Nano Silica Dispersion in Epoxy: Mechanical Stirring with Zirconia Ball Media

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ABSTRACT

This paper explores the efficiency of dispersing nano-silica particles in epoxy resin using zirconia ball media by mechanical stirring. Stirring speed (RPM), the time of milling and temperature are the main parameters studied at a fixed media:total silica-epoxy volume ratio. The results show that ball media and heating aid dispersion. At low stirring speed, short stirring duration is more suitable but the reverse is true at high stirring speed.

Keywords: nano-silica, epoxy resin, mechanical stirring

INTRODUCTION

Incorporation of silica fillers is commonly employed to reduce the coefficient of thermal expansion (CTE) of epoxy resins. However, the presence of aggregates poses a major challenge as a homogeneous and discrete dispersion is the ultimate. This challenge is even greater with the usage of nano-sized fillers, an issue recognized by researchers. Varied attempts have been employed to solve this issue, with differing outcomes [1 - 4]. This paper explores the efficiency of dispersing nano-silica particles in an epoxy resin using zirconia ball media by mechanical stirring. The effect of applied heat is also explored and so is the stirring speed (RPM) and the time of milling under a fixed temperature and media:total silica-epoxy volume ratio. The factors explored are based on the hypothesis that shear stress and possibly the duration of milling will have an effect on nano particle dispersion. It is possible that the process of dispersion involves (1) breaking the aggregates to discrete particles and (2) wetting of the particle surfaces by the epoxy resin.

EXPERIMENTAL SECTION

Material

Materials used are nano silica and Bisphenol-A type epoxy resin, DER331. Nano silica was prepared using a modified Stober method and has a bi-modal size distribution averaging at 9.75 nm and 30.38 nm (Figure 1).

Procedure

Nano silica dispersion in epoxy is conducted by mechanically stirring the mixture in the presence of zirconia ball media, 60-microns in diameter. The media is supplied by Revertex Malaysia Sdn Bhd. The media to total silica-epoxy volume ratio is fixed at 3:5 for all dispersion preparations.

In accordance to the interest of this study mentioned in the Introduction, the experiment is divided into 2 sections, with the first section dedicated to the comparison of nano silica dispersion, with and without zirconia ball media. For this experiment, both mixtures are stirred at 300 rpm under room temperature. The second section is a study of the effect of mixing speed and duration on nano silica dispersion with zirconia ball media present in the mixture. During mixing the temperature is maintained constant at room temperature, except for the hourly heating at 60°C, each lasting for 5 minutes.

RESULT AND DISCUSSION

A comparison of systems stirred with (Figure 2a) and without (Figure 2b) the presence of zirconia ball media shows that the ball media improves the dispersion of nano silica. Although Figure 2b shows better homogeneity in the nano silica dispersion, there are still aggregates, the smaller ones average at about 200 nm, while the bigger ones average at 3389 nm. There is the possibility that the nano silica dispersion can be further improved and the 2nd experimental section explored this by applying 60 °C heating at every 1 hour interval, for five minutes, to improve mixing and dispersion of nano silica. DER331 epoxy is at optimum viscosity at 60 °C. The results show that intermittent heating is effective in reducing the aggregate sizes, from 3389 nm to around 262 to 970 nm, varied by the stir speed and duration.

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The effect of varying shear stress and milling duration is also studied. The results show that, the stir speed and duration affects the nano silica dispersion, given that all the other conditions (zirconia ball media to nano silica-epoxy ratio and intermittent heating) remain constant. Table 1 (which is concluded from Figure 3) shows the larger aggregate size of each mixture, while Figure 4 is the SEM micrograph of each corresponding mixture, at a lower magnification, that clearly shows the trend of the particle dispersion. Table 1 and Figure 3 results have shown that shorter stirring duration is the more suitable condition for nano silica dispersion at low stirring speed (100 rpm). On the reverse, higher stirring duration is the more suitable condition to disperse nano silica at high stirring speed of 500 rpm.

CONCLUSION

Mechanical stirring with the presence of zirconia ball media improves nano silica dispersion. Intermittent heating further improves the dispersion. Given that the ball media to nano silica-epoxy ratio and temperature is kept constant, the nano silica dispersion is affected by the stir speed and duration. Aggregate sizes are also found to be affected by the same trending.

ACKNOWLEDGEMENT

The authors of this paper would like to thank Intel Technology Sdn Bhd. for the financial assistance granted to this project – Grant No. 304.PKIMIA.605336.I104.

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