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Effects of IR pre-curing conditions on wear resistance of metal flake powder coatings

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ABSTRACT

Hybrid IR/convective oven baking of high quality industrial powder coatings is one of the most attractive method to achieve significant economic and process time savings. This method is based on two curing steps: at first, a pigmented decorative basecoat is electrostatically sprayed and, then, pre-cured with IR-radiation, secondly, a transparent protective topcoat is sprayed on the basecoat and the resulting bilayer coating is oven baked. The optimization of the IR pre-curing conditions and the correlation between the effect of polymerization degree of the basecoat and the wear resistance of the whole coating system are investigated. In particular, an experimental study in which the degree of chemical conversion of the pigmented basecoat, the overall coating morphology and its thermal, mechanical and tribological properties are analyzed in the light of IR-radiation time and power, has been carried out. Experimental results show that the intermediate range of curing time and IR power investigated leads to properly cured basecoats and subsequently to better morphological, mechanical and tribological behavior of the whole coating system. These results were also validated by comparison with the coatings cross-linked by the traditional two-step oven baking process.

1. Introduction

Energy savings is also of outmost importance in almost all industrial applications regarding powder coatings [1–5]. Numerous were the attempts to manufacture low curing temperature or radiation curable powder coatings to lower the overall energetic demand of the painting processes. In the past decades, the research efforts were paid on the reduction of the cure temperature of polyester–TGIC systems, whilst preserving acceptable aesthetic and functional properties of the resulting powder-based formulations [6]. More recently, Gedan-Smolka investigated low temperature curing uretdiones powder coatings trying to develop new solutions to accelerate the deblocking process and reduce its stoving temperature at 150 °C [5]. In 2003, Subramanian described the potentiality of new formulations to lower the cure temperature of polyurethane powder coatings, with particular attention focused on various trimethyloyl propane and aliphatic isocyanate trimers based on isophorone diisocyanate [7]. In 2004, Facke et al. introduced the usage of diethylmalonate blocked isocyanate as crosslinkers to reduce the curing temperature of hybrid and TGIC cured powder coatings [8]. Although many research efforts, most of commercially available low curable powder coating formulations should be baked at temperature of 135 °C or more and for long time (>20 min). Indeed, they can be extremely expensive and sometimes lead to film with limited mechanical properties and poor visual appearance [9]. Powder coatings involving radiative curing systems are potentially attractive alternatives to formulations involving thermally activated reactions. For example, they allow coating of heat sensitive substrates as plastics or MDF boards [10]. UV-powder coatings belong to the class of radiation curable powder paints and they have always been widely reported in the literature [11–14]. UV-curable powder paints allow no significant emission of volatile organic compounds and their usage entails fast curing speeds, minimal health risk and independency of the flow system from the cure system [15,16]. More recently, N'Negue Mintsa et al. proposed a new UV-curable formulation based on α,ω-unsaturated copolyamide 6/11/12, which was found to reduce the activation temperature during radiative curing at 80 °C with potential application in highly thermal sensitive plastic substrate as Acrylonitrile Butadiene Styrene (ABS) [17]. Although thoroughly studied, radiation curable powder coatings play a minor role in the industrial coatings as they account for a very limited market share (well below 1%). Beyond the high cost of radiation curable powder coatings, the lost of a part of functional and aesthetic properties as well as the low stability during the storage remain all major drawbacks which limit their commercial diffusion [18].

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Potential interest for hybrid curing technologies which involve both radiative and convective baking steps is recently arousing [19]. Such class of curing methodology is of outmost importance when multi-layer powder coatings are dealt in. For example, the deposition of a bilayer metal flake powder coating requires two deposition steps: the application and baking of a metal flakes decorative basecoat followed by the application and re-baking of a transparent protective topcoat, with the topmost layer being of crucial importance as it ensure the corrosion protection as well as the scratch and wear resistance of the whole coating system [20–24]. Such twofold baking process is, of course, energy and time-demanding as it requires two application steps followed by two separated baking steps at high temperature (170 °C) and which can last long (i.e., about 20 min each). Further, a cooling phase is required after the first baking process and before the second deposition process to allow the bottom layer to stabilize before being coated with the protective topcoat.

