

Effect of curing on the broadband dielectric spectroscopy of powder coating

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Abstract

The effect of curing process of thermosetting powder coating consists of carboxylated polyester resin cured with triglycidyl isocyanurate has been investigated using broadband dielectric relaxation spectroscopy over a wide range of frequency (10^{-1} – 10^6 Hz) and temperature (70–105 °C) for different constant curing times. The molecular dynamics of the glass relaxation process (α -process) was investigated as a function of curing time, frequency, and temperature. It has been found that, only one common α -relaxation process has been observed for all measured samples of different degree of curing stages, its dynamics and broadness were found to be curing time dependent. In addition, the curing time dependence of the dielectric relaxation strength, $\Delta\epsilon$, has also been examined for the α and β -relaxation processes. The $\Delta\epsilon$ for the two relaxation processes decreased strongly at the beginning of curing process and then became almost constant at longer curing times. This finding implied that the numbers of reoriented dipoles decrease with curing time as a result of the formation of three-dimensional polymer network. Furthermore, the dislocation energy, ϵ_s , calculated from the Meander model was found to be increased with increasing the curing time, i.e. the formation of a three-dimensional polymer network produces many structural defects or dislocation points. In addition, the activation energy of the curing process was calculated from the analysis of the calorimetric exothermic peaks of the curing process at different heating rates.

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1. Introduction

Powder coatings are industrial important materials used in great variety of applications, such as, manufacturing automobiles, garage doors, window profiles, office furniture, storage shelving and pipes [1,2]. The curing process of thermosetting powder coating involves two stages, the first is the formation

of an elastic gel or an infinitely crosslinked molecular network which contains in its bulk free molecules of the same type as the networks. The second is the conversion of the elastic gel to a rigid glass after almost all free molecules have combined to form a dense network with greatly reduced crosslinking distances. Normally, the curing process of thermoset materials accompanies by an elevation in the glass transition temperature which leads in some cases to a vitrification. This process (vitrification) occurs when the T_g of the material becomes equal to the curing temperature and involves cessation of the reactive process.

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Melting, flow, gel point and curing completion are the principal stages in the film formation of powder coatings that in turn play a dominant role for controlling the coating properties such as adhesion, gloss, chemical resistance and exterior durability [3–6].

The curing kinetics of thermosetting polymers has been extensively investigated by different techniques such as differential scanning calorimetry (DSC) [7–10], dynamic rheology [11–15], dielectric spectroscopy [16–28] and mechanical measurements [29–31]. The dielectric relaxation spectroscopy is a powerful tool capable of providing information about the molecular dynamics and the nature of the interactions in polymers by monitoring the motion of dipolar groups attached to molecular chains [32,33]. The dielectric spectroscopy has been widely used in polymer relaxation analysis and has the advantage over the dynamic mechanical methods in that it covers much wider frequency ranges. The dielectric properties during the curing of thermosetting powder coatings are obviously determined by changing the chemical composition as chemical reactions occur, which change the concentration of various dipolar molecular segments as well as their rotational degree of freedom [34,35].

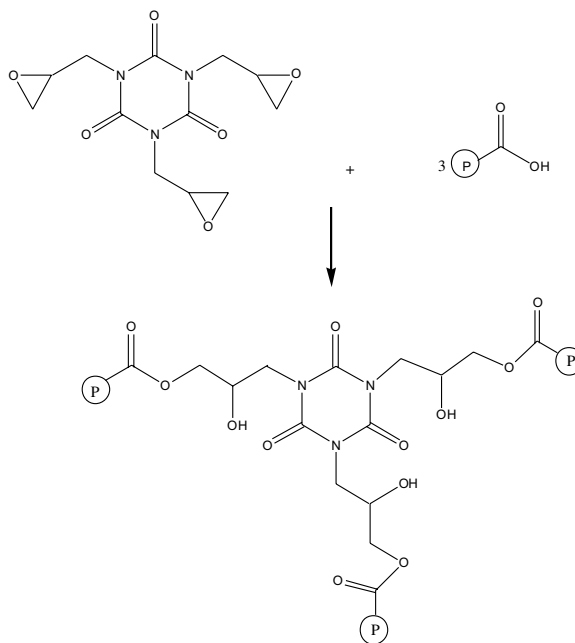
In this paper, the broadband dielectric spectroscopy for thermosetting powder coating consists of carboxylated polyester resin cured with triglycidyl isocyanurate will be investigated over a wide range of frequency (10^{-1} – 10^6 Hz) and temperature (70–105 °C). A different degree of crosslinking powder coating will be obtained by curing the sample at 200 °C for different time intervals. The influence of curing will be examined dielectrically by following the change in the molecular dynamics of the α -relaxation process as a function of curing time, frequency and temperature. In addition, the relaxation strengths ($\Delta\epsilon$) of the α and β -relaxation processes will be calculated as a function of curing time from the analysis of dielectric relaxation spectra. Furthermore, the activation energy of the curing process will be calculated from the analysis of the calorimetric exothermic peaks of the curing process at different heating rates.

2. Experimental

2.1. Materials and samples preparation

The carboxylated polyester resin, Crylcoat 441, was obtained from UCB Chemicals with an acid

value = 33 ± 3 and viscosity = 5000 ± 1000 mPa s at 200 °C. The molar mass of the Crylcoat 441 is 1700 g/mol. The crosslinking agent (hardener) is triglycidyl isocyanurate (TGIC), Araldite 710 (CIBA Resins), with an epoxy index = 8.8–9.8 equiv./kg. The sample was prepared in the ratio of polyester/hardener = 93/7 wt% using APV extruder at a temperature = 90 °C and 300 round per minute rotation speed. The applied pressure was the same in the preparing all samples. No any detectable increase in the T_g of the pure polyester resin and its mixture with the hardener after extrusion indicates that at 90 °C no any reaction takes place during the mixing process. About 0.1 g of the extruded powder coating was taken and pressed between two copper electrodes at $T = 90$ °C. The sample will be cured at a typical industrial curing temperature (200 °C) for different time intervals after that the sample will be quickly quenched to room temperature in order to obtain samples of different degrees of crosslinked polymers. The chemical reaction between the polyester resin and its hardener during the curing process is represented as follows:



2.2. Measurements

The dielectric set-up contains an impedance/gain analyzer SI1260 (schlumberger), an electrometer amplifier and a measuring cell, which was very similar to that used before [36]. The effective diameter

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