

CONSTRUCTION OF A SCRATCH TEST MACHINE

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Abstract. *This present work refers to the construction of a scratch test machine build in the Federal University of Rio de Janeiro. The Scratch test is used to quantify the quality of a coating material. It is used to study material, failure, and development of theories of coating. This test consists on a tip used to scratch the coating and measure the normal and friction force necessary to break the coating. The scratch test was conceived taking advantage of an ABBE-Komparator. Three systems were developed to convert this machine. One to move the specimen, other to apply a normal force, and another to measure de friction force. Those systems were designed to allow the machine to be used in the former use with little trouble. The system used to apply normal force was conceived using pneumatics, and was conceived to apply forces up to 200 N. The drive system was designed using a rolled ball precision screw. The velocity of the specimen is specified up to 10 mm/min. Both the normal and friction force are measured to analyze the data of a scratch test. The machine described in this paper is able to work under the same conditions of commercial machines.*

Keywords. *Scratch Test, Coating, Tribology.*

1. Introduction

With the current technological advances, the demand for materials with better tribological qualities has been increasing. To obtain materials with these characteristics, techniques of coating and superficial treatments are used. There are some materials used for coating, some of them have the advantage of mechanical properties and others have tribological properties. Usually, intending to obtain a material that combines the two characteristics, a substrate with those mechanical properties and a covering with the desired tribological properties are used.

There are many techniques of superficial treatment and coating deposition capable to modify the properties of the surface of materials as describe by Hogmark et all (2000) and Podgornik et all (2003). The choice of the coating and adequate technique depends on the material of the substrate, the efforts that the material is submitted and the tribological necessities.

To obtain the tribological properties, the superficial finishing and the desired resistance, the coating must possess some specific properties. This combination of properties is not easily achieved, since some factors (hardness, elasticity, etc) can influence more than one property.

Thus, the choice of the superficial treatment and the coating requires a great knowledge of each used technique as well as a compromise between the desired properties and the economic limitations.

The adhesion is defined as being the capacity of the film to adhere to the substrate. The measurement of this property is not trivial because it depends of the properties of the film, the substrate and the interface. That way, the adhesion is defined qualitatively, based in comparisons between patterns.

The value of the adhesion load can be calculated between the value of Van Der Waals forces and the cohesion forces, it can be between 100 and 3000 N mm⁻² according to Perry (1983). Most of the adhesion tests only offer results when the load is relatively low (100 N mm⁻²) except for the scratch tests and the shock waves.

To study the adhesion of a coating many techniques can be used as described by Rickerby (1988), some of which are scratch test, shock waves and "Topple" test.

The scratch test is the most used technique to measure the adhesion of hard films.

2. The scratch test

The scratch test is used to estimate the adhesive strength between the film and substrate according to Bull et al (1988). Basically, this test consists in applying a normal load concentrated in one region using an incisor and producing a relative movement between the sample and the incisor. Changing the value of the load you can obtain the critical load to which the coating is removed. This value is used in a comparative way, in other words, this test can obtain information related to the adhesive straight but, a more complex theory is necessary to determinate its precise value.

A Rockwell C diamond is used as incisor (120 degrees cone with a 200 μm tip radius). The groove made by the incision must not exceed the incisor's diameter to assure that only the edge of the diamond acts on the film.

The incisor, in contact with the sample, is submitted to a variable load. This load usually varies in steps of 1 N (the maximum load is 200 N). This variation (1 N) is due to the fact, described by Sekler et al (1988), that experimental and measurement errors make a better resolution enviable. According to studies, accomplished by Bellido-González et al (1995), in the critical load measurement of some materials (like Al_2O_3 on tool steel, TiN on tool steel, DLC on stainless steel), these presented critical load around 100 N.

Normally grooves from 5 to 10 μm of width and spaced of 1 mm are made to avoid the mutual influence. These grooves length vary according to the necessity (12 – 30 mm). This variation is due to different types of tests, they can be accomplished making multiple grooves with different loads or one groove with a varying load ramp. The choice between these two kinds of tests depends on the equipment and needed information.

According to Bul (1999), the scratch test can be use to make a correlation between its parameters and the wear rate. Other correlation possible is with the hardness, Burnett (1987) describes this relationship in terms of elastic-plastic indentation and illustrates it with experimental data.

3. Critical load definition

The critical load is defined as been the maximum normal load that can be applied in a sample without causing damage to the film. Nevertheless each researcher has a different opinion on how much damage is permitted. This choice is based on the propose of the film and the capacity of identify the damage on the film.

According to Perry (1983) the critical load is achieved when the incisor removes in a constant way all the film in the groove. In other hand, to Sekler et al (1988) it's defined as the minimum load in which surface damages start to occur, originating in adhesion or cohesion failure.

