weeks

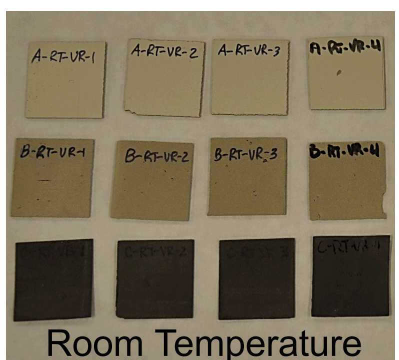


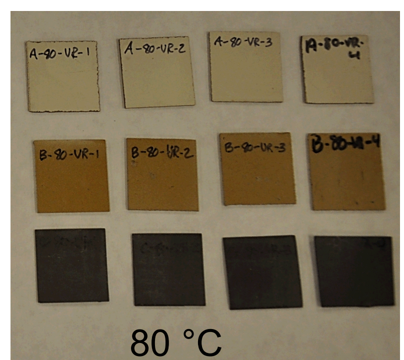
12 weeks



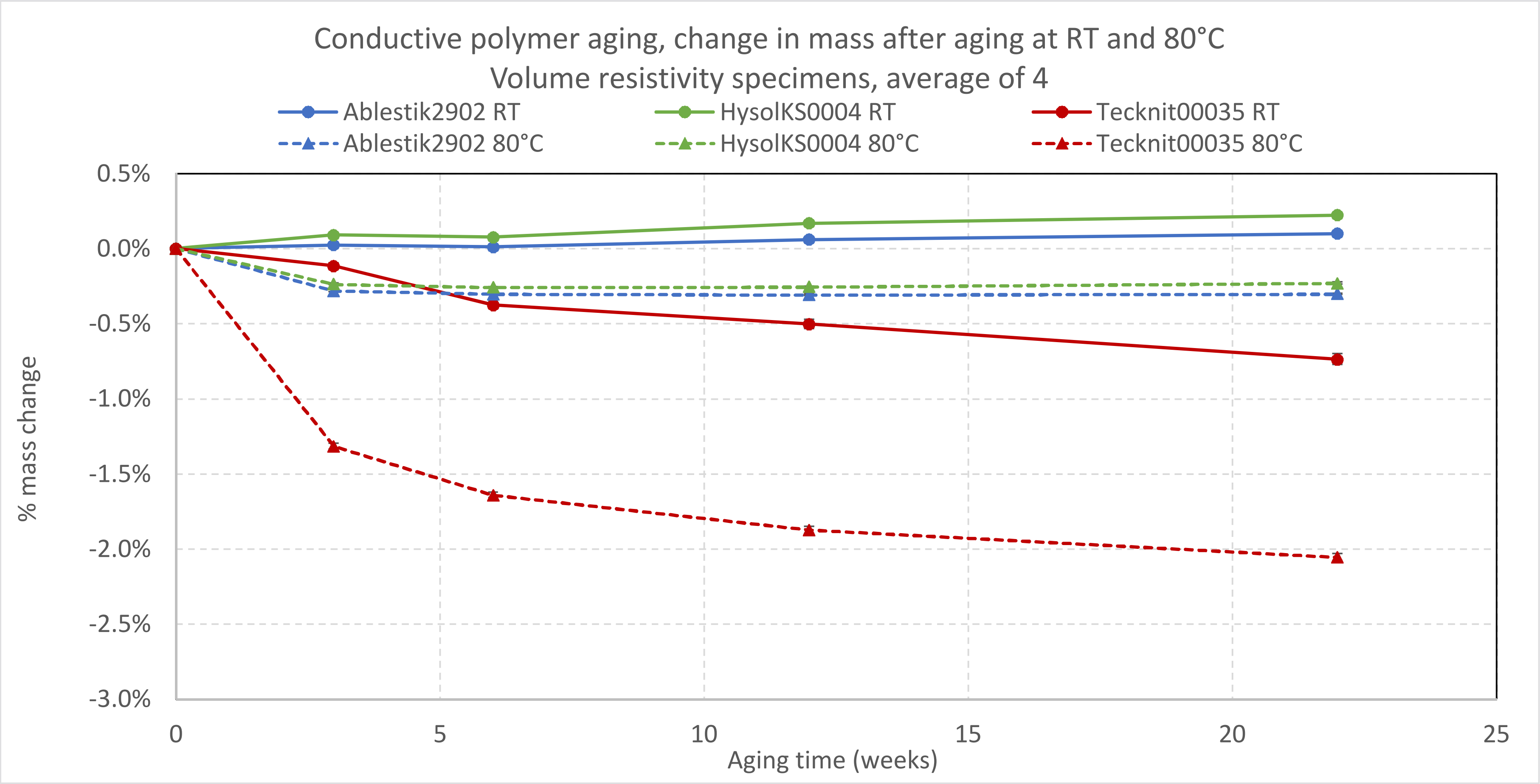


24 weeks





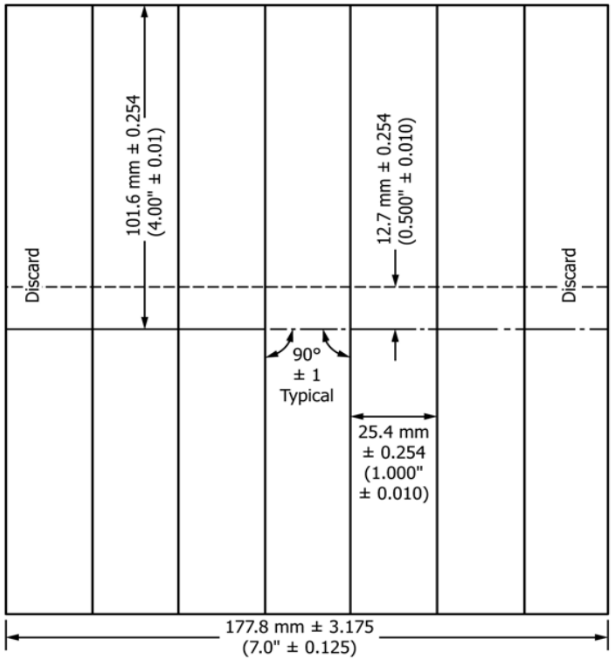
Mass Change Due to Aging

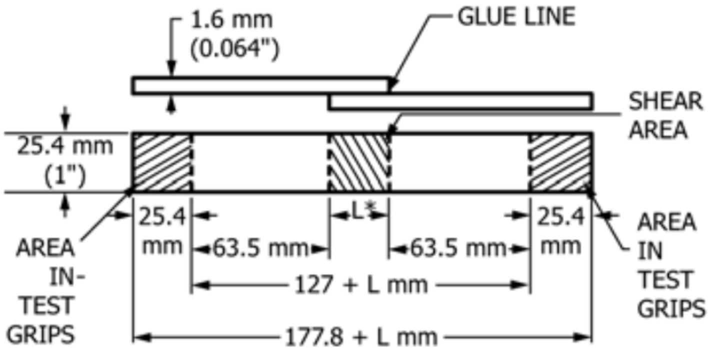


At 22 weeks both Ablestik 2902 and Hysol KS0004 samples at RT show a slight increase in mass, possibly due to the adsorption of moisture. Ablestik 2902 and Hysol KS0004 at 80°C show an initial decrease in weight due to residual solvent loss and then show a fairly consistent mass for 22 weeks. Tecknit 00035 shows a decrease in mass loss, especially at elevated temperature.

