Study Committee D1

Materials and Emerging Test Techniques

Paper D1-PS2-10498

AGEING STUDY ON GLASS FIBER COMPOSITE ROD OF SILICONE RUBBER INSULATORS

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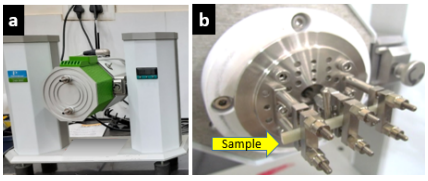
Motivation

- Silicone Rubber Insulators (SRI) are widely used in electrical transmission network. Failures of SRI are costly and disruptive to reliable service
- Our experience of failed insulator showed poor quality of insulator rod can result in failures. There are limited tests available for quality evaluation of composite rod
- Evaluation of composite rod using Dynamic Mechanical Analysis (DMA) can help in quality evaluation during manufacturing and before procurement of the insulators

Method and Measurement Parameters

- Dynamic Mechanical Analysis (DMA) was used for analysis of composite rod from insulators
- In DMA, a small cyclic stress is applied to the sample and response is measured as a function of temperature, frequency and time
- Measured properties: storage modulus (E') and loss modulus. Peak of loss modulus indicates 'glass transition temperature' (Tg)
- Dual cantilever assembly was used for DMA measurement
- Measurement parameters: Frequency – 1 Hz and Applied strain – 0.05 %

Figure. a) DMA instrument and b) dual cantilever assembly with sample



Sample Details

- Composite rod samples were collected from various insulators with different service history
- Specimen were cut from the composite rod with a precision saw using diamond cutting blades
- Sample size – 50 mm x 4 mm x 2 mm in length, width & thickness

Sample Details

Figure. Composite rod specimen for DMA test

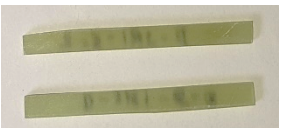


Table: Table below shows sample history of the analyzed samples

Sample Name	Service History
Type - 1 failed	Failed insulator after 10 years of service (Make - 1)
Type - 1 from Store	Not in use insulator of same batch (Make - 1)
Type - 2	Insulator with good service history (Make – 2)
Type – 3	New insulator used for ageing study (Make – 3)

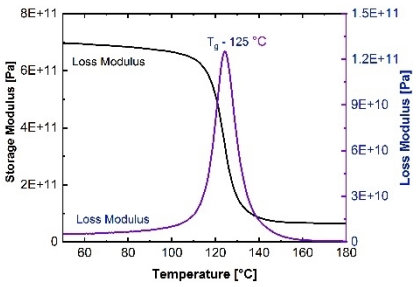
Experimental Detail: Ageing

- The composite rod samples were immersed in 0.5 M Nitric acid solution for 12 months period
- Samples were removed from ageing solution every 30 days and dried at 80 °C for 1 hour before DMA measurements

DMA Measurements

- Below figure shows DMA result of storage modulus and loss modulus as a function of temperature
- Peak of loss modulus at 125 °C indicate glass transition (Tg) temperature of composite rod

Figure. DMA result of composite sample



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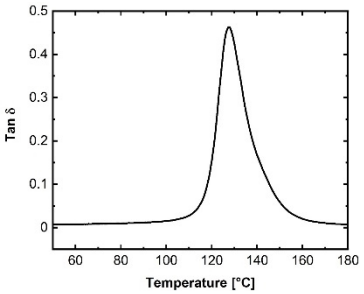
DMA Measurements

- DMA measures the response of the resin matrix from the composite as it is the mobile phase in composites
- The ratio of loss modulus/storage modulus gives damping factor (Tan δ) as shown in Figure below
- The Tan δ peak also indicate the Tg
- Glass fibers in composite are in solid state and does not contribute to the Tan δ peak measured by DMA

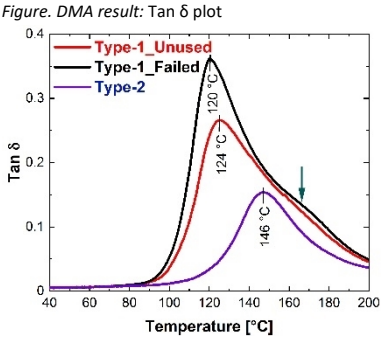
Figure. Tan δ plot



Results & Discussion: Samples with Known History

- **Poor service history**: 1) Failed sample with service life of 10 years showed high Tan δ value. Also, additional shoulder to the peak was observed and 2) Store sample (not used) showed lower value of Tan δ compared to failed sample
- **Sample with good service history:** sample having same service life as the failed sample above, showed lower Tan δ values, peak position at higher temperature and reduced peak width.

