

A Study on Microstructure Characteristics of TEPs-modified Adhesives

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Thermally expandable particles (TEPs) were developed by Dow Chemical Co in the early 1970's [1] and were further developed by others [2, 3]. They are particles made up of a thermoplastic shell filled with liquid hydrocarbon. On heating them, two transformations will occur. One is the softening of shell material and the other is the gasification of the hydrocarbon liquid inside it. As a consequence, the shell will expand as the gas inside it will push the softened shell from inside out causing it to grow in size [4]. When fully expanded, the growth in volume of the particle can be from 50 to 100 times [3]. Owing to this unique behaviour, TEPs are used by the industry in a wide variety of applications mainly for weight reduction and appearance improvement for thermoplastics, inks, and coatings. In adhesive bonding, TEPs have been used for recycling purposes. Moreover, TEPs might be used to modify structural adhesives for other new purposes, such as: to increase the joint strength by creating an adhesive functionally modified along the overlap of the joint by gradual heating and/or to heal the adhesive in case of damage. Nevertheless, the success of the concept of using TEPs with adhesives depends on the combination of the TEPs and the adhesive system as the resulting properties of the TEPs-modified adhesives are influenced by the mechanical, physical and chemical properties of the used TEPs and adhesives. However, there are few studies in the literature about the properties of TEPs-modified materials and in particular of TEPs-modified adhesives. In a previous study by the authors, a thermal analysis was performed in order to investigate the behaviour of TEPs while encapsulated in adhesives and it was shown that the TEPs expansion takes place in a range of temperatures, between 90 and 120 °C [4]. In this work, the fracture surfaces of the unmodified and TEPs-modified thermal specimens, as well as the dispersion and the morphology of the particles, were examined by a scanning electron microscopy (SEM) analysis.

A structural polyurethane adhesive SikaForce[®]7888, supplied by Sika (Portugal) and an epoxy adhesive Betamate[™]2098, supplied by Dow Automotive (Dow Europe, Switzerland) along with Expancel 031 DU 40 particles supplied by Expancel Nobel Industries (Sweden) were used. The TEPs-modified adhesive was mixed with a Speed Mixer (DAC 150.1 FVZ Speedmixer, Hauschild, Germany) for 60 s at 2500 rev/min. The TEPs-modified adhesives cured matrix structure was examined using a high resolution Scanning Electron Microscope with X-Ray Microanalysis: JEOL JSM 6301F/ Oxford INCA Energy 350 (Gatan Alto 2500, Tokyo, Japan) at the CEMUP laboratory (University of Porto, Portugal). Secondary electron images were collected at different magnifications.

The particles morphology before and after expansion can be seen in Fig. 1. From Fig. 2 it can be noted that TEPs-modified specimens (Fig. 2b) present a completely different structure morphology compared to the unmodified specimens (Fig. 2a). The morphology of the cured unmodified adhesive structure is a typical fracture surface appearance of a tough ductile adhesive with white shear bands which indicate a large ductility, while the morphology of the cured TEPs-modified adhesives structure is complex (cellular structure). The TEPs-modified adhesive samples showed a uniform dispersion of particles within the samples

no matter of TEPs content and adhesive. SEM images of sample at different temperatures (i.e. room temperature (RT), at maximum expansion ($T_{\max}=100\text{ }^{\circ}\text{C}$) and after thermal tests ($150\text{ }^{\circ}\text{C}$)) can be seen in Fig. 3. It can be observed that at maximum expansion temperature the TEPs cross-sections and the hollow voids are considerable larger than at RT, indicating the evidence of TEPs expansion. Finally, above the temperature at maximum expansion ($T_{\max}=100\text{ }^{\circ}\text{C}$) the TEPs start to deteriorate (degradation and/or explosion of TEPs occur) as can be seen in Fig. 3c.