Thus, the definition of the critical load depends on the capacity of their laboratories to determine the failure of the film and the function of the coating.

4. Methods to detect critical load

There are several methods applied to determine the critical load. These methods allow detecting the moment of the films rupture and linking it to the applied load. Some of the most used techniques are described below.

4.1. Acoustic Emission (AE).

An accelerometer mounted solidary to the incisor is used to detect acoustic emissions. This accelerometer is appropriately chosen to not be sensitive to environmental vibrations and equipment frequencies vibration. According to Sekler et al (1988) a sensor that can detect acoustic emissions from 50 – 400 kHz is used, avoiding equipment vibration (0-30 kHz), this sensor's signal must be amplified (120 dB).

The acoustic signal obtained is produced when the tension made by the incisor overpasses the limit tension of the material, making it fail. This tension produces a shock wave diffused in the sample. After achieving the critical load, series of shock waves are created making the AE signal not continuos.

Since the intensity of the AE signal contains information about the energy released by the deformation of the film, it is integrated providing an energy spectrum. The sensor's sensibility can also be limited so that small energies are discarded adjusting the sensor operator's standard failure, as shown on Fig. (1). The occurrence of these failures events has random nature, so it's impossible to predict the amplitude or the repetition tax.

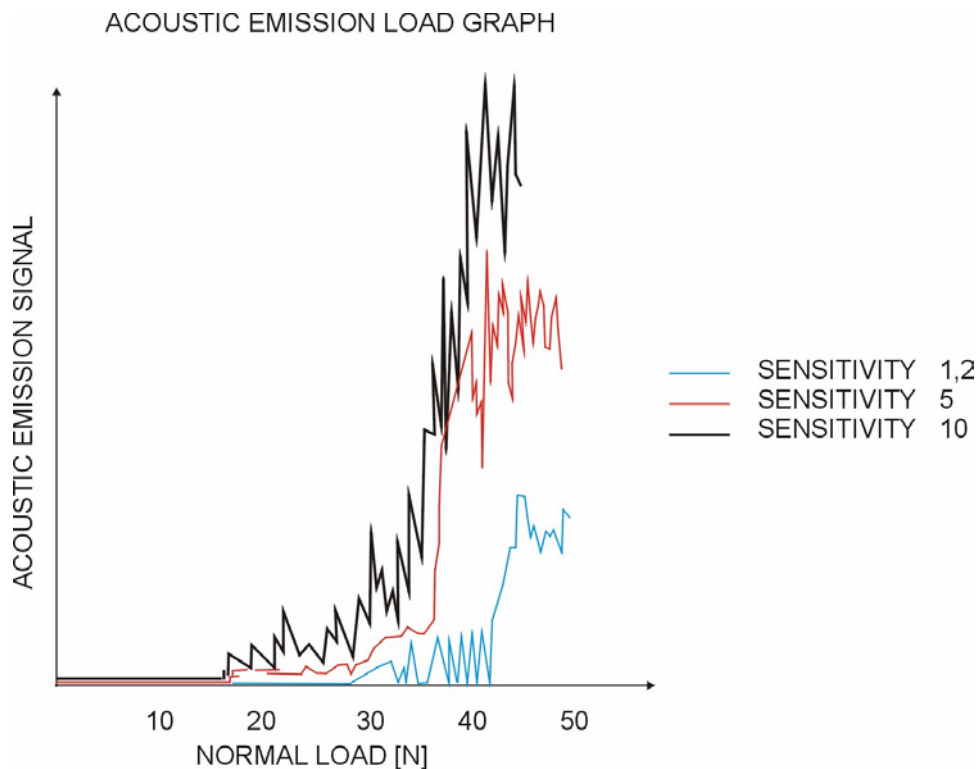


Figure 1. Example of sensor's sensibility variation (Sekler, 1988).

To determine the critical load two techniques can be used. The first one is based on the determination of the straight line tangent to the curve when the signal starts to rise. The intersection between the tangent line with the base of the diagram ("zero" AE) represents the critical load. The other technique involves determining the point at 10% of AE signal amplitude. At this point the load is considerate critical. That way, the critical load determination depends on the used criterion, but the techniques described above provide similar results according to the ones obtained by Sekler et all (1988).

4.2. Tangential force

By monitoring the tangential force between the incisor and the sample, it's possible to relate the increase of it to the film's rupture. To determine the adhesion is necessary that the measurement of this force has been influenced only by the adhesion force. Nevertheless, the tangential load measurement is influenced by the loss of adhesion between the film and the diamond tip, the dynamic influence and friction strength.