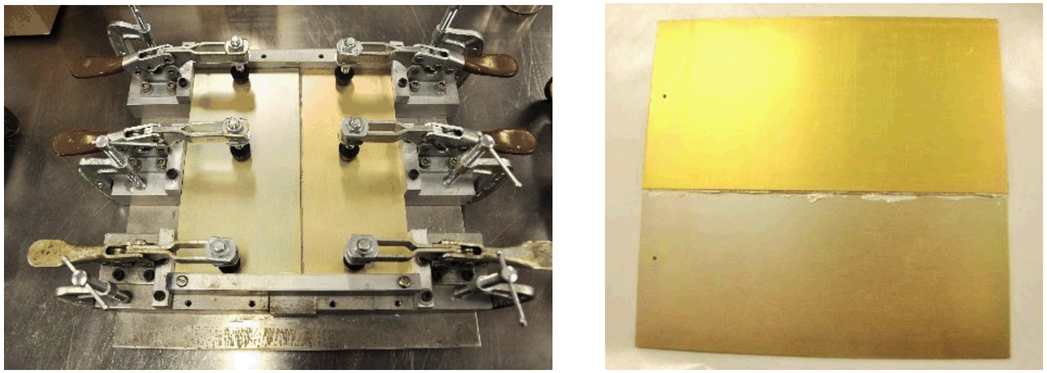
Overlap Shear

- Overlap shear samples were prepared using the guidance of ASTMD1002-10
- Aluminum panels were coated with Alodine before bonding