Figure. DMA result: Tan δ plot



Sample with Known History

- The shoulder observed on the Tan δ peak for failed sample with poor history is more prominent when the DMA measurements are done at lower frequency
- The DMA peak with shoulder was fitted with Gauss multi-peak function showed two relaxation processes observed at: 1) 112 °C and 2) 141 °C
- The high temperature Tan δ peak can be the result of poor interface

Figure. Tan δ measured at different frequencies

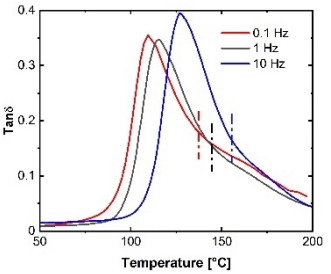
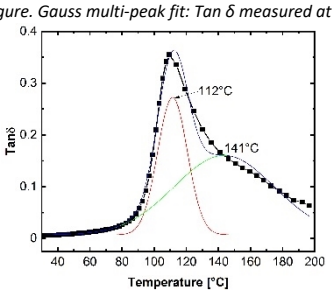
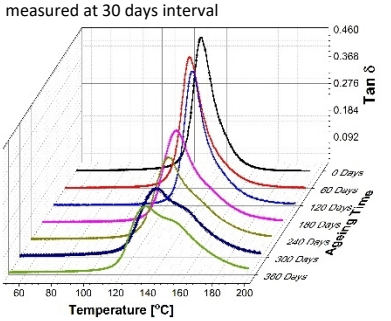


Figure. Gauss multi-peak fit: Tan δ measured at 0.1 Hz



Results & Discussion: Aged Samples

- Below plot shows Tan δ of chemically aged samples measured at 30 days interval



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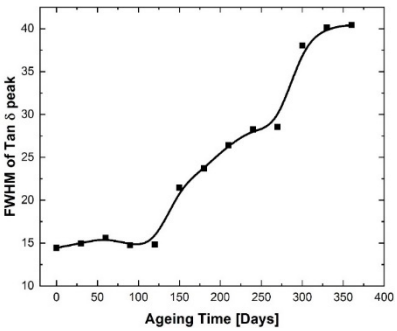
Results & Discussion: Aged Samples

- As sample ageing progresses, the Tan δ peak gets wider, with additional shoulder appearing after ageing of 240 days
- The Full Width of Half Maximum (FWHM) is plotted for the Tan δ peak, it shows three distinct zones

1. Zone-1: FWHM remains constant
2. Zone-2: due to chemical cross-linking the FWHM increases or possible initiation of interface degradation
3. Zone-3: due to degradation of interface second relaxation peak appears leading to rapid increase in FWHM

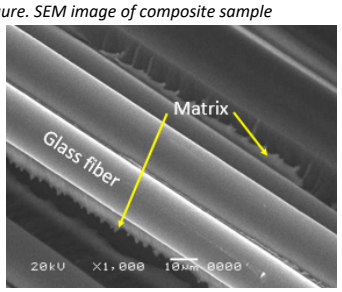
Figure. DMA result: Tan δ plot



Interface Degradaton in Compsoites

- Properties of composite materials strongly depends on inter-facial adhesion between glass fibers & resin matrix
- Poor interface can lead to inferior properties and faster ageing and degradation of composite material

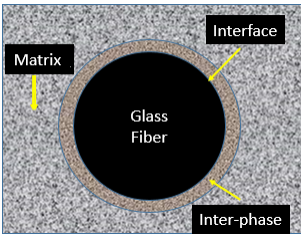
Figure. SEM image of composite sample



Interface Degradaton in Composites

- Stronger interfacial properties increases the **inter-phase** region which contributes for better mechanical and thermal properties of composite
- Highly restricted chains (lower mobility) in the inter-phase may not contribute to Tan δ signal
- Degradation of the inter-phase leads to increase in mobility of chains which makes them visible as a shoulder to Tan δ peak

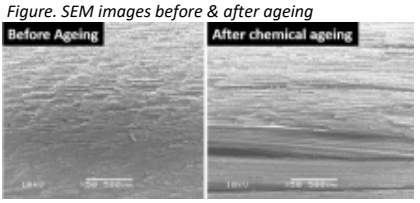
Figure. Microscopic sketch of composite



Degradation and Micro-cracks

- Chemical ageing of composite above 240 days leads to micro-cracks which is visible in SEM images

Figure. SEM images before & after ageing



Conclusion

- Mechanical dissipation (Tan δ) was analyzed using DMA for composite sample from insulators with different service history and chemically aged sample
- Low Tan δ values obtained for samples from insulators with good service history
- Samples with poor service history showed increased Tan δ values and broad Tan δ peak
- Chemical ageing confirmed the above results showing broadening of Tan δ peak as chemical degradation of composite progresses
- SEM images showed micro-cracks in chemically degraded samples confirming the degradation mechanism