Measuring the tangential load, a scratching coefficient can be obtained, which is the relation between the normal load and the tangential load (F_n/F_t). This coefficient is plotted in relation to the relative displacement between incisor and sample or to the normal load, Fig (2). Through this graphic the value of the critical load is obtained and the moment when it occurs. This determination is made using a constant variation of normal load and estimating the friction force and displacement. It is important to remember that this coefficient doesn't refer to the friction coefficient, because in this case scratching coefficient depends also on the plastic deformation of the set substrate-film.

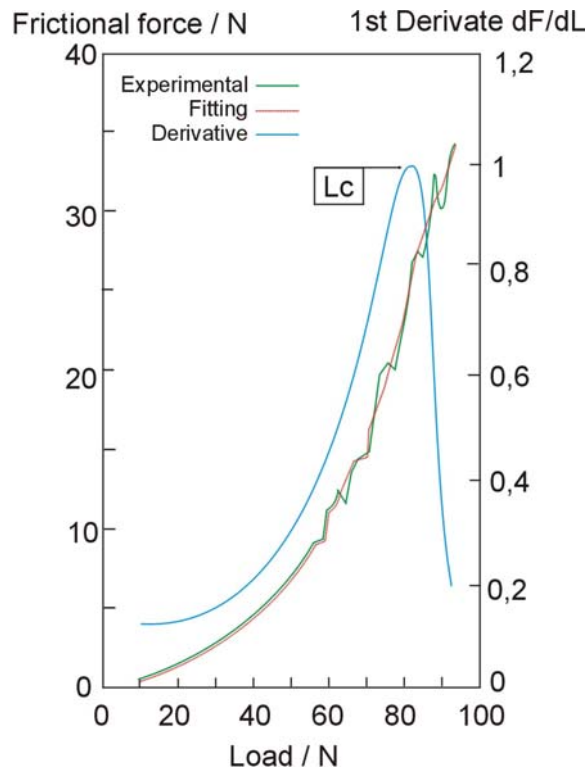


Figure 2. Example of tangential load graphic of Al_2O_3 in steel Bellido-González et all (1995).

Sekler et all (1988) questions the utilization of this method to detect the critical load, because it's not possible to determinate precisely the critical load without other technique. However, for films with great adhesion strength, this is an important tool to study film's properties. The measurement of the tangential force is the more reliable method of determination, according to Bellido-González et all (1995).

The measurement of the tangential force can also be used for test to estimate the wear of the film with constant load. For this type of measurement the optical methods don't have great precision. By measuring the tangential force, mathematically, the point of the curve's inflexion can be determined, and it marks the moment in which the incisor makes contact with the substrate, according to Bellido-González et all (1995).

The types of tangential load and normal load graphics can indicate the kind of groove as shown in Fig. (3).

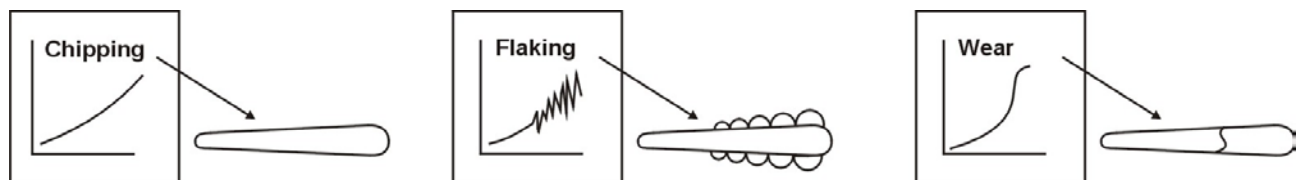


Figure 3. Types of curves obtained (force X load) and their failure mechanisms (Bellido-González, 1995)

4.3. Microscope observation

The most used method is the microscope scratch's observation. This can be set up on the test equipment or be used after the test conclusion. According to Sekler et all (1988), to verify the existence and examine these scratches, increases from 50X to 100X are used to locate the scratch and increases from 200X to 250X for examining the scratch.

To determine the critical load using microscopy is needed a complete comprehension of the mechanisms that lead to the failure and how to identify them. Identifying the groove it can be related with the normal load used to make the scratch.

5. Groove's identification.

The types of grooves that occur in coatings depend on the properties of the substrate and film.

If the film is soft comparing to the substrate Plastic deformation will occur. In that case the critical load is defined as been the load capable of exposing the substrate. However the qualification and quantification of this groove are not trivial, techniques to its determination must be used.

To a hard film in a soft substrate the most common failure comes from the groove interface, however the presence of cracks and deformation on this region can be observed.

The recognition of grooves and their failure modes can be study in more specific paper of Arai et all (1987), Bull (1997).