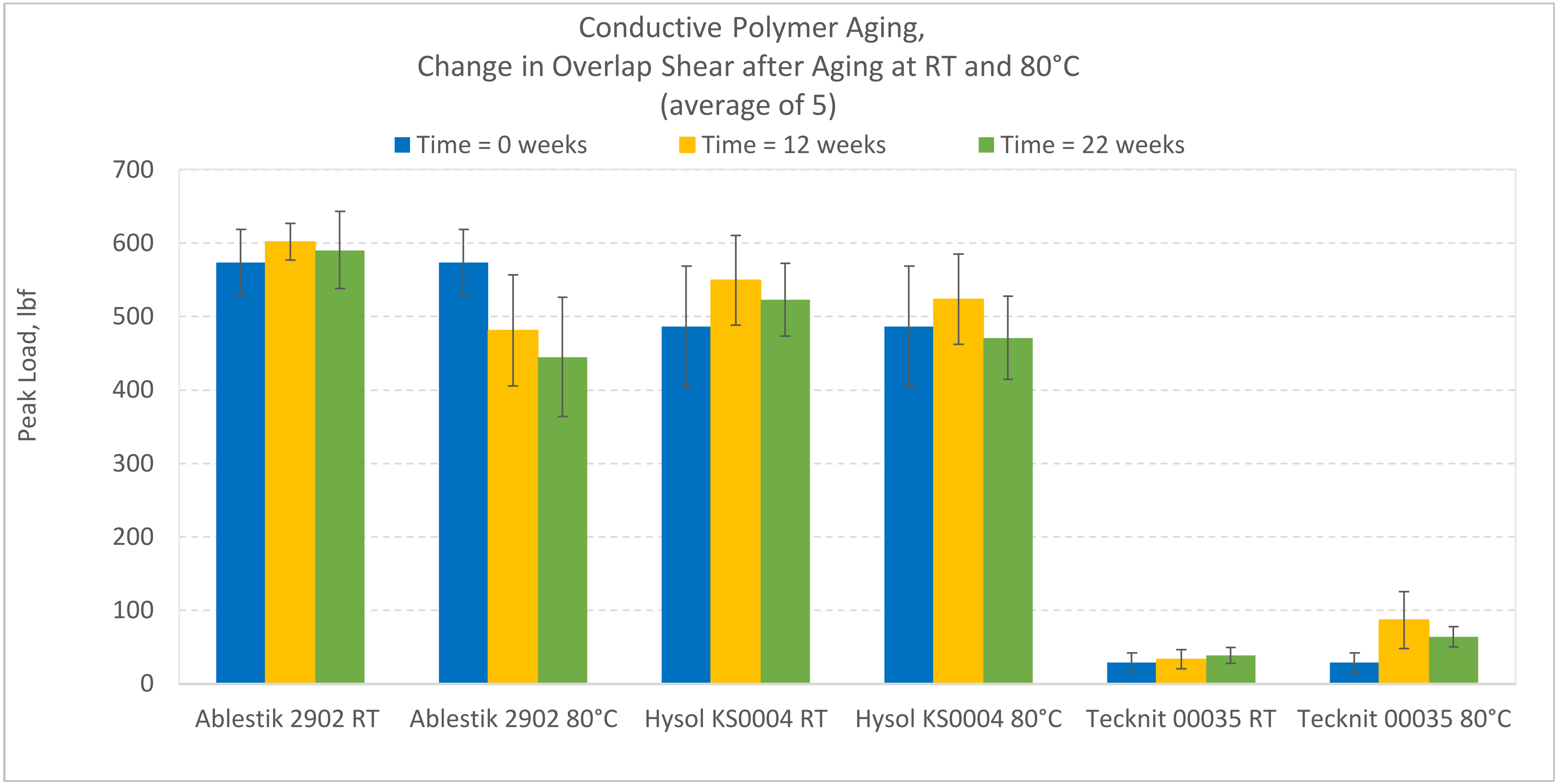


Form and Dimensions of Test Specimen. Picture taken from ASTM D1002 with dimensions used in study



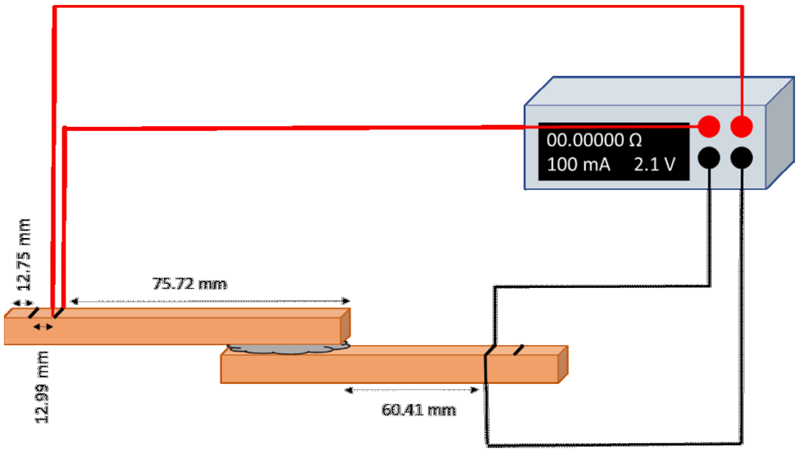
Preparation of samples *(a)* overlap shear samples being prepared using Loctite Ablestik 2902, *(b)* cured overlap shear panel which will be cut to obtain 5 lap shear samples that will be aged at elevated or room temperature.

