Systematic evaluation of thin electrically insulating layers on common engineering materials

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Abstract:

Thin insulating layers are often required for typical engineering materials, e.g. steel, alumina, titanium and the respective alloys. A very dense insulating layer for subsequent conducting layers acting as electrical paths is crucial. Experiments have shown, however, that the electrical insulation of such substrates is often not sufficient or fails completely and is seldom repeatable.

To determine the most important influences regarding the insulation, a systematic evaluation based on a screening design of experiments for the variation of ten parameters is introduced. The films were deposited using a radiofrequency magnetron sputter system and a non-reactive process as well as a SiO_2 target for the deposition of the insulation.

A thin layer of a Cu-Ni-alloy was successively deposited through a mask on top of the insulating layer. The resistance between the substrate material and the conducting layer was investigated to determine the major influencing parameters with respect to the quality of the insulation layer.

Based on the results, further experiments on film thickness variations with less parameters were carried out and Al_2O_3 layers were compared to those utilising SiO_2.

Keywords: thin-film insulation, sputtered insulation layer, steel substrate, titanium substrate

1. Introduction

In today’s semiconductor technology thin electrical insulation layers are commonly used and are essential for its functionality. Typical materials for insulating layers are SiO_2 and Al_2O_3 respectively [1]. They have, therefore, been widely investigated when applied to Si-wafers serving as the substrate material.

Nevertheless, there is a variety of applications, where the use of thin insulating layers is needed and typical engineering materials, e.g. steel, alumina, titanium and their alloys, have to be used. Even more as certain materials are given by fixed standards, as this is often the case especially in the field of metrology, the National Metrology Institute’s main task.

As an example, sensors comprising thin conducting films are applied directly onto the component to be measured and microstructured by distinct photolithographic steps. Results have proven very promising for precision measurement technology in the future [2, 3]. Besides the possibility of sensor fabrication on a large scale, achieving more precise sensors with a higher spatial resolution and accuracy than conventional sensors is of foremost interest. To achieve a low impact on the measurement, insulating layers as thin as possible are to be found.

However, experiments have shown electrical insulation layers being not sufficient or failing completely when applied to common engineering materials. Therefore, we analysed thin SiO_2 layers thoroughly on titanium alloy Ti6Al4V and 1.3505 steel (100Cr6) substrates, utilising different surface roughnesses.

2. Electrically insulating layers – deposition and quality determination

The insulating layers presented in this paper are being deposited by a magnetron sputtering system (*Von Ardenne – LS320S*) using a non-reactive sputtering process. The system is equipped with two 13.56 MHz generators connected to the target and the substrate which allows the deposition of insulating materials.

Layer thickness is determined by spectroscopic reflectometry *(Micropack NanoCalc-VIS)* which is connected to a microscope for a small spot size and is capable of measuring layers even on rough surfaces. Additionally, the substrate surface and the layer quality were examined using SEM.

The samples used are made of Ti6Al4V and 1.3505 steel (100Cr6) and have a diameter of 30 mm, allowing the sputtering of three samples in one batch. The surface facing towards the target is either turned (Ra < 0,7 µm) or polished. Polishing was done with a *“Struers LaboPol”*, achieving a surface roughness of Ra < 15 nm by using a colloidal silica suspension in the last polishing step.

The most important factor is the determination of the electrical insulation capability of the layer to the substrate itself. Electrical insulation is normally tested by a voltage ramp until a disruptive discharge occurs. This method is not applicable for these layers as, firstly, this would destroy the layer and secondly, the normal operating voltage for the conducting layers on top of the insulating layer (e.g. thin-film sensors) is below 10 V. Hence, the insulating capabilities are tested by measuring the resistance to the substrate.

Experiments have shown that a simple contact of the layer with a conventional measuring tip is not sufficient as small defects in the layer will not be recognised and only a very small spot is inspected. Another possible solution is to contact the layer's surface with a liquid electrolyte. Yet the best and most demanding way to test the insulation is to deposit a conducting layer on top of the insulation and, therefore, have a test scenario similar to the intended use of the layer. If there are pin-holes in the insulation layer, they are most likely to be detected by using this method.

