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Development of a scratch test method to determine adhesion and strength properties of coated steel sheets

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Abstract. A simple method to assess adhesion properties of organic coatings by Ω parameter was developed and tested, where Ω is a ratio of indentation speed at a linearly increasing normal load to a resulting force on the indenter at the end of plastic deformation, when the indenter reaches a substrate. A relationship between Ω parameter and a coating hardness H determined by Oliver and Pharr method is shown.

1. Introduction

Presently, many goods are produced with various coatings. These coatings serve as anti-corrosive, decorative, wear resistant, tribological (for low or high friction purpose), galvanic, biocompatible, optical, magnetic and conductive coatings, as well as chameleon coatings for military machinery, waterproof coatings, intumescent fire-resistant coatings, transparent coatings for optics and glassware, highly antibacterial coatings, etc. [1].

The main requirements to coatings include provision of expected performance, low cost of a coating material and that of the process involving its application onto a substrate.

The most important performance parameters of any coating are adhesion properties, i.e. resistance to damaging and peeling off a substrate. There are plenty of methods used to determine these properties [2]. Most common are: a bend test [3-5], a cross-cut test [6], a pencil test [7] and a pull-off test [8].

A lot of attention is now paid to evaluation of adhesion properties of various coatings using a scratch test [2, 9, 10, 11]. The test involves scratching of a coating (i.e. making a 'track') with a small-radius tip indenter (see figure 1) in two measurement modes: 1) at a constant vertical speed of the indenter, and 2) at a constantly increasing load on an indenter. The first mode is generally used more frequently. Horizontal speed of the indenter is not high and is usually about 3-4 cm/s for both modes.

After scratching, a sample is analyzed with a microscope. Use of a 3D-surface analyzer as a microscope makes it also possible to determine the surface characteristics along the scratch, such as its depth, width, curvature radius etc. This allows to evaluate a coating failure pattern alongside the scratch (local cracking, wedging, recovery, spallation, etc.) [12, 13] and to calculate a load which results in this failure.

A scratch test is most advantageous for being able to reproduce very realistic coating failures (i.e. scratches, pin holes, galling, etc.). In some studies, scratch test results are correlated with physical parameters of a coating (surface energy, mechanical properties of a surface layer, friction properties) [1, 11, 14-16]. Another benefit of a scratch test is that the results can be expressed numerically to exclude any subjective estimation.



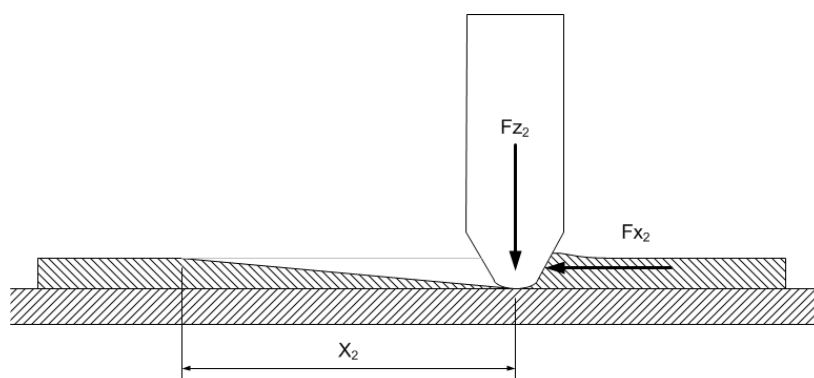


Figure 1. Coating thickness and adhesion measurement principle.

A scratch test, however, has got a disadvantage which lies in the fact that its results strongly depend on the initial frictional properties of a surface to be analyzed [17]. It requires a proper pre-cleaning of a surface from absorbed molecules. Unfortunately, the cleaning procedure is specific for every type of coating and is therefore to be thoroughly described while representing the test results.

The purpose of this work is to develop a simple scratch test procedure for numerical evaluation of adhesive properties of coil coatings. This method shall allow for an unbiased selection of suppliers of various raw materials for production of coated steel at PJSC MMK (Magnitogorsk, Russia), as well as improved application techniques to achieve better coating adhesion along with lower consumption of raw materials.

2. A simple method for evaluation of adhesive and strength properties of organic coatings

The research was done using the following equipment of Microtopography Research Center, MSTU: 1) an optical surface analyzer Contour GT K1 by Bruker, USA; 2) a stylus surface analyzer MarSurf XR20 with XT20 by Mahr, Germany; 3) a measurement station UMT-1 by Bruker, USA, for determination of physical and mechanical properties of materials.

