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Development of matte finishes in electrostatic (EFB) and conventional hot dipping (CHDFB) fluidized bed coating process

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Abstract

This study focuses on the correlation between the thermo-rheological properties of a thermosetting powder coating system with its surface structure build-up. Epoxy powder coating systems, which displayed surface matting and surface wrinkling, were examined. Firstly, the evolution of the complex viscosity was correlated with the cure kinetic. Secondly, the structure build-up on the surface of the coatings was investigated with a combined SEM-CLA profilometry analysis at different stages of curing process for both EFB and CHDFB coating processes. Different finishes were found to characterize the films applied by using EFB and CHDFB coating processes as a result of the different way the film is heated by. Finally, a strict relationship of film morphology to the degree of conversion and to the evolution of the complex viscosity was found out for both EFB and CHDFB coating processes. The surface structure is built up after gelation point and continues to evolve after gelation with a full development of the film fine structure. Differences were observed in the surface structure build-up when different curing temperature was used, thereby indicating an influence of minimum viscosity on achievable finishing.

These experimental results lead to further advances in a better understanding of the formation of surface topography and morphology of polymeric films. They also provide important indications for the settings of curing parameters in both EFB and CHDFB coating processes as well as for the development of new powder coating formulations.

Keywords: Powder coatings; Fluidized bed; Film morphology

1. Introduction

1.1. Premise and historical background

In electrostatic (EFB) and conventional hot dipping (CHDFB) fluidized bed coating process, an understanding of the stages by which a deposited powder is converted to a fully consolidated film requires the investigation of several properties such as substrate roughness, surface tension, thermo-rheological behaviour, curing kinetic of the resin and the operational parameters during coating process. Film properties like modulus, adhesion, impact, scratch and wear endurance, residual stresses distribution, chemical resistance to aggressive agents as salt fogs, acid or alkaline environment, physical characteristics and, above all, appearance are strictly dependent upon operational choices in deposition and curing phases, in preparation of substrates surface and, mostly, upon chemical composition of the resin, curing agents and additives.

Several studies have attempted to correlate the film appearance to the operational settings of fluidized bed coating processes. Since 1962, Richart and Pettigrew tried to find a correlation between surface appearance and part location and vibration, preheating temperature and coating time in a CHDFB coating process [1–4]. In 1977, Strucaly started studying correlation between electrostatic parameters (voltage and current intensity) and surface appearance in EFB coating process of armatures for electric motors [5]. In nineties, Leong et al. developed experimental and numerical studies about correlation among film properties and substrate materials and geometries in CHDFB coating process [6]. Inculet and co-workers modelled the relationship between part shape and film uniformity in both electrostatic and tribo-charged fluidized bed [7,8]. More recently, Barletta et al. built first approximation models describing the trend of average roughness of polymeric film deposited

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by EFB [9,10] and CHDFB [11,12] by varying the deposition conditions. First time, they also attempted to model the evolution from the status of loose powders to the status of consolidated film, which a thermoplastic polymeric film run through [12], hence individuating three different phases: powder melting, levelling and formation of a continuous smooth film.

On the other hand, since 1976, Hannon et al. [13] understood the correlation between surface appearance and the rheological characteristics of the coatings, suggesting that the complex viscosity, η^*, might be a useful measure of the progress of two important aspects of the coating process. They found out that η^*, at the early stages of cure, measured the viscosity which, together with the surface tension, determined the surface appearance and, in the later stages, η^* was a measure of the modulus, which in turn measured the degree of cross-linking of the resin and its mechanical properties. During the following years, scientific world attention was moved on definition of new powder paint formulations based upon polymeric blends, which were expected to provide diverse coating texture such as low gloss (i.e. matte finishes with gloss <5%), high clarity for automotive topcoats, high abrasion resistance and polychromatic or gonioapparent effects [14,15]. Accordingly, the influence of new resin additives like curing agents, flow promoters and extenders onto final film morphology was also widely discussed [16–18]. However, most publications have been mainly concerned only with the levelling of powder coatings and not with the development of matte finishes [19–21]. In fact, few attempts have so far been made to correlate the thermal and rheological behaviour of powder coatings with their surface structure build-up during curing. In general, surface appearance has always been described only by gloss and colour of the surface as well as by surface roughness trends according to settings of coating process [22] and no correlations between film morphology and thermo-rheological properties of the resins were sought.

In this context, Lee et al. were the first to study, at relatively high resolution, the surface structure build-up during curing and to establish a correlation between the microscopic surface structure and the thermal and rheological properties of the thermosetting powder coatings during curing applied by electrostatic spray deposition [23]. However, the factors generating the surface structure, especially matte and wrinkled surfaces, have not yet been investigated in detail. Furthermore, the correlation between gelation point and coarse and fine structure formation, the influence of minimum viscosity on final film morphology, the relationship between the evolution of surface morphology and the evolution of cure kinetic and viscosity are important issues, which still remain mostly unresolved.

1.2. The aim of the work

This study deals with the analysis of relationships between the thermal and rheological behaviour of thermosetting powder coatings and the surface structure build-up during curing in EFB and CHDFB coating process. In particular, the evolution of film morphology versus curing time and temperature in EFB and versus preheating temperature in CHDFB has been the main issue of the led experimental investigation. Deposition conditions of powder coatings by EFB and CHDFB process have followed the indications provided by several past studies which one of the authors developed [9–12]. This way, the influence of coating process parameters on starting characteristics of the deposited films has been minimized.

This is the context in which the present study moves to build the groundwork for a better understanding of the evolution of polymeric film surface appearance and its relationship with thermosetting powder coatings cure kinetics and complex viscosity. For this purpose, the thermo-rheological behaviour of the resin has been studied by a combined analysis between differential scanning calorimetry (DSC) and a torsional parallel-plate rheometer. The surface roughness and structural pattern of film morphology have been investigated, at different stage of the coating process, using a combined analysis of a field emission gun scanning electron microscope and a non-contact profilometry based upon a chromatic aberration lens.

The aim of this study is thus, manifold: (i) to follow at high resolution the evolution of film surface structure during curing at different stages for both EFB and CHDFB coating process, (ii) to document the necking phenomena between powder grains which leads the polymeric coating from a status of loose powder to a status of a full consolidated film, (iii) to interpret the difference in mechanisms which leads to different finishes for EFB- and CHDFB-coated substrates, (iv) to establish a correlation between microscopic surface structure and the thermal and rheological properties of the thermosetting powder coatings during curing for EFB and CHDFB coating processes, and, finally, (v) to single out the influence on final film appearance (development of coarse and fine structure) of degree of conversion, minimum viscosity and gelation point of the resin.

2. Experimental

2.1. Materials

A commercial epoxy-based thermosetting powder (20 μm as mean diameter of the granulometric distribution, 0.80 as factor shape) supplied by Becker Powder Coatings was employed during experimental tests. The formulation was based upon bisphenol-A type epoxy. Cross-linkers and other additives such as a flowing agent (silica and aluminium oxides), flowing promoter after melting and UV stabilizer (titanium oxides) completed the formulation. Such powder was specifically developed for fluidized bed coating process. It was a low-gloss powder coating, representative of matte powder coating systems used in the industrial, decorative and maintenance industries.

2.2. Cure kinetics

Cure kinetic studies were performed using a Differential Scanning Calorimeter (DSC Netzsch model DSC200PC). First,