6. Scratch test equipment.

The scratch test equipment was elaborated adapting an equipment of photographic films measurement. It's the ABBE – Komparator modell B, manufactured by Karl Zeins de Jena in the 70's. Fig. (4).

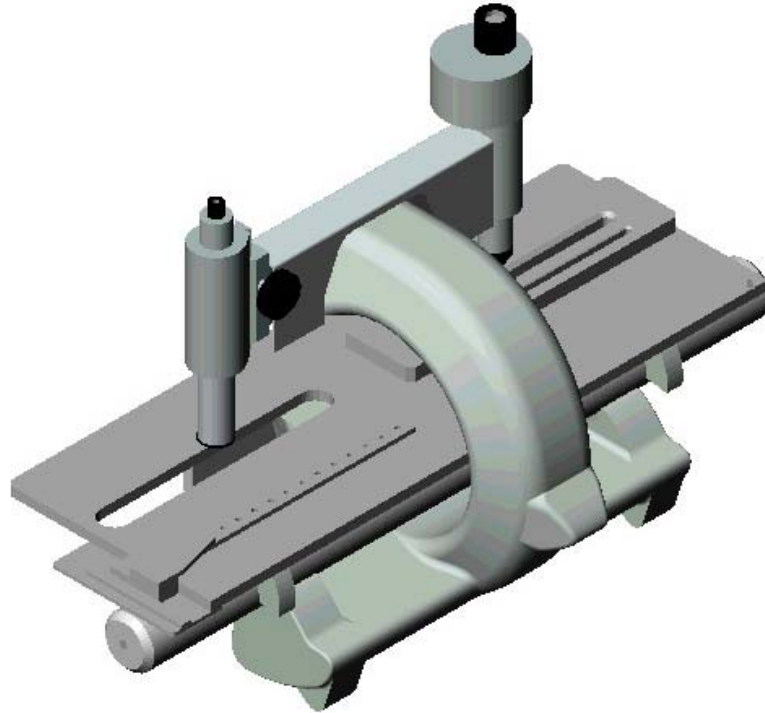


Figure 4. Abbe Komparator modell B without modifications (Soares, 1997)

6.1. Abbe Komparator's description.

The choice of adapting this equipment was based on these aspects:

(1) This equipment has a rigid shaft fixed to the base of the guide capable of supporting the efforts produced by the appliance of the normal load. This base is made of cast iron in the shape of a "C" with the guide table located in the center. This construction allows the utilization of the base to provide the normal load to the sample.

(2) The equipment has a guide precise and rigid enough to make possible the displacement of the sample over the load without considerable deformations. This guide is made of a stainless steel tube with a 46 mm external diameter and 590 mm length. On this guide there is a cylinder of half-revolution with an 85.5 mm external diameter and 380 mm length. This cylinder is supported on the guide through three rolling in each edge put in 120 degrees as Fig. (5) shows.

To assure the precision of the main guide, a secondary guide provides a third base point, avoiding the table to turn over the guide axle. This secondary guide is made by two rolling based on a stainless steel plate fixed to the base as shown on Fig. (5).

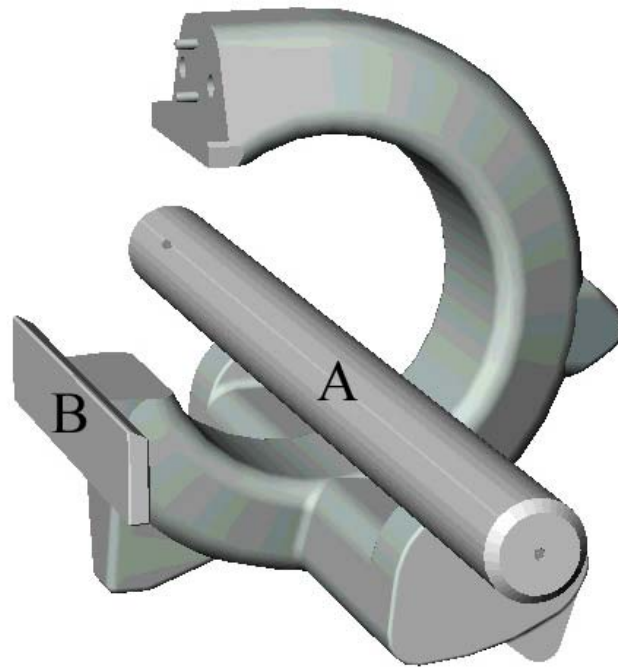


Figure 5. Guide's diagram: (A) Main guide e (B) Secondary guide (Soares, 1997)

The position of the rolling on both guides allows the adjustment of the clearance and consequently a better precision of sliding.

Another main guide's characteristic is the existence of course bounders that allow a better placement of the table. This bounder has an adjustment system with a manual screw thread capable of a sharp adjustment of the bounders.