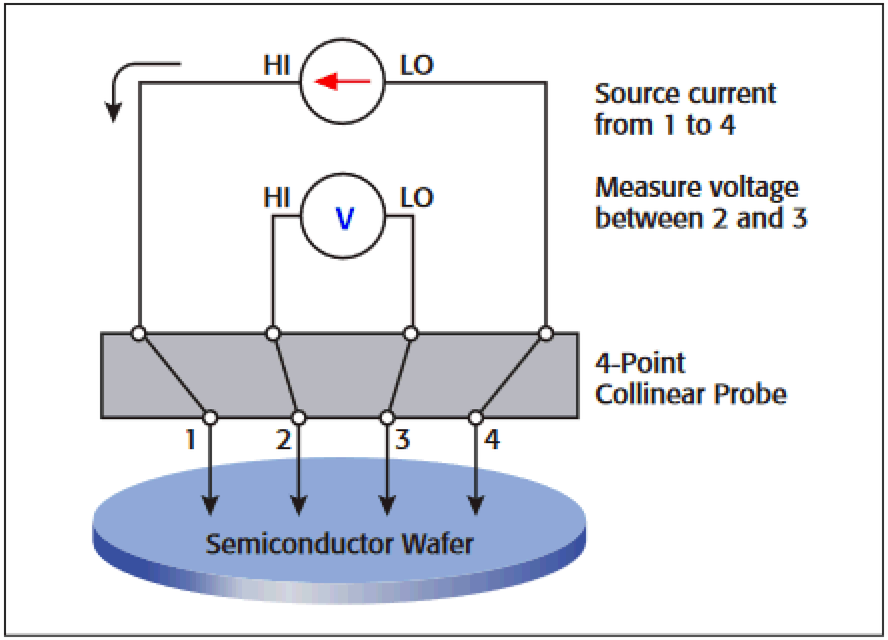
Overlap Shear



For Ablestik 2902 the overlap shear strength is decreasing as the material is aged. The overlap shear strength remains relatively consistent for Hysol KS0004 for both RT and 80°C samples. Tecknit 00035 shows an increase in shear strength which could indicate further cure at elevated temperature.