Figure 2 gives an example of the coating thickness and adhesion measurement report. Upper image shows the results of scratch test track measurements made with Contour GT K1. Lower image shows the measurements results obtained by UMT-1. Both images are in the same horizontal scale.

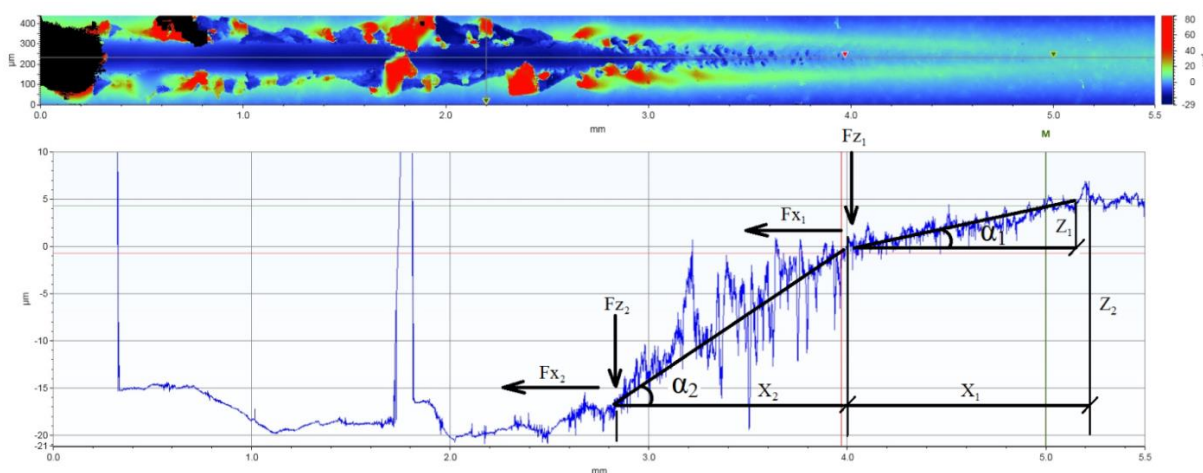


Figure 2. Results of a scratch test track measurement.

X_1 – track length of elastic deformation;

X_2 – track length of plastic deformation;

Z_1 – track depth at the end of elastic deformation;

Z_2 – track depth at the end of plastic deformation;

F_{z1} – normal force on indenter at the end of elastic deformation;

F_{z2} – normal force on indenter at the end of plastic deformation when indenter reaches a substrate;

F_{x1} – tangential force on indenter at the end of elastic deformation;

F_{x2} – tangential force on indenter at the end of plastic deformation when indenter reaches a substrate.

Adhesion properties of coatings have been measured under the following conditions:

- 50x50 mm samples were glued onto a steel substrate and fixed in a measurement station;
- vertical loading mode was applied, i.e. linearly increasing load F_z from 0.2 N up to 15 N with testing time $t=720$ s;
- indenter speed is $V = 0.007$ mm/s on 5 mm track length;
- type of indenter: Rockwell C scale (spheroconical diamond tip with 120° angle and 0.2 mm radius);
- the tests are done at a room temperature;
- measurement is made at least 5 times;
- cracking start (F_{z1} , Z_1) and reaching of zinc substrate by indenter (F_{z2} , Z_2) are determined by optical images and load diagrams (see figure 2).

Table 1 gives characteristics of samples submitted for analysis done by Coatings Laboratory, PJSC MMK.

Table 1. Samples of coatings for analysis.

Sample number	Main coating	Primer	Organic coating
1	Zn	Basf	no coating
2	Zn	Basf	Valspar RAL 9003 (top)
3	Zn	Basf	Prime top (back)
4	Zn	Basf	DYO (back)
5	Zn	DYO	DYO RAL 6005 (top)
6	Zn	DYO	DYO (back)
7	without Zn coating		no coating
8	without Zn coating	Basf	Valspar RAL 9003
9	without Zn coating	Basf	DYO (back)

Main purpose of the analysis was to compare adhesion properties of the following samples pairs: 1–7; 2–5; 2–8; 3–4; 3–6; 8–9

Producers of coated steel were concerned about coating damage between coil laps during handling and transportation, whereas processors/customers were concerned about possible coating damage during manufacture and service life of finished parts.