(3) Availability of equipment and the possibility of using it with the scratch test function or the photographic films measurement.

6. 2. Adaptations and Assemblage

To adapt this equipment, four basic systems were developed: the first one to make the displacement of the table possible, the second to allow the appliace of a normal load, a third one to measure the tangential load and the fourth to allow the sample's placement. Each one of the systems is explained below.

6.2.1. Table displacement mechanism

The table where the sample is fixed must be displaced with a constant and relatively low speed (up to 10 mm/min), so Soares (1997) developed a system composed by an engine, a shaft for spheres and a screw nut of spheres. Fig. (6). This system is based on fixing the nut to the equipment's pole, the engine operates the shaft that displaces the table.

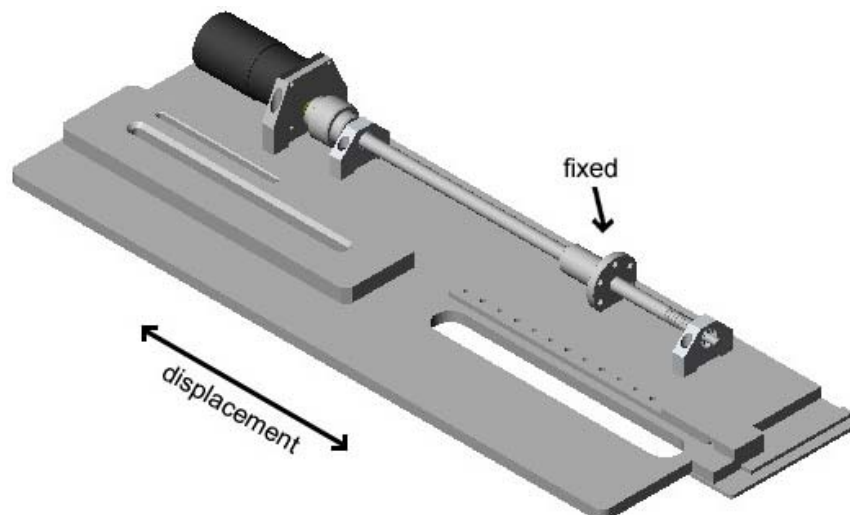


Figure 6. Table displacement mechanism graphic (Soares, 1997)

When the nut is fixed, the table's displacement is proportional to the engine's rotation. The table's speed is determined by Eq. (1):

$$V_{mesa} = \omega_{fuso} \times P_{fuso} \tag{1}$$

Where, the shaft pace (P_{fuso}) is defined and the angular speed of the shaft (ω_{fuso}) is determined by the rotation speed in the reducer's speed connected to the engine.

The shaft and the sphere's nut used were both chosen from NSK's Catalog (1995). The shaft external diameter is 10 mm and the pace is 3 mm. The spindle is based in two rolling, chosen for having a useful life capable of attending the necessity of the equipment besides having the appropriate sealing. The calculus of the efforts suffered by the shaft, as the movement system's dimensioning, were accomplished by Soares (1997).

The engine used is from Maxton with a 1/100 reducer connected. This engine is controlled by a controller plate LC-3003, which allows the engine's rotation speed to be determined by the feeding tension. This plate supplies a necessary current to guarantee that the speed is independent of the torque required by the system.

The engine-shaft connection is made using an Oldhan joint, this choice was based on its constructive convenience.

6.2.2. – Normal load appliance mechanism

The normal load appliance system was developed using pneumatic equipment. This choice was based on the simplicity of the system, the availability of a pneumatic line in the laboratory, and on the interest on development of this line to academic and research ends.

The load application system composition is:

The pneumatic cylinder Festo, with a 25 mm diameter, is dimensioned to guarantee the normal load appliance on the desired range (0 – 200N). This calculus considers the line pressure in the laboratory of metrology of UFRJ with have a capacity of 0.6 to 0.8 MPa. Using this information and Eq. (2) it can be calculated the maximum force that this cylinder can give.

$$F = P \times A \tag{2}$$

For this pressure line, the normal force is from 290 to 390 N. Since the desired range of forces are between 0 to 200 N, it is used a pressure regulator valve. This valve is capable of controlling the pressure from 0.05 to 0.8 Mpa.

A Bosh guide's is used to guarantee that the cylinder and the sample are perpendicular. This guide also stops the cylinder from spinning.

In the control system, Fig. (7)., many sub-systems of the pneumatic control system can be noticed.