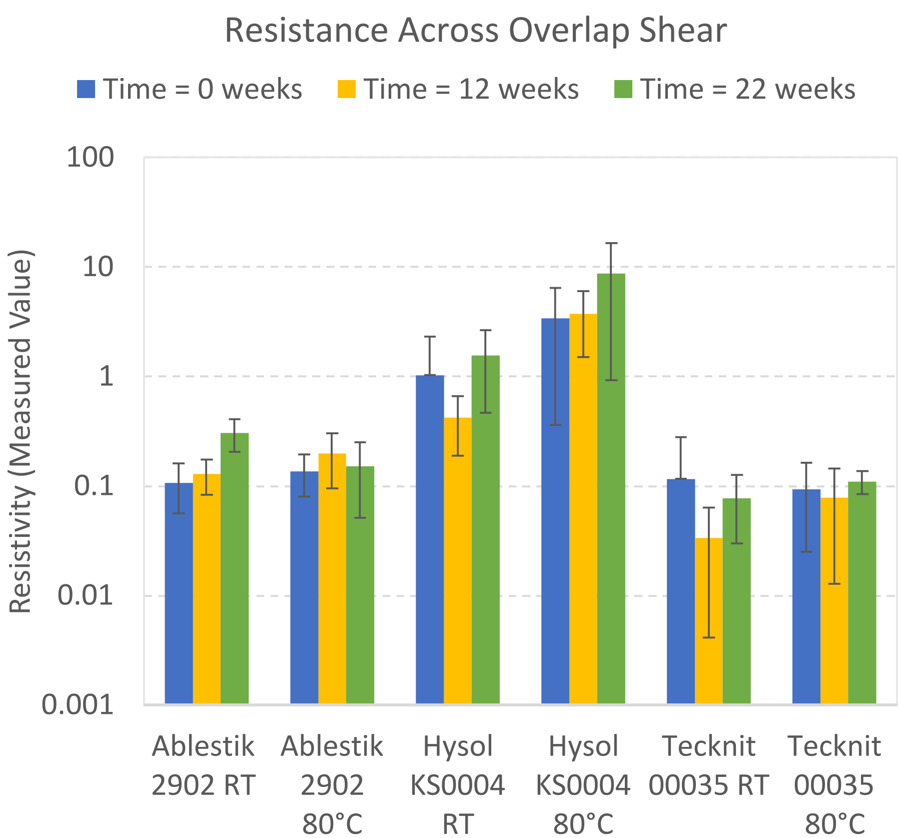
Volume Resistivity – Overlap Shear and Four-Point Probe