Plastic properties of coating materials were determined by a tangent of α_2 (see figure 2), i.e. by the following parameter:

$$K = \frac{Z_2 - Z_1}{X_2} \quad (1)$$

This parameter indicates the indentation speed at a linearly increasing normal load on an indenter. Coated samples with smaller K values are less damageable.

The load when plastic deformation is considered to be complete and the indenter reaches a substrate may be defined as follows:

$$F = \sqrt{F_{x2}^2 + F_{y2}^2} \quad (2)$$

Coated samples with greater F values are regarded as less damageable.

Therefore, the adhesive properties of a coating can be estimated by its resistance to damage (failure) as follows:

$$\Omega = \frac{F}{K} \quad (3)$$

Coated samples with greater Ω values are regarded as less damageable.

Results of adhesion measurements for tested samples are presented in table 2 (see also figure 2).

Table 2. Adhesion characteristics of analyzed samples.

Sample characteristics	Sample number	1	2	3	4	5	6	7	8	9
	Main coating	Zn	Zn	Zn	Zn	Zn	Zn	no Zn	no Zn	no Zn
	Primer	Basf	Basf	Basf	Basf	DYO	DYO	Basf	Basf	Basf
	Paint coating	–	Valspar RAL 9003 (top)	Prime top (back)	DYO (back)	DYO RAL 6005 (top)	DYO (back)	–	Valspar RAL 9003	DYO (back)
Measured characteristics	X_1 , mm	0.45	1.22	1.24	1.50	1.12	1.16	0.62	1.13	1.64
	Z_1 , μm	2.36	1.61	3.70	5.30	6.50	4.46	2.50	2.98	5.50
	F_{z1} , N	1.6	3.6	4.0	4.6	3.6	3.6	2.0	3.5	5.13
	F_{x1} , N	0.24	0.96	0.7	1.0	1.0	0.8	0.4	1.0	1.2
	X_2 , mm	1.34	2.29	2.44	2.35	2.71	2.14	1.53	2.60	2.49
	Z_2 , μm	7.6	19.6	17.1	14.4	23.4	13.9	7.4	22.2	16.5
	F_{z2} , N	4.2	6.8	7.2	7.2	8.3	6.6	4.4	8	7.7
	F_{x2} , N	1.1	2.8	2.5	2.2	2.8	2.5	1.2	3.3	2.8
	Oliver and Pharr hardness, GPa	–	1.13	1.13	2.31	0.75	1.74	–	1.37	3.77
	Thickness, μm	7.60	19.60	2.44	2.35	23.40	13.90	7.40	22.20	16.50
Coating characteristics	K	3.91	7.86	5.49	3.87	6.23	4.41	3.22	7.39	4.42
	F , N	4.3	7.35	7.61	7.53	8.76	7.06	4.56	8.65	8.19
	Ω , N	1.10	0.94	1.38	1.95	1.40	1.60	1.42	1.17	1.85

Ω parameter allows for numerical representation of adhesion properties without any subjective estimation.

Based on obtained values of Ω parameter, the coil coating technique at PJSC MMK was updated.

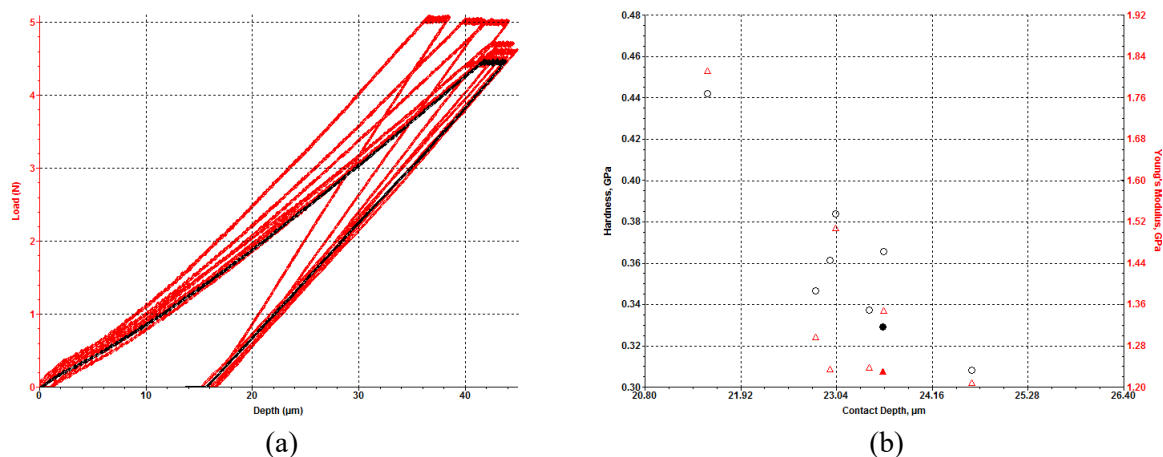
3. Correlation of Ω parameter with Oliver and Pharr hardness

The obtained data made it possible to assess any relationship between Ω parameter and Oliver-Pharr coating hardness [14, 16].