At first, the thin insulating layer of SiO_2 is sputtered, followed by a layer thickness measurement. Next, a 150 nm

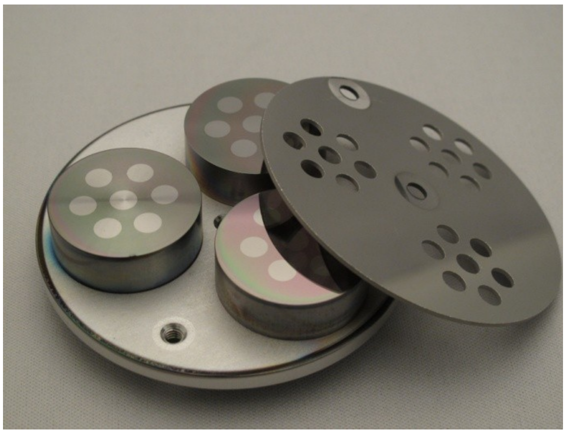

Fig. 1: Samples with spot mask for conducting layer

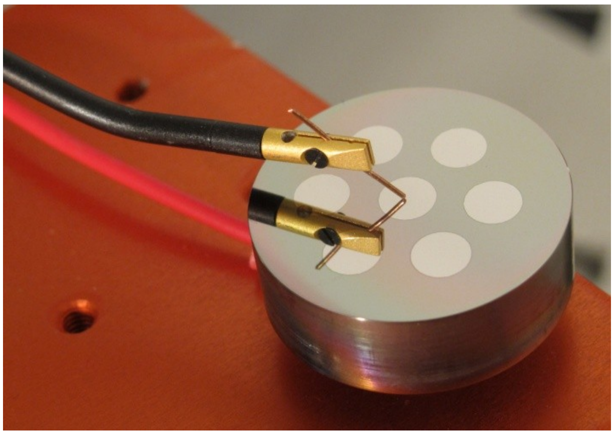

Fig. 2: Probe head contacting one spot

layer of CuNi is sputtered through a mask, to obtain seven spots with a diameter of 5 mm each, evenly distributed over the sample's surface (compare fig.1). The spot size and their positions ensure contrastable results with an even layer thickness distribution (differences below 10 %). For the resistance measurement, one terminal of the multimeter is connected to the uncoated reverse side of the sample, the other one to a probe head with its small tip located on one of the conducting spots. With this setup the contact force is comparable for all measurements (see fig. 2). To evaluate the single resistance value of the spots and to improve comparability, a point rating system was introduced which is shown in tab. 1.

Subsequently, the single values were added which results in the insulating capability of the whole sample, weighted between 0 (best) and 21 (worst).

Resistance value	Rating point
< 1000 Ω	3
< 1 MΩ	2
< 100 MΩ	1
> 100 MΩ	0

Tab. 1: Point rating system

3. Design of experiment

Prior to the start of the investigations the influencing factors on the system consisting of substrate material and preparation, surface, layer material and deposition were analysed. Out of the vast number of factors, the ten most interesting ones were chosen for further investigation.

By variation of only one factor at a time, the standard experiment design, 2^{10} = 1024 test runs are required. Therefore, a proper design needs to be selected. As the determination of the most influencing factor on the insulation is the main goal at first, a screening design of experiment (DoE) was chosen. With this design, one can reduce the amount of experiments to a total of 16: every factor has one low and one high level setting only and multiple factors are changed at the same time [4, 5]. The results need to be interpreted by using statistical evaluation methods afterwards. It has to be remarked that nonlinear processes cannot be appraised by this kind of experimental setup.

Tab. 2 lists the chosen influencing factors with their corresponding low and high level setting and a short description of the same. These are the values used for the screening experiment and, hence, for the results given below. One setting of each factor is the standard value (marked with a star) already used prior to these investigations, the other one is, therefore, the modified value.

Factor	Level low / high		Description
Material	Ti6Al4V	1.3505	Titanium alloy or steel as substrate
Surface	Turned	Polished *	Turned surface Ra < 0,7 µm Polished surface Ra < 15 nm
Preheating	No *	Yes	Heating was done under vacuum at 150 °C
Target distance	40 mm *	60 mm	Distance between sputter target and substrate
Sputter time	2 h	4 h *	Total time of sputter process
Interruptions	0	2 *	Number of interruptions (1 h each) of the sputter process
Sputter power	150 W	200 W *	Supplied power to target
Sputter pressure	8e-3 mbar	10e-3 mbar *	Sputter gas pressure during the sputter process
Oxygen supply	0 sccm *	3 sccm	Inlet of supplementary oxygen
Bias voltage	0 V *	50 V	Bias voltage on substrate