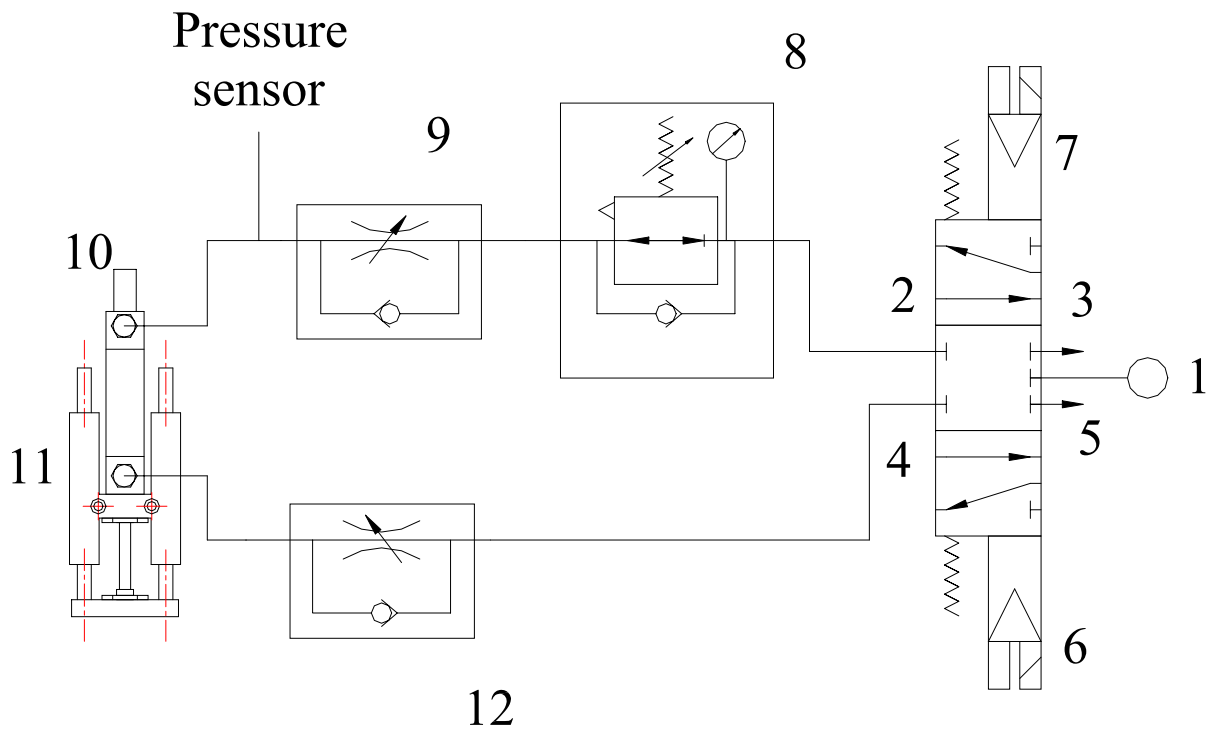


Figure 7. Normal load appliance mechanism graphic

In Figure (7), the number 1 (one) represents the system's air entrance and points 3 (three) and 5 (five) are airway outs with silencing filters. In the system's entrance there is a directional valve with three controlled positions. This valve allows the fluids direction control so it is possible to move back or forward the cylinder. Due to the fact that it's a three-position valve, the third position allows the system to maintain the pressure with no need to maintain the compressor's pressure.

The sub-system 8 (eight) is a pressure regulator valve Festo, capable of adjusting the maximum pressure of the feeding line of the cylinder. That way, the maximum normal load that the cylinder can do is determined by this valve.

The sub-systems 9 (nine) and 12 (twelve) are flux controller valves Festo. These valves control the feeding flux of the cylinder. Their function is to act like timers. That way, as time goes by, the quantity of air in the cylinder increases, and so does the pressure. This system can be used to apply a ramp variation of the normal load.

Using the sub-systems 8 (eight) and 9 (nine) is possible to control the variation of the load made over the sample. This control limits the ramp's impulses. To obtain a better control of the real pressure on the system a Baumer Electric membrane pressure sensor is used. This sensor gives instantaneous information of the pneumatic line pressure.

6.2.3. Tangential load checking mechanism

The tangential load checking mechanism is, as seen above, of fundamental importance to determine the critical load. The used system should have precision capable of checking the tangential load and still guarantee perpendicularity of the incisor and the sample.

These characteristics are obtained using a load cell designed to this measurement. This load cell, when submitted to normal load and relative displacement between the sample and the incisor, suffers deformation, as Fig. (8) shows, however the incisor doesn't suffer perpendicularity variations with the sample.

