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Epoxy-based thermosetting powder coatings: Surface appearance, scratch adhesion and wear resistance

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Abstract

The deposition of protective coatings from thermosetting polymer powders is undergoing rapid growth as an ecological, economic and energy-efficient technology. Nevertheless, a combination of conflicting needs is characterizing new formulations: (i) minimization of premature cross-linking during material production and storage; (ii) film levelling and cross-linking at the lowest possible temperature in the least possible time. We address the problem by developing an experimental study in which the evolution of film morphology and its thermal, rheological, mechanical and tribological properties are analyzed in the light of film baking time and temperature.

Experimental results show that smooth films can be achieved at relatively low baking time and temperature, while film mechanical performances are not yet fully developed. In contrast, good film strength, adhesion and wear endurance can be attained, only if longer baking time or higher temperature is set. However, a generalized scratch map, which correlates the adhesion strength of the polymeric films with the thermo-rheological behaviour of the base material, has been also built.

Our results open up the possibility of predicting best baking programs and how full film performances are progressively established with time and temperature by better understanding of the mechanisms involved in polymeric films formation from loose powders.

Keywords: Powder coatings; Film morphology; Scratch adhesion; Wear endurance

1. Introduction

Powder coating market has recently been undergoing rapid growth. Powder coatings possess advantages over conventional coatings in that no polluting solvent loss occurs on application, flammability and toxicity are substantially reduced, thick films of 250 μm and over can be applied in a single operation, owing to the use of electrostatic spray, little material is lost and, what is more, during baking, energy consumption can be reduced compared to solvent-borne or water-borne coatings [1].

The low energy requirements of powder coatings make them extremely attractive to the industrial world. Powder coatings possess higher baking temperatures but, with little or no volatiles being emitted into the oven during baking, the air in the oven can be recirculated with almost no make-up air, thus permitting significant savings. On the contrary, with solvent-borne coatings, the solvent concentration in the oven atmosphere must always be kept well below the lower explosive limit, resulting in the need to heat considerable volume of make-up air, thus increasing energy consumptions [2].

The growing interest in powder coatings has opened new challenges towards the definition of new material formulations, which were able to concurrently improve film properties, reduce environmental impact, and, above all, allow energy savings. Thus, the challenge facing formulators is satisfying a combination of conflicting needs: (i) minimization of premature cross-linking during production; (ii) stability against sintering during storage; (iii) coalescence, degassing and levelling at the lowest possible baking temperature; and (iv) cross-linking at the lowest possible temperature in the least possible time.

Appearance qualities of paints and coatings, their mechanical properties, durability, adhesion and wear endurance do strictly depend on baking time and temperature as well as on material

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formulations [1,2]. Furthermore, the establishment of each individual film property requires specific baking procedure in order to fully express the performances, which the films are designed for [3]. Finally, most of powder coatings application costs belong to baking procedure. For such reason, in the last years, a large number of new compliant powder coating formulations have been put on the market, hence pushing many scientists to examine the evolution of resulting film properties according to baking procedure.

In this sense, the main aim of the studies reported in the literature has always been to optimise the choices of baking temperature and time according to the end use of the films and their formulations, thus minimizing energetic consumption and allowing the establishment of full film properties [4–14]. In this field, Lee et al. were the first which tried to establish a correlation among thermo-rheological properties of an epoxy-based coating and baking time and temperature. In that work, they were able to interpret the surface structure build-up of the film during curing in the light of the evolution of the viscosity and conversion of the epoxy-based system according to time and temperature [4]. Next, Andrei et al. tried to relate the surface levelling of an acrylic powder coating system to the evolution of its viscosity with time and temperature, hence opening up the possibility of predicting the final surface topography given the rheological properties of a polymer [5]. More recently, Barletta et al. have reported some results concerning the development of smooth finishes in electrostatic fluidized bed coating process of high-performance thermoplastic powders (PPA 571 H) [6] and of matte finishes in electrostatic and conventional hot dipping fluidized bed coating process of an epoxy-based system [7]. In those works, the authors looked into the evolution of powder coating films at different stages of curing process using the in-lens detector of a field emission gun scanning electron microscope to catch high resolution images of the film topography and a non-contact 300 μm chromatic aberration length gauge to catch the 3D morphology of the coatings [6,7].

The correlation among film mechanical properties, coating materials and baking procedures is the other approach widely reported in the pertinent literature [8–14]. Since early nineties, dynamic mechanical analysis was used for characterizing ‘free standing’ cured films, thus deducing information about films glass transition temperature T_g, their storage and loss modulus at different stages of curing process [8,9]. Later, further scientific works have reported on internal stresses, Young’s modulus, film wear endurance, impact resistance and adhesion strength for different coating materials [10–14]. In 1999, Bouchet et al. paid a great deal of attention to the relationship among mechanical properties, residual stress intensities and film typologies of different epoxy based systems [10]. Later, Francis et al. detailed the relationship between the development of polymeric film stresses during curing and the establishment of full film mechanical properties [11]. In 2001, Trezona and Hutchings reported on specific erosion resistance of acrylic coatings by submitting them to multiple solid particle impact tests [12]. Next, Ramsteiner et al. tried to correlate scratch adhesion and coating toughness for different polymeric blends [13]. Finally, Krupicka et al. reported on a more extensive use of the scratch test to check the overall response of different coating materials to real contact conditions, hence providing extensive information on the physical state, the yield and break behaviour of the polymer coatings [14].

However, systematic approaches in which the evolution of main film properties (visual appearance, mechanical properties, durability, adhesion and wear resistance...) is studied at the different stages of curing process do still miss. Such approach would be of extreme importance as it would allow not only the detection of the final film properties, but even their establishment from the status of uncured film to the status of fully cured one. This way, the mechanisms involved in polymeric films formation from loose powders would be better understood and the optimisation of baking procedure could be definitely pursued.

This is, therefore, the context in which the present study moves to analyze the evolution of morphology, scratch adhesion and wear endurance of an epoxy-based powder coating system and their relationship with the polymer thermo-rheological properties at different stages of film curing process.

First, a thermo-rheological characterization of the epoxy-based powders was performed. This allowed tracking the trend of cure conversion and viscosity of the powders according to desired time–temperature programs. In particular, cure kinetic and complex viscosity were modelled by using experimental data and first approximation analytical models. Levelling and flowing capabilities of the polymeric powders were also analyzed by using an Environmental Scanning Electron Microscope (ESEM). This technique allowed the on-line tracking, at high resolution, of the surface flattening of the epoxy-based powders under desired time–temperature programs.

Next, Electrostatic Spray Deposition (ESD) was used to apply the epoxy-based powder coatings onto standard metal substrates. Baking time and temperature were varied to cover a wide field of possible curing conditions. This way, polymeric films with different characteristics were achieved. Produced films were characterized at all stages of the curing process. First stages of films curing process were examined in order to track the fast morphological evolution of the polymeric films from the status of loose powders to the status of full consolidated films. Both high resolution images and 3D morphology of the coatings were caught to support the led analysis. Intermediate and last phases of film curing process were used to monitor the establishment of smooth finishes and to evaluate the film strength, scratch adhesion and wear endurance, thus trying to correlate the evolution of film properties with baking time and temperature.

Experimental results showed a significant delay in the establishment of full film properties and the approach of the film to a smooth finish. In fact, good film visual appearance is already established in the early stages of curing process, while scratch adhesion and wear resistance are still very poor. In contrast, only fully cured film can express their full potential in terms of scratch and wear resistance. However, consistent trends of surface roughness, film strength as well as frictional coefficient, penetration depth and wear track produced by scratch and tribological tests according to baking time and