Investigation of Mechanical Properties of Polymer Coatings on Surfaces

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Article History Received: 10 January 2021; Revised: 12 February 2021; Accepted: 27 March 2021; Published online: 28 April 2021

Abstract: In this work, the mechanical properties of organic coatings based on epoxy resin (DGEBA DER 332) and two diamines comonomers (IPD and 3DCM) on the surface of aluminum and titanium alloy plates were investigated. It has been proven that Young's modulus and internal residual stress of coatings depend on the thickness of the coating, the degree of reaction and the substrate material. During the thermal cure cycle, a loss of curing agent was observed and thus a change in the degree of reaction of the coating. The results showed that Young's modulus and stress of thin coatings are very different from volumetric values.

Keywords: Metal plates, coatings, stress, deformation, polymers.

1. Introduction

Elasticity and resistance to deformation are one of the key physical characteristics in the modern coating industry [1-9]. In wide practice, three test procedures are used that determine the behavior of the coating under the influence of different types of deformation - resistance to cracking and/or flaking [10-17]. These are impact tests, flexural strength tests and indentation elasticity tests.

The impact test method also does not allow determining the values of mechanical characteristics. Impact strength indicates the ability of a coating to resist rapid deformation. A blow from the side of the coating is regarded as direct, from the side of the substrate - as a back blow. Both of these methods can be used to assess the impact resistance of coatings. The test consists of placing a coated metal plate with the painted or reverse side up under a freely falling weight, and then inspecting the damage caused by deformation. Depending on the weight of the striker weight and the height from which it falls, the coating will either break or break (rupture, cracking, peeling). The impact resistance value is essentially the magnitude of the maximum impact force that did not break the coating at a given film thickness. When testing for impact resistance, the thickness and elasticity of the substrate are important factors and must be controlled to obtain reliable results. The thickness of the paint film is directly related to impact resistance - the thicker the coating, the lower its impact resistance [18-24]. The degree of damage caused by an impact to the coating must be considered in conjunction with the other properties of the coating. Many coatings with the highest degree of resistance to external influences or to chemicals do not have high values of impact resistance [25-36].

Most often, the elasticity of a coating is controlled by two methods, the first of which is the bending test, which determines the ability of the coating to resist the bending of the substrate. The flexibility of the coating is checked as follows: a painted rectangular metal plate is bent on a cylindrical or conical rod, after which the coating is examined for cracks, discoloration, rupture and peeling from the substrate. If cylindrical rods are used, then a set of rods of different diameters is required, the conical rod allows simultaneous testing of the coating on different diameters, and the device is completed with one rod. Bending on a tapered rod exposes the coating to varying degrees of stretching, making it possible to determine, in addition to its elasticity itself, its adhesion characteristics. The purpose of the test on both types of cores is to determine the minimum diameter at which there is no cracking or peeling of a single or multi-layer paint film of a certain thickness. This diameter in millimeters is taken as the test result.

2. Methods for studying the mechanical properties of coatings

Tensile strength is determined using a press. The diagram of the press device is shown in Fig. 1.

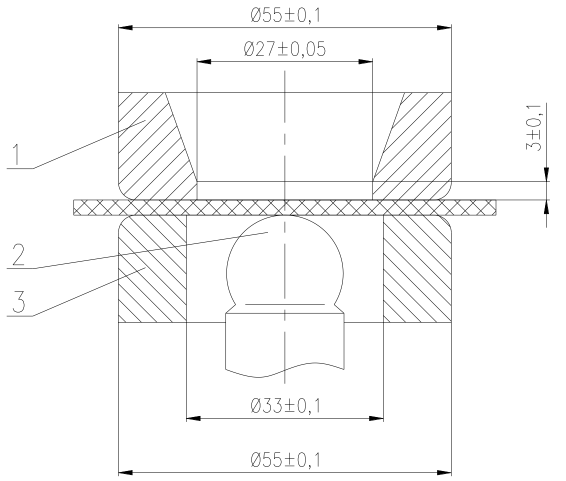

Figure: 1. Device-press. 1 - matrix, 2 - punch, 3 - pressure ring.