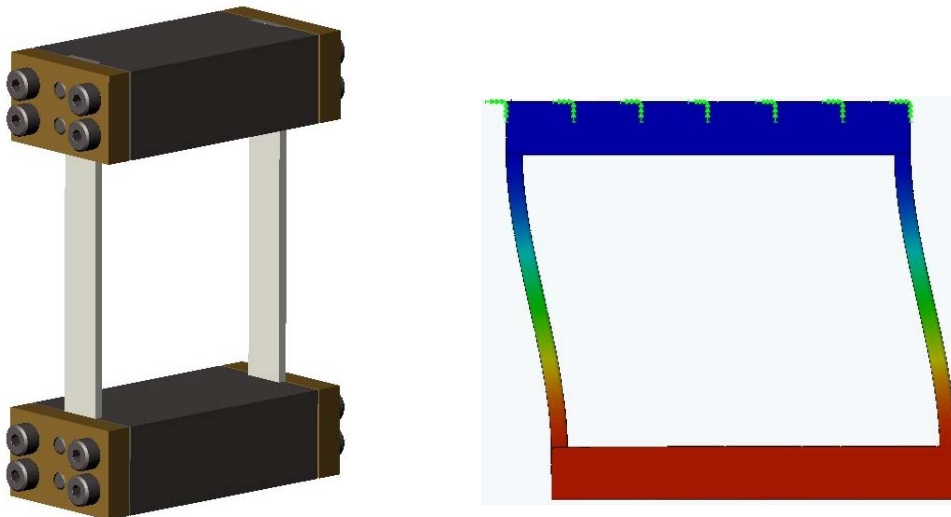


Figure 8. Load cell graphic (load cell and displacement diagram)

The load cell is dimensioned to obtain a better measurement precision in the utilization range (0 – 120 N), and resisting to buckling. The resistance calculus were made using Crandall (1978).

6.2.4. Sample positioning mechanism

The sample positioning mechanism have manual adjust to accommodate 50 x 50 mm samples with a large range of thickness.

7. Proceedings to perform test

The scratch tester should be calibrated according to standard calibration. This calibration includes applied load (Pressure sensor), horizontal displacement and stage planarity.

The Stylus should be check with a magnification of 200x or greater to ensure that there is no visible chipping, cracking or wear.

Samples should be clean to remove dust and grease, both can have some influence on the adhesion data. To maximize the number of scratches that can be done with one sample, it should be placed with the larger dimension perpendicular to the incisor. Scratch should be made with at least 2 mm from the edge of the sample and 1 mm between scratches.

The test should have a start load of 5 N and depending on the kind of test (Multiple scratch at constant load or single scratch with ramp load) the end load can be set. The horizontal displacement rate can be set according to the sample dimension, material or standard procedure.

Multiple tests have to be performed in order to validate de test. Between tests, the diamond tip has to be clean in order to obtain repeatable data.

Temperature and relative humidity of the test environment should be recorded.

8. Conclusion

Scratch test machines are a powerful technique for monitoring coating adhesion, nevertheless a careful interpretation of the results and meticulous performed test are necessities to reach reliable conclusion about the coating-substrate adhesion.

This machine described above shown in Fig. (9) proves that is possible with a low-cost budget, construct a machine able to work under the same conditions of commercial machines. It can provide normal load up to 250 N. The feeding rate can be set to 10 mm/mim (usual feeding rate). Tangential force measurement is made with strain gage connected to Labview interface.