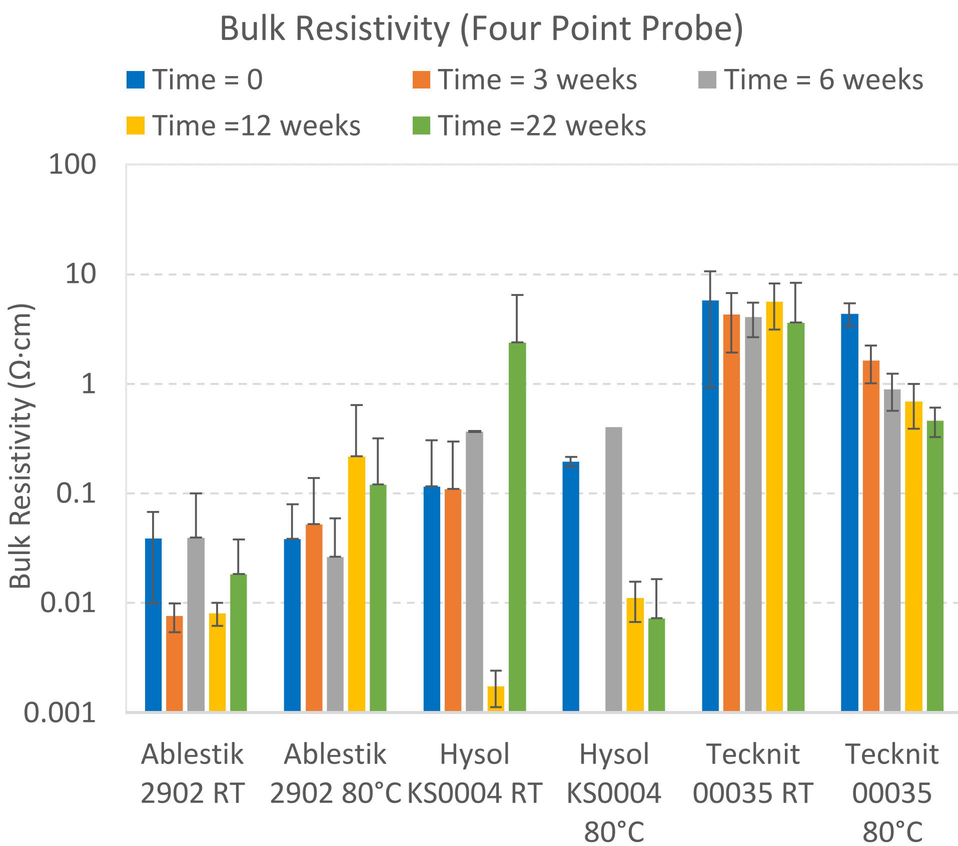




Cartoon drawing depicting overlap shear resistivity measurement setup (left) and four-point probe setup (right).

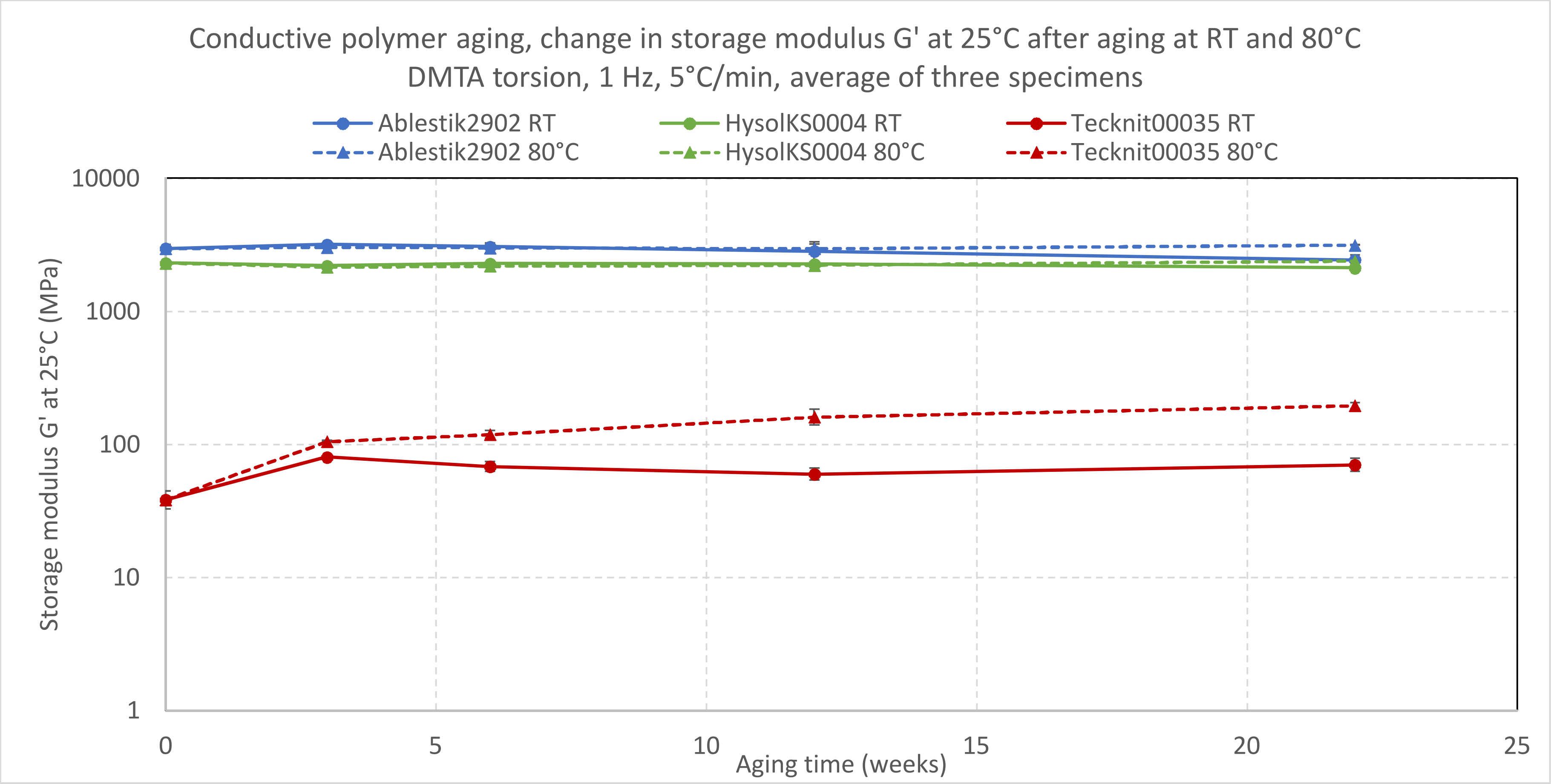
Volume Resistivity –
Overlap Shear and Four-Point Probe





