Thin Film Adhesion Measuring of Conductive Silicon Rubber Depending on Power and Coating Time in Magnetic Sputtering Method

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***Abstract*- Coating silicon rubber with another material is not facile task due to the fact, that this substance exhibits very poor adhesiveness coming from the low surface tension. Adhesion properties can be improved by means of addition filler and plasma treatment. The analyzed sample is a conductive silicon rubber, which was coated using magnetic sputtering method. This method bases on plasma activity in strong magnetic fields. The adhesion in modificatied material was evaluated on the basis of cross-cut test by different coating time and target power.**

I. INTRODUCTION

Silicon rubbers have outstanding properties like excellent elasticity, temperature-resistant, hydrophobia which are in great important in high insulation voltage technique. But silicon rubber can also presents very seriously hurdle in another possible electrical application due to low surface tension, which aggravates coating silicon rubber with metal. In order to increase adhesiveness incorporation of carbon black and filler can be applied [1].This sample consists of silicon rubber and carbon black, which after mixing were manufactured using injection moulding processes. This material unfortunately is not appropriate for much utilization e.g. electrodes, high voltage plugs because of insufficient electrical contact. In order to overcome this problem reliable coating film is necessary, which due to above mentioned reason can be achieve only by means advances coating engineering. Thin film technology, which was used, is called magnetic sputtering. This method is in generally physical vapour deposition process. In magnetic sputtering magnetic field is superposed on the cathode and glow discharge, which is parallel to the cathode surface. The electrons in the glow discharge show cycloidal motion, and the centre of the orbit drifts in the direction of $E \times B$ with the drift velocity of E/B, where E and B denote the electric field in the discharge and the superposed transverse magnetic field, respectively [2].The construction of magnetron(magnetic) sputtering system is shown in Fig.1.

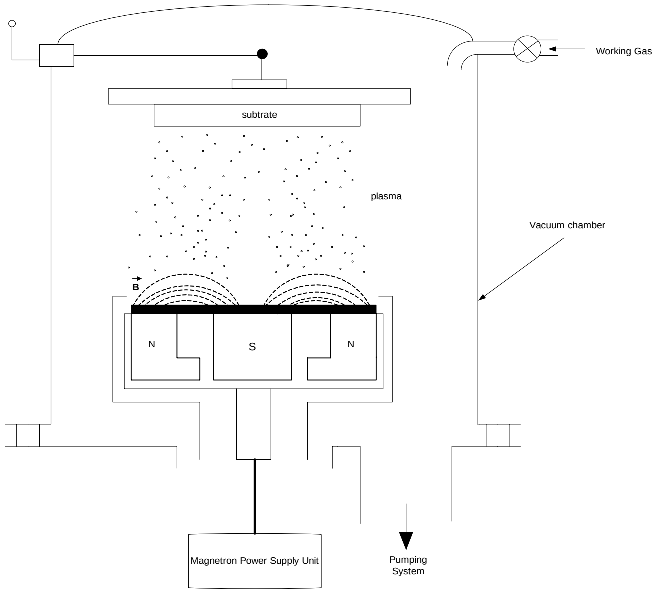

Figure1. Magnetron sputtering system

Magnetic Sputtering Method differs from other sputtering techniques in that the majority of the plasma is confined to region near the target surface by using strong magnetic fields to bend the trajectories of the secondary electrons. Advantages, which conclude: increased deposition rates, reduced substrate heating during deposition, reduced working gas pressure requirements had to taken into consideration by choosing this surface coatings method [3]. Concerning this method there are many parameters, which have an effect on final coating [4,5]. The aim of this research was to draw a conclusion about dependency between target power, coating time and adhesiveness. In terms of power at first we have to distinguish between target power and circling power, whose are connected with very important element concerning magnetron sputtering method - power supply unit. In our experiment magnetron was powered with DPS (Dora Power System).This device use a sinusoidal energy modulation, what enable an achieving great efficiency and power.

What distinguish this system from the conventional power supply unit for sputtering application is in spite of mentioned sinusoidal energy modulation also presence so called circling power P_c (see Figure 2) Supply circuit consist of among others rectifier block, impulse generator and in series resonating circuit LC. The system of quality stabilization Q transfers a part of energy back to the capacitor of main supply system, which as a result appears circling power P_c. If quality Q is differ from unity than on capacitor C resonating circuit accumulate surplus energy, which is transferred subsequently through quality limiter back to power supply unit. That means that impedance plasma-target is a series silencer resonating circuit.

