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# Relaxation properties of particle filled coatings: Experimental study and modelling of a screw joint

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#### Abstract

The present study describes the mechanical behaviour of powder coatings used under very high compressive loads in clamping force joints. Carboxyl functional polyester powder coatings cured with hydroxyl functional β-hydroxyalkylamides with variations in amount of filler have been studied. The coatings were subjected to relaxation tests in tension and in compression. The tests in compression were performed in specially designed tests developed to study the behaviour of powder coatings under compressive loads in clamping force joints. The relaxation results for the matrix were used in a unit cell in micromechanical finite element (FE) model to predict the homogenised viscoelastic properties of the particle composite. These constitutive properties were subsequently used to evaluate the behaviour on a macromechanical scale in a screw joint. The model corresponds well with experimental data at ambient temperature. When increasing the temperature above the glass transition of the coating, however, the model predictions and experimental data differ. Experiments in compression show a much lower relaxation as compared to the FE model. The relaxation simulations of the coating under compressive loads from screw joints showed a significant sensitivity to the Poisson's ratio of the polymer matrix. As the Poisson's ratio approaches 0.5, the matrix becomes hydrostatically incompressible, which resulted in a negligible relaxation of the coating at the screw joint.

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#### 1. Introduction

The mechanical behaviour of a coating is dependent on both the chemical and physical properties of the material as well as the type of load the sample is subjected to. One area were this is evident is the use of pre-coated metal parts in load carrying constructions, e.g. bolted or screw-mounted clamping force joints. Especially in the vehicle industry, more and more details are coated before they are assembled into the final product. The coating in a clamping force joint will be subjected to high compressive loads. Rigid and dense coatings with excellent mechanical properties such as powder coatings have been introduced to fulfil the demands in these applications.

It is also of interest to model the behaviour of the coatings under high compressive loads in a screw joint. By using finite element (FE) models of the coating, the behaviour of the complete joint can be evaluated and the development time of new designs can be reduced.

The present study aims to use experimental data and develop a micromechanical FE model of a filler reinforced coating, which is homogenised and subsequently applied on a macromechanical scale in a screw joint. The goal is furthermore to evaluate both experimentally and by modelling how microstructural factors such as filler content and Poisson's ratio of the polymer matrix affects the behaviour of a thermoset polymer film on metal substrates under high compressive loads.

#### 2. Experimental

#### 2.1. Materials

Powder coatings based on carboxyl functional polyester resin (supplied by DSM) cured with  $\beta$ -hydroxyalkylamide (Primid XL-552, EMS-Chemie) were used to produce coatings

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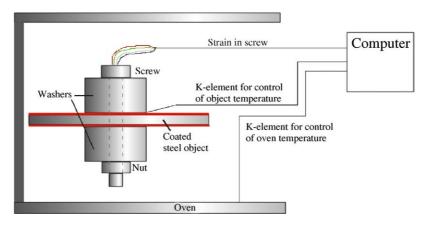


Fig. 1. Set-up of clamping force test.

with various pigment volume concentration (PVC). Barite, crystalline barium sulphate, was used as filler (Portaryte B15 supplied by Ankerpoort) with a mean size of 15 μm and a density of 4.4 g/cm<sup>3</sup>. All coatings were prepared with flow agent (Resiflow PV88) and Benzoin in order to achieve smooth coatings without defects due to evaporation of water during curing. The mixing recipes are listed in Table 1.

#### 2.2. Sample preparation

All coatings that were to be tested on a substrate were applied on phosphatised steel plates. The phosphatisation was performed in a nine step Bonder 28 Zn/Ni-phosphatising process with zirconium fluoride as passivation. Free-standing, unsupported films were prepared by applying the powder coatings on aluminised steel with a thin layer of poly vinylidene fluoride (PVDF) on the surface. The surface tension of the PVDF was low enough to facilitate poor adhesion between the experimental coating and the PVDF film and to ease the removal of the cured experimental coating, yet the surface tension was high enough to avoid crawling of the coating during flow. The coatings were applied with an electrostatic gun and cured at 190 °C (object temperature) during 15 min. The thickness of the cured coatings when applied to a steel substrate was measured with magnetic induction (Minitest4100). The thickness of the free-standing films was measured with a Mitutoyo micrometer device.

Table 1 Variations in content of coatings

Substances	Mixing recipes of coatings (g)		
	Unfilled	PVC: 15 vol.%	PVC: 25 vol.%
Curing agent, Primid XL-552	57	57	57
Resin, Carboxyl functional polyester	1000	1000	1000
Filler, Portaryte B 15	_	645	1218
Additive, Benzoin	3	3	3
Additive, Resiflow PV88	20	20	20

#### 2.3. Tensile properties

The tensile properties of the coatings were measured on free-standing films with a dynamic mechanical analysis (DMA), TA Instruments Q800. Relaxation measurements were performed with constant strain (0.2%) applied at the indicated temperature (25, 60 and 100 °C). The samples were 15–20 mm long and 3–5 mm wide with a thickness of 70–100  $\mu$ m. The length-to-width ratio was approximately 4:1 in all samples.

#### 2.4. Compressive properties in screw joint

A special experimental set-up was used to study the creep and relaxation of coatings under load, simulating the set-up of a clamping force joint constructed of coated steel objects, screw and nut (Fig. 1). In the set-up used, strain gauges were glued onto the elastic screw in order to measure the loss of clamping force due to relaxation of the coating during time and increase of temperature. The coating is applied on steel rulers and compressed between rigid steel washers with an inner diameter of 15 mm and an outer diameter of 37 mm, giving a compressive area of 900 mm<sup>2</sup>. A more extensive description of the experimental set-up can be found elsewhere [1].

The sample is mounted at ambient temperature and the loss of clamping force is measured with time. The samples were kept at 25  $^{\circ}$ C or heated to 60 or 100  $^{\circ}$ C, respectively, to accelerate the relaxation of the coating.

## 3. Modelling

#### 3.1. Viscoelastic model

A generalised Maxwell model consisting of an arbitrary number of rows with springs and dampers,  $1, \ldots, N$ , was used for the modelling of a viscoelastic material in shear, as shown in Fig. 2. The FE calculation software ABAQUS 6.4 was used and it supports a maximum of 13 rows. A very fine mesh of eight-node, reduced integration, linear hexahedral elements was consistently used for all FE simulations. The steady state value  $G_{\infty}$  denotes the equilibrium shear modulus. In hydrostatic loading of the

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