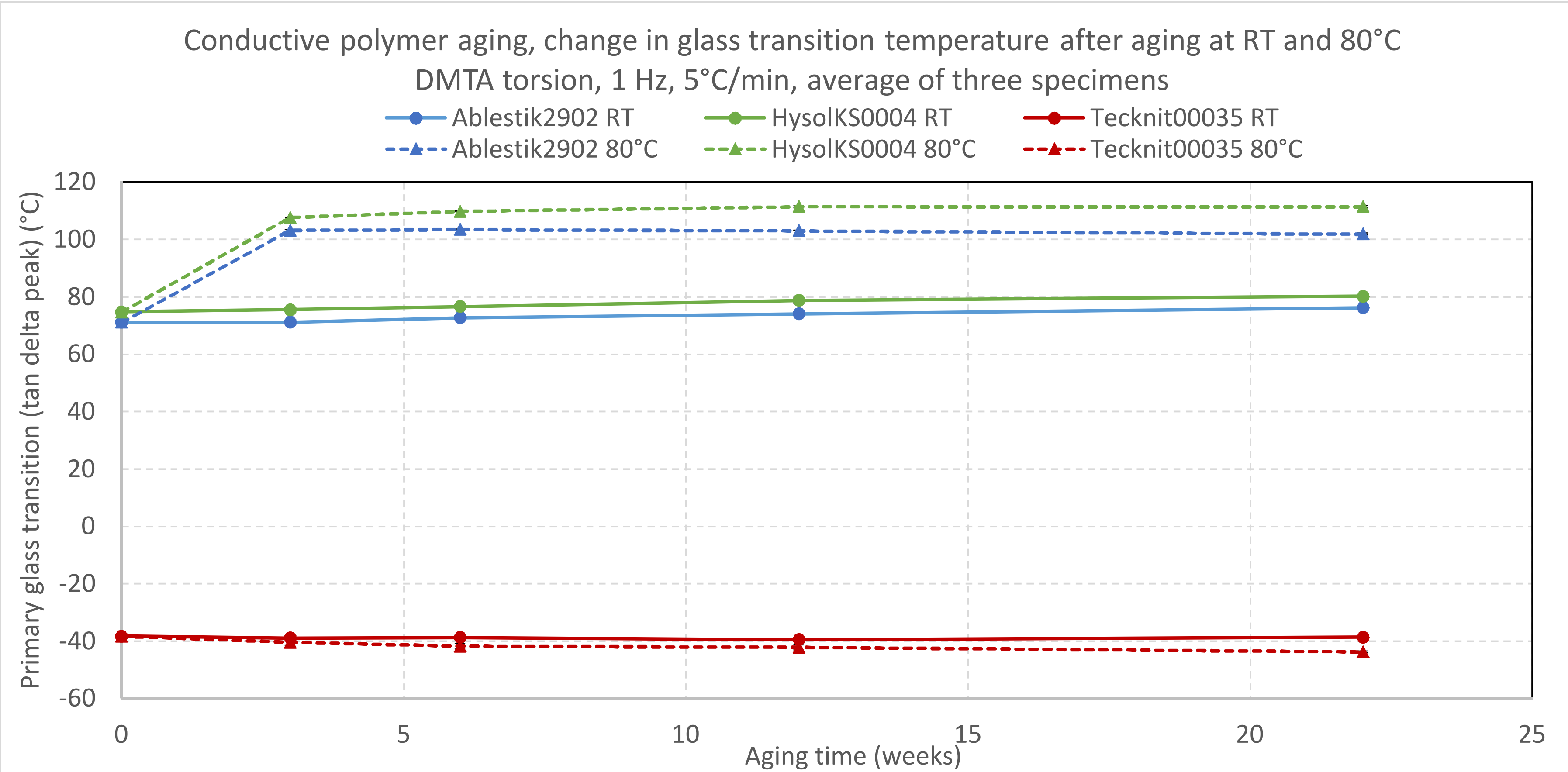
At 22 weeks Ablestik 2902 shows a consistent resistance across lap shear samples but a slight increase in resistivity for four-point probe samples at elevated temperature. Hysol KS0004 samples at RT show a slight increase in resistance for lap shear samples and a decrease in bulk resistivity for four-point probe samples. Tecknit 00035 shows a consistent resistance across lap shear samples but a decrease in resistivity for four-point probe samples at elevated temperature.