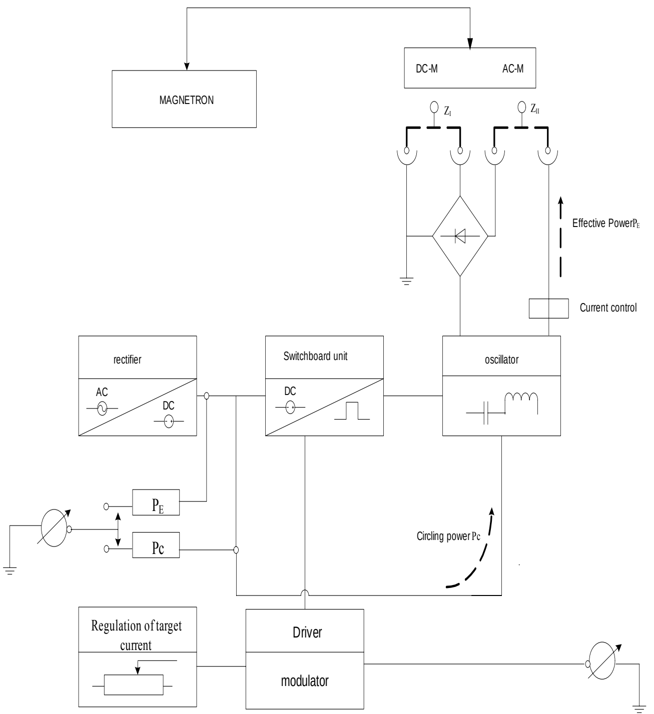

Figure2. Dora Power System

II. EXPERIMENTAL DETAILS

Conductive silicon rubbers were supplied by GE Bayer Silicones. Typical properties of the sample (without coating) are showed in Tab1. In order to achieve meaningful results 9 samples have been coated, where 4 have been coated with titan and 5 with aluminium.

TABLE I
PROPERTIES OF SILICON RUBBER

external diameter		mm	**Ø** 152
internal diameter		mm	**Ø** 37
thickness		mm	25,4
Density	DIN 53 479	g/cm^3	1,08
Shore A hardness	DIN 53 505		40
Tensile strength	DIN 53 504 S2	N/mm^2	7,0
Elongation at break	DIN 53 504 S2	%	650
Tear strength	AST D 624	N/mm	40

In our experiment we have used two methods: scratch test and cross-cut test

A. Scratch test

The first method, which was used in order to analyze coating, was scratch test. Scratch test measurement is commonly used in terms of adhesion evaluation according to scratching procedure presented in figure 3.

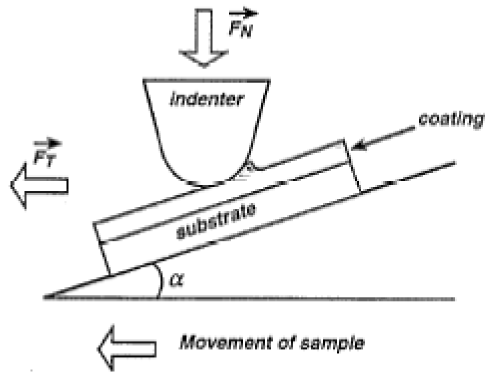

Figure3. Principle of scratch-testing: after contact of diamond indenter on the surface of the coating, the inclined sample is moved with constant speed. The intender remains fixed and produced an increasing load on the inclined sample [4]

This method as surmised was not appropriate for our goal due to influence of substrate material. The influence of the substrate material means that adhesion test cannot take place on silicon rubber because the load goes beyond the carrying capacity of the soft substrate. On another words the coating

cannot withstand specific for scratch test condition, if the substrate is not hard enough.
Figure 4 and 5 show the failure of this method- impossibility of determination critical load for delaminating.

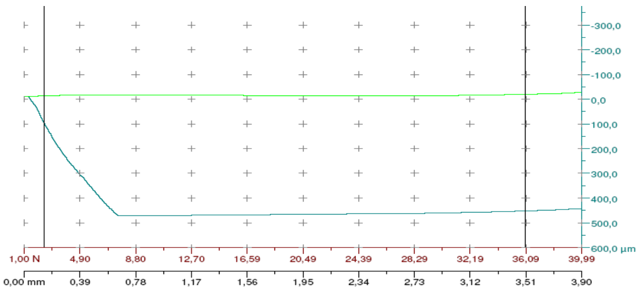

Figure4. Scratch test faulty results

Penetration depth

Residual depth

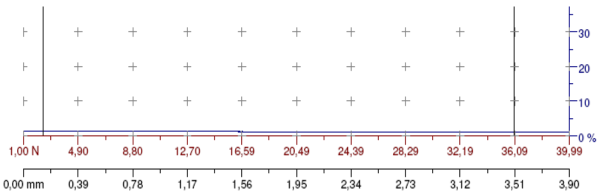

Figure5. Scratch test faulty results in terms of acoustic emission

Acoustic emission

B. Cross-cut test

The second adhesion test namely cross-cut test was carried out according to the norm DIN EN ISO 2409. The test consists in making a series of parallel cuts through the film in one direction and a second series at right angles to the first. I make six cuts 2 mm apart in each direction, forming 25 squares (Fig.6 and 7). Cutting distance was chosen adequate to above mentioned norm for soft material with layer thickness till 60 µm.