The sample is placed in the press with the painted side to the die and clamped tightly between the die and the clamping ring. The punch head must be in the zero position, i.e. touch the test specimen, and be at least 35 mm away from the transverse edges of the plate relative to the punch axis. The tensile strength of the coating is determined by the depth of indentation of the punch into the plate, expressed in millimeters.

Attention should be paid to an effective method for studying the mechanical characteristics of films and membranes, based on both experimental data and theoretical relations obtained from the nonlinear theory of thin shells, the theory of elasticity and plasticity [1-7]. This method uses the experimental setup shown in Fig. 2, intended for testing thin-walled round specimens, on one surface of which some medium acts, and on the other, a uniformly distributed surface pressure. The installation contains a load tank 1 with a flange 2, on the landing area of which a test sample 3 is installed, hermetically closing the cavity of the tank 1. Sample 3 is fixed along the contour with fasteners 6 using a counter flange 4. A branch pipe 5 is installed on this flange for filling the working medium 17. A line 7 with a valve 8 from a pressure source 9 (for example, compressed air or a compressor) is connected to reservoir 1 to provide one-way pressure on sample 3. Valve 10 serves to stabilize the pressure. A manometer 12 is connected to the reservoir 1 through pipes 11 to measure the pressure. The installation is equipped with a bracket 15 with a dial gauge 14 fixed on a platform 13 for measuring the geometric parameters of the shape of a deformed sample 3. The reservoir 1 has a valve 16 to relieve pressure before removing the sample from the device.

The tests are carried out as follows. The investigated sample 3 is fixed on the flanges 2 and 4 of the load tank. If necessary, the working medium 17 is poured into the cavity of the pipe 5. The liquid level is set at 0,03-0,05 m above the sample so that it is completely buried in it. The surface load on the sample under study is created by supplying pressure to the reservoir 1 from the pressure source 9. The sample then sags, forming a dome, the height of which is fixed, for example, by the indicator 14. Periodically, at a predetermined time interval, measure the geometric parameters of the sample, for example, the deflection and thickness at the top formed dome.

In theoretical processing of experimental results, in particular, relations for thin plates and flexible elastic membranes with large changes in curvature and plastic deformations are used. For example, for an elastic thin plate of thickness h in the case of average bending, the elastic modulus can be determined by the formula:

$$E = \frac{3(1-\nu^2)pa^4}{16hH(h^2+0{,}488H^2)},$$

where ν – Poisson's ratio, a – radius of the working part of the sample, p – tank pressure, H – plate deflection, h – plate thickness.

To determine the mechanical characteristics of coatings, including nanocoatings, obtained in the “coating-substrate” system, the following approach is used: the properties of the substrate and the “substrate-coating” package are studied separately, and then the mechanical characteristics of the coating are determined. With this approach, the modulus of elasticity of the coating is determined by the formula:

$$E_{coat} = \frac{E_c\left(h_{coat}+h_{surf}\right) - E_{surf}h_{surf}}{h_{coat}}$$

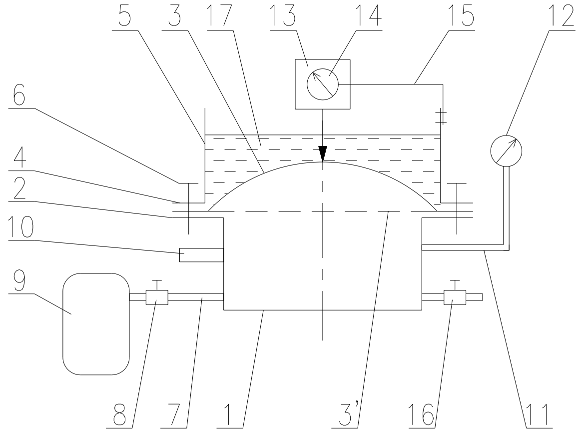

Figure: 2. Scheme of the experimental setup.