Tab. 2: Influence factors and the corresponding low and high level settings

4. Systematic evaluation – results and discussion

For validation of the DoE and the statistical methods, the influencing factors on the sputtering rate and, thus, on the layer thickness were analysed as these are generally known and, therefore, comparable. The values of the samples' layer thicknesses of the according influence factor level are totalled and averaged:

$$d_{avg,x}=\sum_{i=1}^{16} d_{x,i}$$

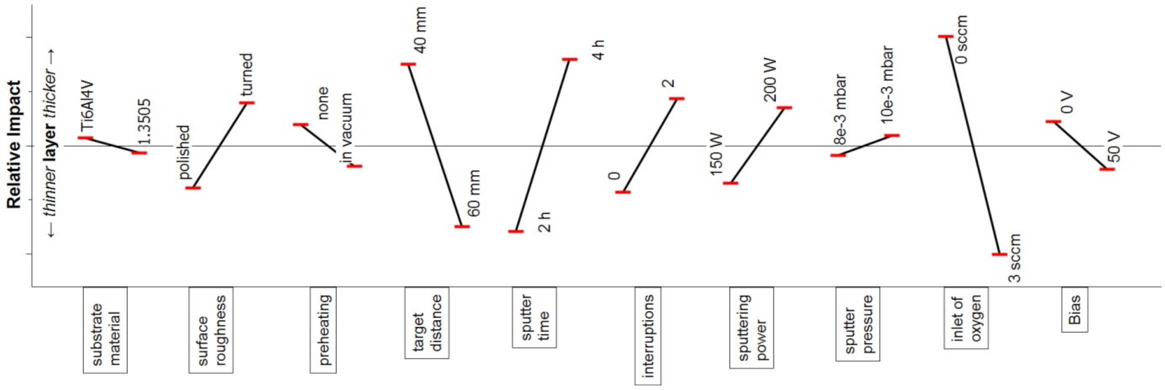

Figure 3: Relative impact of each factor on layer thickness

The highest influence can be determined by the biggest difference between the low and high level setting of each factor, the relative impact, as displayed in the diagram in fig. 3.

Factor number 9 – supplementary oxygen – has the biggest influence, obtaining a thinner layer with oxygen influx. This is followed by the sputter time and the substrate to target distance. All three values are reasonable, not only in magnitude, but also in orientation, and are a validation for the statistical method.

Using the point rating system described above (compare tab. 1), the same calculations were being performed for the insulation values of the samples and drawn into the diagram depicted in fig. 4.

The major influence on the quality of insulation is the substrate material. Insulation of Ti6Al4V substrates was considerably better than those utilising 1.3505. Another significant influence factor is the bias voltage, with an improved insulation if turned off.

The very small difference in insulation with respect to surface roughness modification, however, is misleading. Ti6Al4V samples with a polished surface have considerably better insulation values than their turned counterparts, whereas the turned samples of 1.3505 are better than the polished ones. Therefore, the results relating to the different surface roughnesses interfere with each other, which cannot be resolved by the screening experiment.

For the identification of the main influence on the layer thickness and on the quality of insulation, the chosen design of experiment has proved to be suitable. The insulating layer is influenced considerably by the substrate material and its pre-treatment (i.e. surface roughness) and less by variation of the sputter parameter. Nevertheless, the results reveal that further investigations are necessary with respect to surface roughness and layer thickness. If there is a direct dependency, thicker layers should have a better insulation capability, especially on rough surfaces and also in the case of steel substrates. However, for the application in sensor systems, thick layers have a negative impact on the accuracy of measurement.