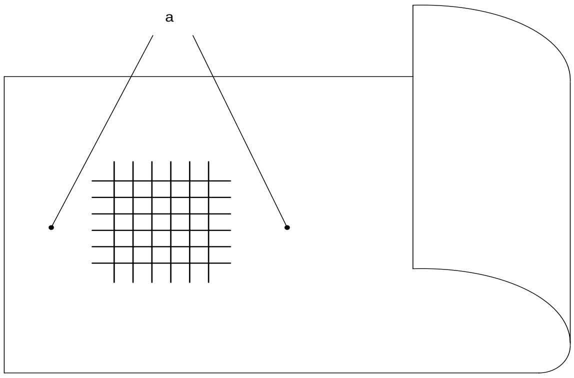

Figure6. Location adhesive tape for cross-cut surface [5]

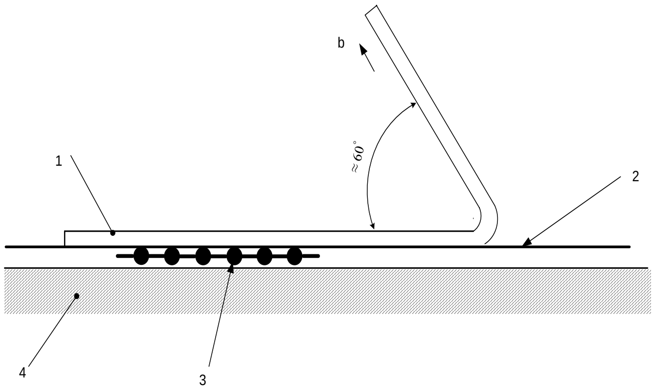

Figure7. Location of cross-cut surface straight before peel off [5]

Legend
1 adhesive tape
2 coating
3 cuts
4 substrate

a smoothed
b peel off direction

III.RESULTS AND DISCUSSION

As mentioned above scratch test method turned out an inappropriate method for such king of coated material. The result of the second method has been juxtaposed in Table 2.

TABLE 2

ADHESION EVLUATION

Lp.	coating material	target power	time	pressure	adhesion evaluation according to DIN EN ISO 2409
		kW	min	Tr	-
1	titanium	2,2	5	$9,8*10^{-3}$	0
2	titanium	3,1	5	$9,8*10^{-3}$	0
3	titanium	4,3	6	$9,8*10^{-3}$	0
4	titanium	5,7	6	$9,8*10^{-3}$	0
5	aluminium	2,0	2	$9,8*10^{-3}$	0
6	aluminium	2,0	2	$9,8*10^{-3}$	0
7	aluminium	2,0	5	$9,8*10^{-3}$	0
8	aluminium	2,0	10	$9,8*10^{-3}$	0
9	aluminium	3,1	5	$9,8*10^{-3}$	0

The evaluation oscillates between 0 till 5, where 5 is the indicator for the poorest adhesion.

The results pointed clearly out that coating shows excellent adhesion regardless of target power and coating time and metal coatings. Figure 6 shows the sample after test with any indication of peeling off.

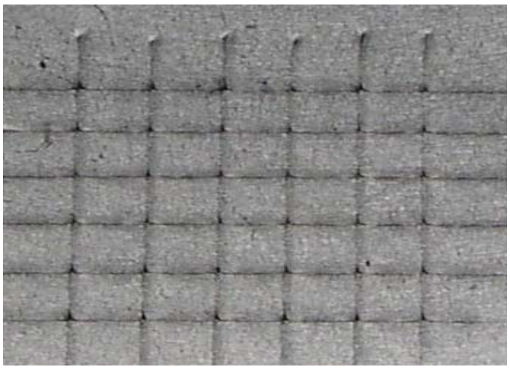

Figure8. View of the sample after cross-cut test

Unfortunately it is very difficult to evaluation adhesion by means another method due to properties of aluminium. It is very difficult to solder aluminium, what impede other adhesive test methods.

The adhesion of a film to the substrate is strongly dependent on the chemical nature, cleanliness, and microscopic topography substrate surface. The adhesion is better for high values of kinetic energy of incident species, absorption energy of the deposit, and initial nucleation density. Effective power P_E is proportional to coating velocity [6], which indicates that with power increase will also coating thickness rise. In our experiment the maximal value of target power was limited to 5,7 kW due to considerably sample temperature increase, which in turn could cause undesirable side effects. The explanation for outstanding adhesion is twofold. First reason is that general magnetron sputtering technique guarantee for very good adhesion. The second one is due to the fact that our parameters were determinated maybe in too narrow range, which demand further research.

IV. CONCLUSION

Film adhesion measuring of conductive silicon rubber doesn't indicate any significant difference in terms of target power and coating time. Magnetron sputtering coating technique turned out very good tool in terms of reliable adhesion. Our experiments show that in spite of poor adhesion properties of silicon rubber achievement of good coatings are possible. Unfortunately other properties of this material namely low harness make some testing method unfeasible.

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