Coating hardness has been measured under the following conditions:

- 50x50 mm samples were glued onto a steel substrate and fixed in a measurement station UMT-1;
- vertical loading mode is applied, i.e. linearly increasing load F_z from 0.1 N up to 1 N with testing time $t=90$ s;
- exposure for 90 s until 1 N is reached;
- measurement is made at least 10 times;
- type of indenter: Vickers diamond tip;
- distance from a sample edge to indentation point is at least 20 mm;
- the tests are done at a room temperature;
- analysis of load-depth diagrams ($F-h$) was carried out according to Oliver and Pharr method in Viewer software by Bruker, USA.

Measurements were made in full compliance with standard [18]. Examples of Oliver and Pharr hardness measurements are presented in figure 3(a), where the left section corresponds to loading and right section corresponds to unloading. The resulting hardness value was averaged on 10 measurements taken at different places of the sample. Dispersion of these measurements is shown in figure 3(b).



(a) Indentation load-depth diagram of loading and unloading according to [18],
 (b) Martens hardness (GPa) and Young's modulus (GPa) versus contact depth.

Figure 3. An example of Oliver and Pharr hardness measurement report.

Figure 4 shows a Ω parameter versus Oliver and Pharr hardness H .

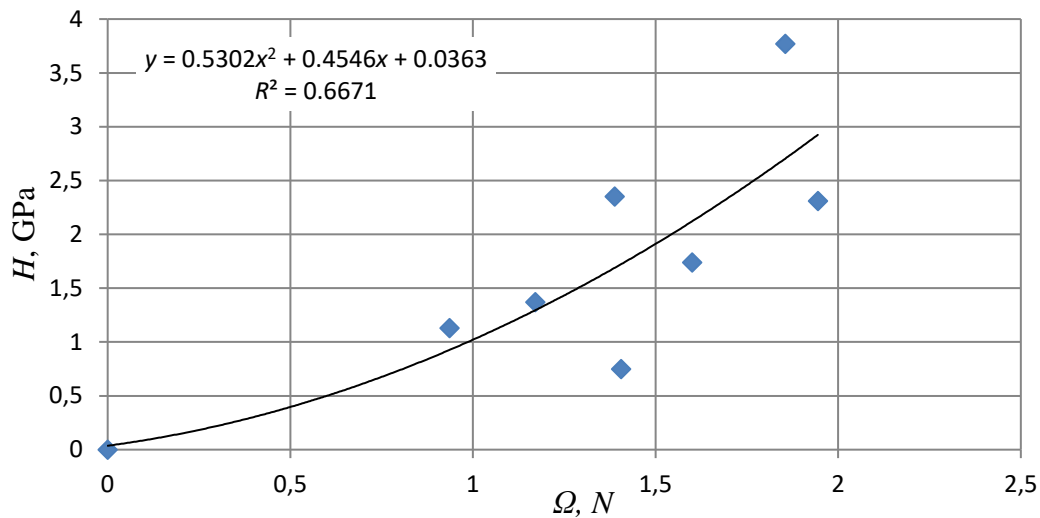


Figure 4. Ω parameter vs. Oliver and Pharr coating hardness H .

According to the diagram, the coating failure with vertical (i.e. normal) indentation (Oliver and Pharr method) and that with simultaneous normal and tangential indentation (a technique suggested to assess the Ω parameter) have a nonlinear relationship. This nonlinearity can be explained by different failure modes used in hardness measurements and scratch tests [19].

4. Conclusions

A simple method to assess adhesion properties of organic coatings by Ω parameter was developed and tested, where Ω is a ratio of indentation speed at a linearly increasing normal load to a resulting force on the indenter at the end of plastic deformation, when the indenter reaches a substrate. A higher value of Ω parameter means a higher resistance of a coil coating to failures.

A correlation of Ω parameter with Oliver and Pharr coating hardness H is presented.

Based on the above, Ω parameter can be suggested for assessment of coil coatings' resistance to damages (i.e. scratches, etc.).

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