



Time and cost effective methods for testing chemical resistance of aluminium metallic pigmented powder coatings

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ARTICLE INFO

Article history:

Received 3 January 2008

Accepted 17 April 2008

Keywords:

Powder coatings
Chemical stability
Metallic pigments
Pigment coating

ABSTRACT

Polyester based powder coatings containing different types of aluminium metallic flake pigments have been investigated with respect to their chemical stability in acid environments. The metallic flakes are made chemically stable by covering them in silica. The degree of silica coverage and the silica morphology are far more important for the chemical stability of the pigments than the silica thickness. The acid resistance of the final powder coating is found to depend on the pigment embedment depth, on the chemical composition and morphology of the powder coating, and on the silica coating of the pigment. The latter being the single most important factor in our study.

New chemical resistance tests have been developed. One for the testing of the metallic pigments alone before embedding the pigment in a coating, and another for the cured coating containing these pigments. Additionally TEM cross-sections have been used for investigating the quality of the silica coating on pigments, and optical absorption for comparison of pigment embedment depth. This approach to powder coating development emphasizing the measurement of the quality of each component separately has proven to be robust, reliable, and time and cost effective.

By testing of separate components the need for time-consuming quality cheques of cured coatings is reduced while maintaining the ability to predict final paint properties. Further, the uncertainty related to testing many process parameters simultaneously is considerably reduced.

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1. Introduction

Powder coating has been established as one of the most efficient coating techniques available. It offers advantages as no or very little volatile organic compounds (VOC), easy application procedures and very high efficiency due to recycling of excess powder. Powder coating has a low impact on health and environment. The level of toxicity has been reduced, and the problems associated with explosive hazard of powder in air have been solved [1–3]. These facts combined with very good economics have made powder coating an attractive coating technology.

It has been claimed that the development in the powder coating industry mainly has focused on cost and health, environment and

safety issues rather than new product development [2]. The development of new products and new manufacturing techniques has been slower than predicted some years ago. One important reason for this is that the test procedures needed for development work are slow (time-consuming exposures, such as the Florida test), expensive and not sufficiently robust. The comprehensive test programs as specified by GSB [4], Qualicoat [5] and others are designed for the purpose of quality assurance of coating products entering the market, but are far too time and cost ineffective to be suitable for R&D work. These tests are (mainly) on finished coatings, and do not address optimization of the quality of single components or constituents in the coating. The tests are not suitable to monitor the effect of changing one single parameter because there are too many uncertain factors. This can to some extent be encountered for by statistics, but this will further increase development cost and time consumption. There is, therefore, a general demand for robust, reliable, quick and cheap experiments isolating single coating components.

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Table 1
Overview of the different commercially available aluminium flake pigments. The grain size “Fine” refers to D_{50} values $<20\ \mu\text{m}$ and “Coarse” refers to $D_{50} >30\ \mu\text{m}$

Pigment no.	Metal	Type	Shape	Surface coating	Size	Predicted chemical durability
I	Aluminium	Non-leafing	Flake	Silica	Fine	Standard
II	Aluminium	Non-leafing	Flake	Silica	Fine	Standard
III	Aluminium	Non-leafing	Flake	Silica	Fine	Improved
IV	Aluminium	Non-leafing	Flake	Silica	Coarse	Improved
V	Aluminium	Non-leafing	Flake	Silica	Fine	Extra reinforced
VI	Aluminium	Non-leafing	Flake	Silica	Fine	Extra reinforced

The “predicted chemical durability” is based on information from the suppliers.

Metallic effect coating is an established technology [6] and aluminium pigments for powder coatings applications have been focused by the pigment suppliers lately. The leafing pigments, having a tendency to float to the surface of the coating and to orient parallel with the coating surface, give a bright silver-like finish. The disadvantage with leafing aluminium pigments is the poor rubbing, weathering and chemical resistance unless a clear topcoat is applied. Semi-leafing aluminium flake pigments have a potential of giving powder coatings with good *in-service* properties without having to apply a clear topcoat. This put some requirements on the pigment properties and has been the reason why pigment suppliers have worked extensively on pigment coating optimization. Many suppliers have focused on rubbing, weathering and chemical stability together with the powder application properties. New variants of organic and inorganic pigment protective layers have been developed recently, including sol–gel encapsulations techniques [7–9]. Some of these have also been introduced to the market.