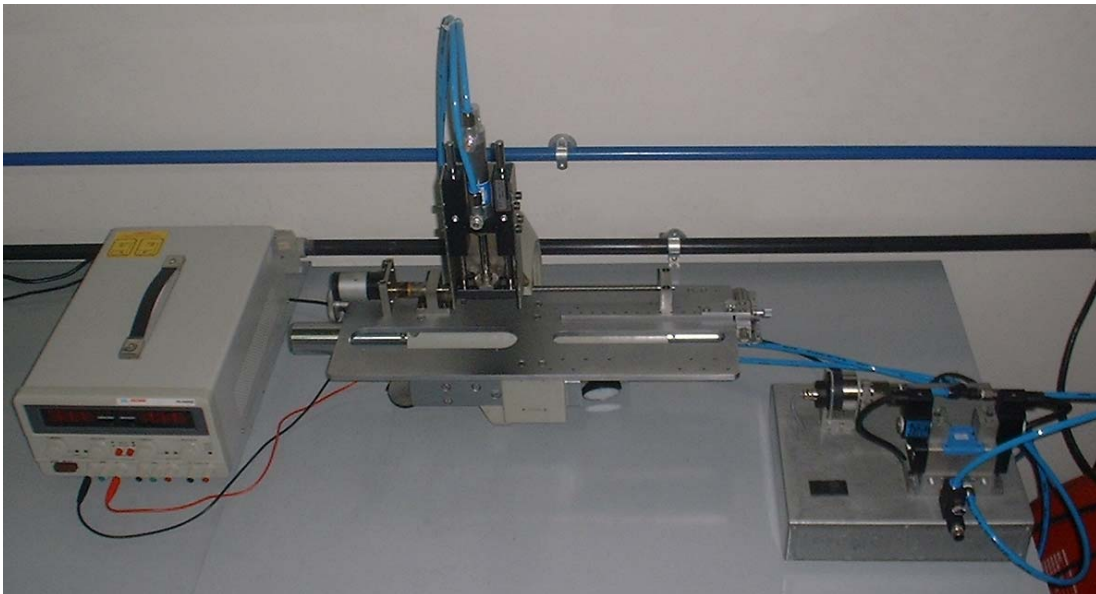


Figure 9. Machine Photograph

The machine has an electronic digital indicator that provides the displacement of the table. Other devices like AE signal can be easily adapted to it.

An example of a test made by this machine is show in Fig. (10).

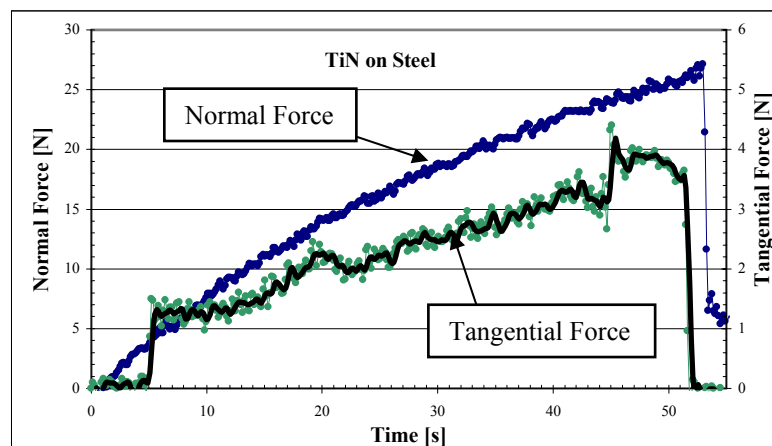


Figure 10. Example of TiN coating on steel Tangential force measurement.

By the construction of this scratch test machine, the laboratory has gained a new research line on coating.

9. Acknowledgement

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10. References

- Arai, T., Fujita, H., Watanabe, M., 1987, "Evaluation of adhesion strength of thin hard coatings", *Thin solid films*, Vol. 154, pp. 387-401.
- Bellido-González, V., Stefanopoulos, N., Deguilhen, F., 1995, "Friction monitored scratch adhesion testing", *Surface & Coating Technology*, Vol.74-75, pp.884-889.
- Bull, S. J., 1997, "Failure mode maps in thin film scratch adhesion test", *Tribology international*, Vol. 30, pp. 491-498.
- Bull, S. J., 1999, "Can scratch testing be used as a model for the abrasive wear of hard coatings?", *Wear*, Vol. 233-235, pp. 412-423.
- Bull, S. J., Rickerby, D. S., Matthews, A., Leyland, A., Pace, A. R., Valli, J., 1988, "The use of scratch adhesion testing for the determination of interfacial adhesion: the importance of frictional drag", *Surface and Coating technology*, Vol. 36, pp. 503-517.
- Burnett, P. J., Rickerby, D. S., 1987, "The relationship between hardness and scratch adhesion", *Thin solid film*, Vol. 154, pp. 403-416.
- Catalogo NSK , 1995, "Prompt delivery series", Cat. No. E3152A.
- Crandall, S. H., Dahl, N. C., Lardner, T. J., 1978, "An Introduction to the Mechanics of Solids", Ed. McGraw-Hill, Singapore.
- Hogmark, S., Jacobson, S., Larsson, M., 2000, "Design and evaluation of tribological coatings", *Wear*, Vol. 246, pp. 20-33.
- Perry, A. J., 1983, "Scratch adhesion testing of hard coatings", *Thin Solid films*, Vol. 107, pp. 167-180.
- Podgornik, P., Vizintin, J., 2003, "Tribology of thin films and their use in the field of machine elements", *Vacuum*, Vol. 68, pp. 39-47.
- Rickerby, D. S., 1988, "A review of the method for the measurement of coating-substrate adhesion", *Surface and coating technology*, Vol. 36, pp. 541-557.
- Sekler, J., Steinmann, P. A., Hintermann, H. E., 1988, "The Scratch Test: Different Critical Load Determination Techniques", *Surface & Coating Technology*, Vol. 36, pp.519-529.
- Soares, L. P. 1997, "Design of a scratching test machine", Final project for mechanical engineering course, Federal University of Rio de Janeiro, Mechanical engineering department.