MASTERBOND®

CASE STUDY

EP114: Utilized to produce nanocomposites via vacuum infiltration

EP114: Utilized to produce nanocomposites via vacuum infiltration

Overview of EP114

Master Bond EP114 is a two-component nano silica filled epoxy system which offers excellent dimensional stability and a very high glass transition temperature (Tg) > 200°C upon curing. Its ultra-low initial mixed viscosity (500–1500 cps at room temperature) can be further reduced by preheating to a slightly higher temperature, making EP114 ideal for vacuum infiltration to produce dense nanocomposites, as shown in the case study below.

Application

Graphene nanoplatelets (GNP) and boron-nitride nanoplatelets (BNNP) show promising application prospects in electric double-layer capacitors due to their large surface area. However, they tend to agglomerate due to van der Waals forces and π-π interactions, leading to a lower surface area and ultimately a reduction in the specific capacity of capacitors. Agglomeration can be prevented by fabricating GNP and BNNP into three-dimensional foams, but the resulting foams often show insufficient mechanical strength and require reinforcement. Vacuum infiltration with an epoxy resin provides a reinforcement method that does not compromise the toughness of nanocomposite foams, but the chosen epoxy resin must have an appropriately low viscosity to penetrate the foam. Master Bond EP114 has a sufficiently low viscosity for vacuum infiltration, so researchers at Florida International University used it to investigate interactions between freeze-dried GNP/BNNP foams and EP114 to understand its infiltration behavior.

Key Parameters and Requirements

Infiltration of EP114

After creating GNP/BNNP foam nanocomposites, the authors used EP114 to infiltrate the foams. The authors separately heated the two components of EP114 (epoxy and hardener in a 50:40 ratio) at 100°C for 1 hour before hand-mixing them for 60 seconds and then pouring the mixture onto the GNP or BNNP foam. Due to the low viscosity of EP114 at 100°C and because this is well below its curing temperature, the authors selected 100°C for their vacuum infiltration experiments. Next, a vacuum of 1 Pa was applied for 1 hour to ensure complete penetration of EP114 throughout the foam structure.

Cure Schedule

Although a typical cure schedule is 2–3 hours at 250°F (~121°C), followed by 5–8 hours at 300 °F (~149°C) with a 2-hour or longer post-cure at 350°F (~176°C), several variations are possible, as demonstrated in this study. After infiltration, the composites were removed from the mold and cured using the following schedule:

1. Isothermal hold for 30 minutes at 80°C to remove moisture
2. Heating at 1°C/minute up to 125°C
3. Isothermal hold for 3 hours at 125°C
4. Heating at 1°C/minute up to 150°C
5. Isothermal hold for 6 hours at 150°C
6. Heating at 1°C/minute up to 200°C
7. Isothermal hold for 4 hours at 200°C
8. Slow cooling (below ~1°C/minute) until below 50°C

Results

Before using it to infiltrate the foam, the authors characterized various thermal and mechanical properties of EP114. The authors then created a variety of GNP and BNNP foams and studied the interactions between the foams and EP114. Using EP114 contact angle measurement, the authors showed that the wettability (and infiltration) of EP114 was affected by the infiltration temperature, foam composition, and foam shape.

As the GNP content increased, infiltration became more difficult due to a decrease in the porosity. In nanocomposites with low loadings of GNP, the pores were incredibly small, and even the prolonged use of a vacuum or higher temperature of 100°C could not ensure complete infiltration of EP114. At higher weight percentages, the foams that were created cracked, resulting in many non-infiltrated regions. As shown in ***Table 1***, all foams infiltrated with EP114 achieved densifications > 96%, indicating good infiltration with minor porosity. Of the infiltrated nanocomposites, those of EP114/BNNP showed the highest densities.

The infiltrated nanocomposites showed hardness values like those of neat EP114, suggesting that the porosity did not compromise the mechanical properties of the foams. The authors did observe a larger standard deviation in these values, which may suggest a heterogeneous dispersion of EP114 throughout the foam samples.

Table 1. Densification and hardness values of infiltrated GNP, BNNP, and hybrid foams. *(Data adapted from Benedetti et al. 2023.)*

Composition	Densification (%)	Hardness (Shore D)
Neat EP114	100	87.17 ± 0.3
2% GNP	99.67	87.67 ± 2.8
4% GNP	98.24	87.17 ± 2.9
6% GNP	96.87	86.83 ± 2.0
8% GNP	96.36	87.17 ± 0.6
75:25 GNP:BNNP	98.90	84.83 ± 3.8
Hybrid foam + EP114 (50:50) GNP:BNPP	99.35	87.67 ± 1.4
25:75 GNP:BNPP	99.56	86.33 ± 3.3
4% BNNP	99.87	85.83 ± 0.3

The presence of GNP and BNNP networks increased the thermal conductivity of the epoxy matrix, which indicates a connected structure within the GNP and BNNP freeze-dried foams, even at low concentrations of nanoplatelets.

This study shows that EP114 can be used to infiltrate GNP and BNNP networks to fabricate nearly 100% dense nanocomposites with improved thermal conductivity compared with neat EP114. These results were obtained without compromising the nanocomposites' hardness, suggesting that this approach may be used to fabricate various freeze-dried foams/polymer nanocomposites without compromising their mechanical properties.

References

Benedetti, L.; Orikasa, K.; Agarwal, A. The Effect of Different GNP/BNNP Foam Structures in the Infiltration of Epoxy Resin: A Fundamental Study. Polymer Composites. 2023. https://doi.org/10.1002/pc.28070.