DMTA – Storage Modulus



At 22 weeks both Ablestik 2902 and Hysol KS0004 samples at RT show a consistent storage modulus. Ablestik 2902 and Hysol KS0004 at 80°C show a slight increase suggesting an increase in stiffness with aging. Tecknit 00035 shows a significant increase in G' upon further curing and especially at elevated temperature.

DMTA – Tg



At 22 weeks both Ablestik 2902 and Hysol KS0004 samples at RT show a fairly consistent Tg. Ablestik 2902 and Hysol KS0004 at 80°C show an initial increase suggesting that we may be changing the completion of the cure at elevated temperature. This increase levels out and is consistent after the initial change in Tg. Tecknit 00035 does not show a significant increase in Tg upon further curing suggesting the cure is complete at room temperature. The Tg is also consistent over 22 weeks at both room and elevated temperatures.

Conclusions

- Hysol KS0004 exhibits a discoloration with aging at 80°C that could be due to the oxidation of the Ag filler. This effect could be contributing to an increase in resistivity with time and upon aging.
- Tecknit 00035 exhibits the highest mass loss with aging at 80°C. Tecknit also shows an increase in storage modulus over this time.
  - Likely the two are related, as the material loses mass it is getting stiffer
- The resistivity remains low (or mostly consistent) for all materials over the 22-week aging period
- A more complete picture will be given at the completion of the study (48 weeks)

Next Steps/Considerations

- Validate accelerated test data by using long-term non-accelerated aged materials such as those used in parts.
- Use accelerated test data and long-term non-accelerated test data for predictive modeling.
- When does the material property change sufficiently to qualify as having “failed”? (i.e. when has the material property degraded enough that a performance requirement cannot be met?).