Significant savings could be achieved by substituting the first baking process with a fast radiative pre-curing focused only to freeze the structure of the decorative layer. In particular, the radiative pre-curing should avoid any pigment migration after the deposition of the transparent topcoat and convective baking at high temperature of the overall coating system, thus avoiding any potential effect detrimental to the attractiveness and functionality of the coating itself [19]. Such a curing strategy of a bilayer coating would require only one convective baking step at high temperature for long time and no need for any intermediate cooling between the first and second deposition process with significant savings in terms of processing time and economic. In this respect, Bellisario et al. showed how opportunely modulating the usage of a IR source during the baking of a metallic decorative basecoat, that is, after the first step of the deposition process, the visual appearance and scratch resistance of the incoming bilayer could be comparable to those achieved by two subsequent oven-baking steps [19]. Yet, in that pioneeristic work, only a limited set of IR operating conditions was investigated. Moreover, no precise indications on how the curing methodology could influence both the visual appearance and tribological behavior of the bilayer coating were detailed. Finally, the interrelationship between the degree of conversion of the bilayer coatings at the different curing stages and the resulting aesthetic and functional properties was not faced yet. This is therefore the context in which the present study moves to further analyze the recently proposed IR pre-curing methodology. In particular, the IR pre-baking step is here employed to pre-cure the underlying decorative basecoat of metallic powder coatings prior to the following deposition with transparent topcoats and baking in convective oven (at high temperature and for long time) of the resulting bilayer coatings. The final aim of this study is to establish how the residual degree of conversion of the resins after the different curing stages, their visual appearance (color, gloss and morphology) and wear resistance (pin-on-disk and linear reciprocating) are interrelated and how to get the best profit in terms of overall coatings performance from the proper setting of the curing operating parameters. Lastly, a first analytical model which correlates the degree of conversion of the polymeric material and the aesthetic, mechanical and tribological behavior of the coatings has been also the matter of the present investigation.

2. Experimental

2.1. Materials and specimen preparation

Detailed description of the thermoset powders chosen, their properties as well as of the deposition process and substrate pretreatments, is elsewhere reported [19]. Here, it is worth to remark that a metal flake powder (epoxy-polyester, 20 μm mean diameter, 0.80 factor shape, PPG-Bellaria) was electrostatically sprayed on galvanized sheets to act as decorative basecoat. Upon curing, a transparent protective topcoat (polyurethane powders, 20 μm diameter, 0.80 factor shape, PPG-Bellaria) was deposited on it by electrostatic spraying and the resulting bilayer was cured again. The deposition parameters of the electrostatic spraying are summarized in Table 1. The decorative basecoats were 'flash' pre-cured by infrared radiations setting the power of the IR lamps in the range 1.0–2.0 kW and exposure time from 30 to 120 s. The IR oven was equipped with a set of linear emitting halogen (94% of radiation in the IR range and the remaining 6% in the visible) lamps with low mass and short focus tungsten filaments (focus distance 50 mm and working temperature 2227 °C) and quartz envelopes. The heat flux delivered to the coating surface during IR pre-curing and the absorption coefficient are reported in Table 2 [19]. Upon pre-curing, the samples were re-coated (without allowing them to cool) using the transparent polyurethane powder and the whole coating systems (i.e., transparent protective topcoat onto the pigmented decorative basecoat) were then re-baked at 170 °C for 20 min in convection oven. For comparative purpose, further samples were manufactured by baking both the decorative basecoat and the protective topcoat in a convection oven at 170 °C for 20 min after the electrostatic spraying each layer. The coatings thickness of each layer was measured by a standard thickness gauge (Mega-Check 5FN-ST) following the regulations ISO 2178 and ISO 2370. The coating thickness of the decorative basecoats was checked to be ~80 μm. The coating thickness of the protective topcoats was checked to be ~50 μm. The overall thickness of the coating was therefore checked to be in the range of 110–130 μm. All the coatings failing to agree with this specification more than ±5% were discarded.

Table 1
Parameters of the electrostatic spray deposition.

Electrostatic spray deposition parameters	
Voltage	90 kW
Feeding pressure	1.5 bar
Auxiliary pressure	1.0 bar
Exposure time	6 s
Substrate material	AISI 304 stainless steel
Adhesion interlayer	Inorganic layer

2.2. Surface characterization

The 3D topography of the bilayer coatings was obtained by intermediate range (511 μm) contact gauge Surface Topography System (Taylor Hobson TalySurf CLI 2000). The substrates, after the deposition of the basecoat and topcoat, were located under the gauge and view optically with an onboard camera to identify the same measurement area. A number of patterns (1000), each 4 mm long and with a lateral resolution of 2 μm was recorded to cover a wider area of 16 mm^2, which could be representative of the entire surface structure. The surface morphology was then analyzed by TalyMap software Release 3.1. As a characteristic of the coatings morphology, standard amplitude, spacing and hybrid parameters for both waviness and roughness profiles were calculated (0.25, Gaussian filter).

Polymeric film gloss was measured by a standard glossmeter (Erichsen picogloss 560MC (0.2 GU from 0 to 150 GU)) with a single measurement geometry of 60°. The equipment is conform to the regulations EN ISO 2813, DIN 67 530, ISO 7668 and ASTM D 528.

2.3. DSC measurements

Thermal analysis was carried out on a differential scanning calorimeter (Perkin Elmer DSC6, Monza, Italy). Tests were mostly