In the present work we report on a powder coating development work where we have used non-leafing aluminium flake pigments protected by an inorganic silica coating, and where the requirements for rubbing, weathering and chemical stability has been normative. The chemical stability of the coating has been important to monitor. The Acidified Salt Spray (ASS) test, originally designed for corrosion measurements, has been used for this in combination with colour measurements. This test is, however, very time and cost consuming.

In the present work we have (i) developed a new procedure for testing the chemical stability of the pigment, (ii) successfully used transmission electron microscopy (TEM) to visualize and evaluate the quality of the pigment coating, (iii) developed a rapid and cost effective test for the chemical stability of the cured coating and (iv) suggested the use of optical absorption to qualitatively measure the pigment embedment depth. The development and utilization of these techniques did not only speed-up the development process, they also gave us a far more detailed knowledge on how different parameters and properties influenced the final coated product. The measurements and tests were far more robust and reliable for R&D work than standardized quality tests.

2. Experimental

2.1. Samples

Six different commercially available aluminium based metallic pigments have been investigated in this study (Table 1). The pigments are all of a non-leafing type and are protected by a silica coating. Silica is throughout this work conveniently used as a description of a Si-containing protective layer on the pigment particles. The silica might be in different microstructural and chemical forms including various oxides and hydroxides.

The chemical stability of the pigments is defined into three different categories based on information given by the suppliers. These pigments are all added to the same polyester based powder coating.

The powder coating used is based on polyester binder, curing agent, filler and additives according to a commercial formula used by DuPont. The components are mixed using a single screw Buss extruder, and grinded to medium PSD of $40\ \mu\text{m}$. The grinded powder was mixed with the commercial available aluminium flake pigments in Table 1 with a predetermined concentration. By using different process conditions, like various bonding techniques or dry blending methods when mixing base powder and the aluminium pigments together, it is possible to achieve different embedment depths of the aluminium pigments. Two different mixing procedures have been used. *Procedure 1* gave a deep pigment embedment and *Procedure 2* gave a shallow and more surface oriented pigment embedment. Application of the coatings has been done using a handheld electrostatic powder gun and cured in a convection oven for 10 min at $200\ ^\circ\text{C}$ giving a film thickness of $80\text{--}100\ \mu\text{m}$.

2.2. Measurements

The chemical stability of the pigments was tested by exposing $0.3\ \text{g}$ pigment to $100\ \text{ml}$ 10% w/w HCl at $25\ ^\circ\text{C}$ for 3 h. The amount of gas evolved was measured during the exposure.

A visual evaluation of the pigments before and after exposure in HCl was also done. 10% w/w HCl is prepared from concentrated (37%) HCl by adding $285.8\ \text{g}$ acid to $714.2\ \text{g}$ distilled water giving $1000\ \text{g}$ solution.

Chemical resistance of the aluminium-pigmented coatings was investigated using an Acidified Salt Spray (ASS) test following the ASTM G85–94 standard [10]. This test is originally designed for measuring corrosion resistance of coated aluminium. In the present work, however, it is used for monitoring the degradation of the aluminium flake pigments during exposure to an acidified salt spray atmosphere.

A new cost and time effective chemical resistance test for the cured powder coating is proposed. The test is denoted the HCl dipping test and consists of dipping the samples in 10% w/w HCl for some hours. As will be discussed in the result section there is a very good correlation between the ASS test and the HCl dipping test.

Chemical degradation of the samples is monitored during exposure both to ASS and HCl by measuring the L -value in standard colour measurements [11]. Samples are taken out of the cabinet/bath, and they are rinsed thoroughly in tap water followed by distilled water before drying. This rinsing procedure is arresting the degradation process. Exposure time is restarted on re-entrance of the samples in the ASS chamber or HCl bath.

The L -value is used as a quantitative measure of how much visible light a surface will reflect. Degradation of a surface will normally result in a decrease in the measured L -value, i.e., the surface becomes darker. A reduction in L -value by 1 percentage point is normally not visible for our eyes. The L -values given are averages for 3 repeated readings on three predefined locations on three parallel samples.

Optical absorption (OA) measurements are in this paper used as a qualitative measure for the average pigment embedment depth. OA is performed in reflection geometry at $\lambda = 8.4\ \mu\text{m}$. This wave-

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