Title: Effect of in-service temperature on the flexural and creep behaviour of carbon nanofiber embedded epoxy nanocomposite

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Abstract

In the present scenario, polymeric composites are becoming more popular for electromagnetic (EM) shielding applications, where internal heat generation in the component is a general phenomenon. Reinforcement of carbon nanofiber (CNF) in epoxy based polymer has drawn significant attention in this regard to enhance the shielding effectiveness. But, at the same time understanding of the mechanical performance of these materials at above ambient temperature environment is equally important to prevent any unwanted damage. Current investigation has been focused to evaluate the mechanical, thermo-mechanical and creep performance of 0.5 wt.% CNF embedded epoxy (CNF/EP) nanocomposite at elevated temperature environments. The nanocomposite exhibits 18% higher strength and 12% higher modulus over neat epoxy, when testing was done at room temperature. However, the rate of strength deterioration was found to be more rapid in case of the nanocomposite than neat epoxy, as temperature rises. Eventually at 90 °C, the nanocomposite shows a negative reinforcement effect due to extensive interfacial debonding. This fact has also been supported by the thermo-mechanical behaviour of both materials, which suggests a higher reinforcement efficiency of the nanocomposite in the early glassy state. Ambient temperature (30 °C) creep test suggests a significant resistance towards creep deformation of the nanocomposite over neat epoxy. However, beyond a critical temperature, the nanocomposite yields a relatively poor creep performance, suggesting the need of suitable interfacial engineering to make these materials more durable under elevated temperature environments.
Effect Of In-service Temperature On The Flexural And Creep Behaviour Of Carbon Nanofiber Embedded Epoxy Nanocomposite

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Size does matter

Increased surface area on nanoparticles

These nanocomposites are very often suggested to be the suitable material for EM shielding applications, where during its service substantial increment in its temperature may take place due to continuous exposure to EM waves.
Carbon Nanofiber

- Ultra-high strength ~ 3 GPa
- High Modulus ~ 240 GPa
- High aspect ratio ~ 2000
- Melting point ~ 3652-3697 °C
- Density ~ 1.9 g/mL at 25 °C

Epoxy

Properties
- Tensile strength ~ 55-70 MPa
- High Modulus ~ 2.5-4 GPa
- Tensile elongation ~ 1.5-3%
- Melting point ~ 3652-3697 °C
- Density ~ 1.1-1.2 g/mL at 25 °C

Applications
- Structural Composites (Aerospace, Marine, Infrastructure etc.)
- EM shielding applications
- Paints and coatings
- Electrical systems and electronics
- Density ~ 1.1-1.2 g/mL at 25 °C

Resin: diglycidyl ether of Bisphenol A (DGEBA)
Hardener: Triethylene tetra amine (TETA)
Preheating of epoxy at 70 °C and 400 rpm for 30 mins.

Addition of required amount of CNF to epoxy and stirring at 1000 rpm and 70 °C for 3 hrs.

Sonication of CNF/epoxy suspension at 70 °C for 1 hr.

Vacuum degassing for 18 hrs.

Addition of hardener to CNF/epoxy suspension followed by pouring it into moulds.

Vacuum degassing of the filled moulds for 15 mins.

Final samples after removal from the moulds.

Sequence of processing steps involved in fabrication of CNF/epoxy nanocomposites.
1. Flexural Testing in 3-point bending mode at 1mm/min.
   - Effect of CNF
   - Effect of in-service temperature (30, 50, 70 and 90 °C)

2. Dynamic Mechanical Thermal Analysis in 3-point bending mode at 1Hz frequency in the temperature range of 40-200 °C with a heating rate 5 °C/min.

3. Differential Scanning Calorimetry in the temperature range of 25-150 °C with a heating rate 5 °C/min.

4. Flexural Creep at various temperatures (30, 50, 70 and 90 °C)
Flexural stress-strain plots for (a) EP and (b) CNF/EP composite at various temperatures.

The effect of service temperature is more prominent on the stress-strain response of the nanocomposite.
Strength and Modulus


At RT
Strength:  +18%
Modulus:  +12%

Almost same strength and Modulus at 70 °C

At 90 °C
Strength:  -23%
Modulus:  -11%
Variation in (a) storage modulus ($E'$), (b) loss modulus ($E''$) and (c) loss tangent ($\tan \delta$) with temperature for EP and CNF/EP nanocomposite.
Creep curves for neat epoxy (EP) and 0.5\% CNF reinforced epoxy (CNF-EP) at (a) 30 °C, (b) 50 °C, (c) 70 °C and (d) 90 °C.
• Addition of 0.5% CNF in epoxy resulted in flexural strength and modulus enhancement by 18% and 12% respectively, when tested at RT.
• With increase in testing temperature, the flexural performance CNF/Ep composite decreases at a higher rate than neat Ep.
• At 90 °C, a negative reinforcement effect in terms of flexural performance was noticed. The strength and modulus of the nanocomposite were 23% and 11% lower than neat Ep.
• Differential thermal expansion and hence the residual interfacial stress developed reduces the stress transfer efficiency from the polymer to the CNF.
• From DMA, it was confirmed that the nanocomposites exhibits lower Tg than neat polymer.
• Low temperature creep testing suggested the superior performance of the nanocomposite over neat Ep, still with time this superiority is reduced gradually.
• However, at higher temperature, due to temperature induced damage at the interface, the creep performance of the nanocomposite is severely affected.
• National Institute of Technology Rourkela (TEQIP 104-FSD),
• Naval Research Board, India (NRB-383/MAT/16-17)
Research Interest of our group

Development of nanophased FRP composites and their environmental durability studies

Materials
- CNT
- CNF
- Graphene
- Glass fiber
- Carbon fiber
- Epoxy

Environmental studies
- Effect of in-situ service temperature
- Effect of thermal conditioning and cycling
- Hygrothermal conditioning and cycling

Characterization Techniques
- Flexural testing
- DMTA
- DSC
- FTIR
Thank you...