EXPERIMENTAL EVIDENCE OF THE INTERFACE/INTERPHASE FORMATION BETWEEN POWDER COATING AND COMPOSITE MATERIAL

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Keywords: Powder coating, Epoxy resin, Interface, Interphase, Composite material.

1 Scope and objective of the study
In recent year, the use of powder coatings has been increased due to its advantages over the conventional solvent based-paints. The ability to apply this modern environmentally coatings on metal and heat resistance composite was a real benefit for aeronautic and automobile industries. Based on the « Montreal protocol, September 1987 » which insisted to protect the ozone layer from the substances responsible for its depletion, powder coatings were considered as a good solution for reducing the emission of Volatile Organic Compounds (COVs). [1]
Practically, deposition of powder coatings is realized by flowing it to the substrate through a gun, which applies an electrostatic charge for the individual polymer particles. The coated substrate is then heated in a curing oven in order to coalesce the polymer particles and form a continuous film on surface. Here, the nature of the substrate plays a major role in adhesion process of powder coatings. When using a metal substrate, this procedure shows effective results due to the conductive properties of the metallic substrate [2,3]. However, when the substrate is a polymer based composite material, some problems related to the adhesion strength can limit the application of powder coatings.
Our objective is therefore to develop an alternative method for powder coating deposition on composite material, and obviously to characterize the organic/organic interface formed between the powder coating and the surface.

2 Experimental section
2.1 Materials
The powder coating is essentially constituted by a mixture of diglycidylether of bisphenol A (DGEBA), carboxyl polyester resins, mineral fillers and other additives. The composite material is composed by epoxy resins containing polyethersulfone with glass and carbon fibers.

2.2 Sample and cross section preparation
In order to prepare the coated-composite plate, an in-mold powder coating process has been performed. The powder coating was firstly sprayed onto a mold cavity and partially cross-linked by heating treatment. The composite plate was then molded on pre-heated powder layer, and the in-mold coating composite sample was fully cured and chemically bonded with a curing cycle (2h at 120°C and 2h at 180°C). In order to study the effect of conversion degree of powder coating on the interface properties, different samples have been prepared by varying, in the first stage, (i) the pre-heating times of the powder coating in the mold (15 min, 20 min, 40 min, 60 min) and/or (ii) the curing temperature (120°C and 160°C). Cross sections were prepared by cutting the coated composite plate, then wrapped by acrylic resin and polished as smooth as possible.

2.3 Characterization methods
Physical and chemical analyses allowed us to highlight in depth the properties of the adhesive interface. Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM) have been used to study the structural properties. FTIR and Raman Spectroscopies have been applied to chemically identify the different components of the interface. Other more conventional techniques (DSC, DMA …) have been used to study the thermo-mechanical properties and understand the adhesion mechanisms between composite and powder coatings.
3 Results overview and discussion

Figure 1 shows SEM cross section micrographs taken for two coated composite plates. Before application of the composite in the mold, the degrees of conversion of the powder coating were 92% in the first sample (Figure 1.a) and 48% in the second sample (Figure 1.b). These degrees were calculated from DSC results after heating the powder coatings at 120°C for 60 min and 20 min respectively. Paint film thicknesses were measured at many random positions around the cross sections. The thickness of final coatings has about 100 ± 20 µm.

As shown in micrograph (a), the coating is homogeneous and the interface between the coating and the substrate is clear. On the other hand, the micrograph (b) shows micrometric nodules (droplets) within the coating. The size of these nodules is widely variable and ranged from few micrometers to about 50 µm. The region, which includes paint matrix and nodules is called «Interphase». We suggest here a diffusion process from the composite material to the powder coating during the curing program. Because the powder coating was not completely cross-linked, the migration across the interface can be occurred. In consequence, the interface between the composite and the coating has been disappeared, and an organic/organic heterogeneous interphase has been created.

To confirm the observed diffusion process and characterize the formed interphase, FTIR and Raman spectroscopies were carried out by collecting spectra at each component of the interphase. Our results showed that the formation of interphase depends on the degree of conversion of the powder coating during the first stage of sample preparation. Diffusion process can occur when the degree of conversion is less than 69%. In addition, preliminary results on adhesion strength show the efficiency of in-mold powder coating method for epoxy based-composites.

This study demonstrates that the used microscopic and spectroscopic techniques are able to reveal interesting physicochemical characteristics of coated-composite material. This can be potentially relevant for industrial applications.

4 References


5 Acknowledgments

The authors gratefully acknowledge the partners and the different financial supports of POPART project (FUI 8).