References:

- [1] D.S.J. Morehouse, R.J. Tetreault, US Patent 3615972 (1971).
- [2] Matsumoto Yushi-Seiyaku Co. L., <http://www.mtmtys.co.jp>.
- [3] Expancel Home page, <http://www.akzonobel.com/expancel/>
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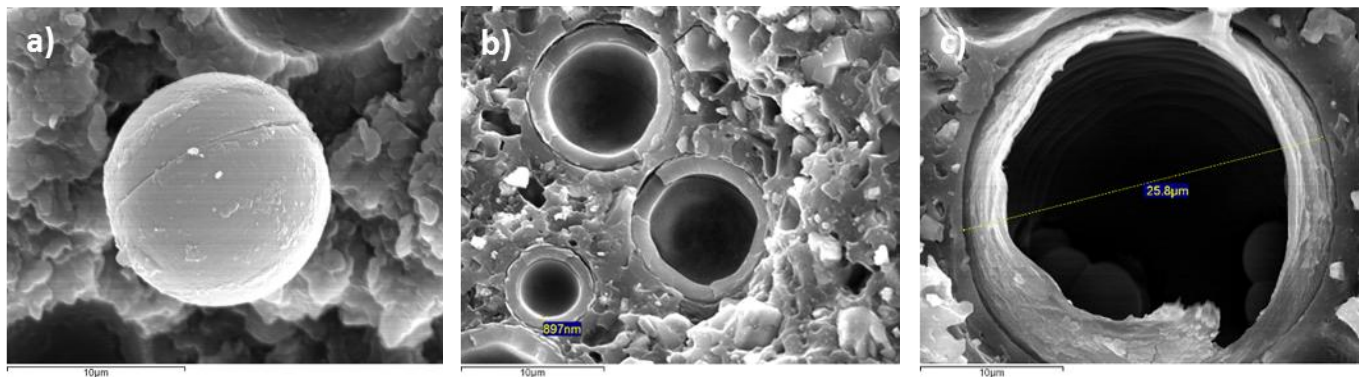


Figure 1. SEM images of TEPs morphology: a, b) before expansion; c) after expansion.

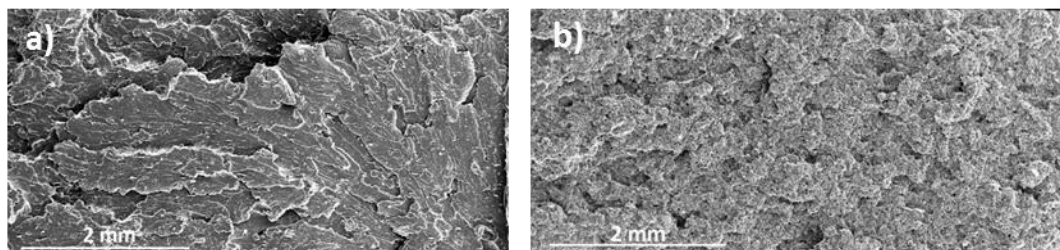


Figure 2. SEM images of: a) unmodified and b) 15 wt% TEPs-modified adhesive specimens.

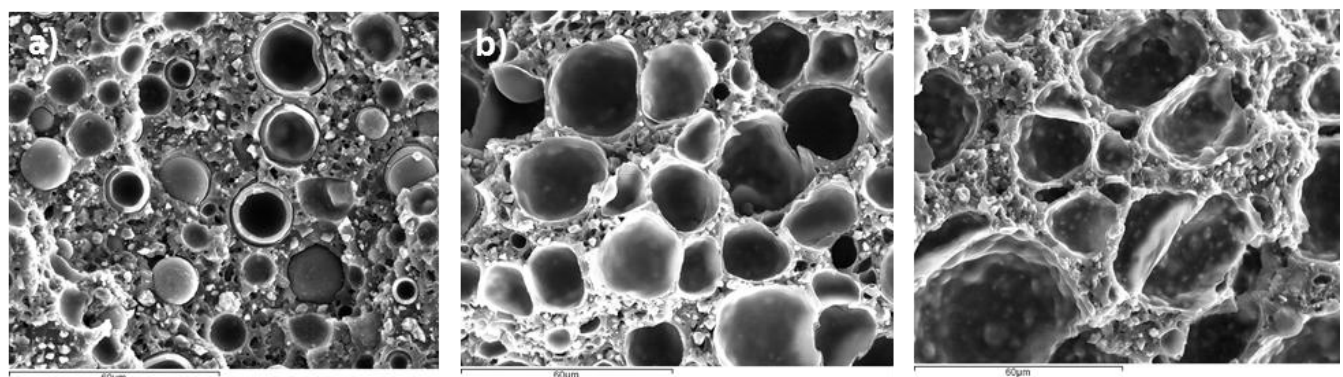


Figure 3. SEM images of thermal samples at different temperatures: a) RT; b) T_{\max} ; c) $150\text{ }^{\circ}\text{C}$.