Young's modulus and residual stress were determined using three-point bending tests on a FLEX 3 (TECHLAB) instrument at room temperature. The distance between the two lower cylindrical supports (each 6 mm in diameter) - span (L) - can be adjusted from 4 mm to 150 mm with an accuracy of 1/20 mm. The traverse was moved by a microcomputer-controlled stepper motor and a speed reducer. The offset corresponding to one step is 25 nm. The slider speed ranged from 0,025 to 1 mm/min. In this work, a constant speed of 0,1 mm/min was used. A full-scale load cell with a sensitivity of 5 mN and a range of 20 N was installed under the crosshead. The upper edge of the support prism (12 mm in diameter) was attached to the other end of the load cell. The movement response of the sample was measured with a load cell. The dependence of displacement on load was recorded using a microcomputer and displayed as graphs on the computer screen in real time. The slope of the curve within the elastic region is calculated using the linear regression program. The slope of the experimental curves was corrected to take into account the rigidity. For a given span and a coated sample, 3 curves were plotted. To minimize the effect of polymer relaxation, the samples were placed on the lower supports for 3 min before any testing. The mean of the respective slope values was compared with its standard deviation. If the standard deviation was less than 1%, then the data was saved, otherwise a new set of tests was started.

3. Mechanical properties of bulk and coated materials

The properties of aluminum and titanium substrates (after degreasing and chemical etching) and bulk DGEBA / 3DCM and DGEBA / IPD for various curing cycles are presented in the table 1.

Table 1.

Mechanical properties of DGEBA/3DCM coatings on various substrates

Substrate	Curing cycle (°C/h)	Thickness (μm)	Curvature radius (mm)	Residual stress (MPa)	Young's modulus (GPa)
Titanium chemically etched	130/2	268 ± 84	609	14.1	2.3
		633 ± 48	272	19.7	1.9
		1072 ± 55	234	32.7	1.6
Aluminium degreased	200/1.5	149 ± 18	1245	11.8	4.8
		280 ± 21	723	9.9	4.4
		415 ± 82	617	7.6	4.4
Aluminium chemically etched	200/1.5	194 ± 31	893	11.7	4.4
		352 ± 51	671	8.0	3.4
		596 ± 34	418	9.4	3.4
		781 ± 58	295	15.0	2.6

Young's modulus was determined by flexural testing, compared with values obtained from tensile tests. Good agreement was noted. There was no significant effect of surface treatment on the values obtained. For polymers, an increase in T_g and degree of reaction was observed, as well as a decrease in Young's modulus with an increase in the curing temperature. This reflects the influence of relaxation processes in polymer materials, which increase with an increase in the degree of polymerization. Residual stresses were found for all coated samples. They depend on the type of substrate, its processing, the type of amine and the curing conditions. Residual stresses can increase with increasing coating thickness. Take the maximum values for cycles 130℃ for 2 hours, or the minimum – 108℃ for 1,5-2 hours.

Young's modulus of coatings depends on the type of substrate, surface finish, amine type and curing conditions. In the general case, Young's modulus decreases with an increase in the thickness of the coating and its approach to volumetric values. Smaller values were observed only for DGEBA/3DCM (130℃, 2 hours) coatings applied to chemically etched titanium. The dependence of the modulus on the thickness of the coating can reflect the formation of the polymer / substrate interphase layer, the change in the network, and the loss of the hardener between bulk and facing materials. The pre-cure yielding the same conversion rate (r) for the base stock resulted in residual stresses and an increase in Young's modulus. Thus, the chemical, physical and mechanical properties of coating materials differ significantly from volume units due to the formation of an interphase layer.

However, the values obtained for a thin coating (67 μm, DGEBA/IPD (130℃, 2 hours) on a degreased titanium substrate) high values are not reliable. This demonstrates the limit of applicability of the model used, which assumes that the coating system can be analyzed as a two-layer system.

4. Conclusion

The results obtained showed that the mechanical properties of the investigated polymer coatings on the surface of metal plates, such as the modulus of elasticity and residual stresses, are very different from bulk materials and thin films. The formation of a mixed polymer / substrate phase is considered. The mechanical model used in this work needs to be improved to take into account the full potential of interfacial formation.

5. Acknowledgements

The work was carried out with the financial support of the state project of the Ministry Science and Higher Education of the Russian Federation “Theoretical and experimental research in production and processing of advanced metal and composite materials based on aluminum and titanium alloys”, project No. FSFF-2020-0017.

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