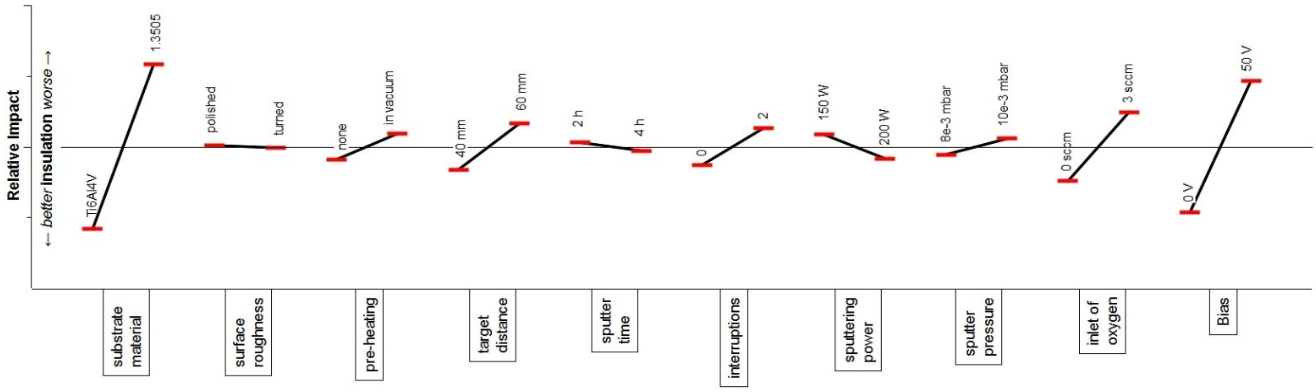

Figure 4: Relative impact of each factor on electrical insulation

5. Follow-up investigations

For the following investigations, the sputter parameters were initially changed to achieve a higher sputtering rate. The standard settings as shown in tab. 2 were altered to a sputtering power of 300 W, a pressure of 8e-3 mbar, no oxygen and 0 V bias voltage. As a result, the rate increased from 1,5 µm/h to 2,2 µm/h, whilst the quality of insulation did not change.

For the investigation of different surface roughnesses and layer thicknesses and their influence on the insulation, a variation of the sputtering time and, therefore, the layer thickness was carried out. Every batch consists of three samples: Ti6Al4V (polished), 1.3505 (turned and polished). As shown in fig. 5 (left) the layer thickness was varied between 600 nm and 3500 nm in six steps.

The samples made of Ti6Al4V show good insulation values throughout the experiment and, thus, a layer thickness of about 600 nm has found to be sufficient. For the 1.3505 samples with a turned surface the insulating capability is not sufficient at first but reaches the level of the titanium alloy at about 2000 nm thickness. The polished samples

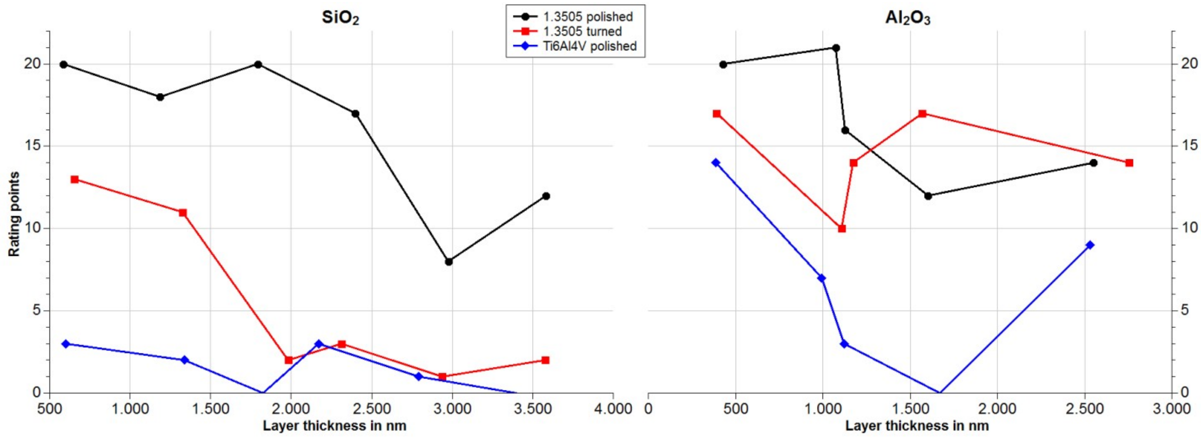

Figure 5: Insulation rating against layer thickness of SiO_2 (left) and Al_2O_3 (right)

have bad results for thin layers, but improve with layers thicker than 2000 nm. Still, these samples only reach a fair level of insulation.

Electrical insulation utilising thin SiO_2 layers is, as a rule, better for Ti6Al4V than for 1.3505 steel. As shown, the surface does have a major influence on the insulation, too, but cannot be described by surface roughness alone. Instead, we have found it to be caused by the composition of surface structure of the material itself.

For the evaluation of the comparability to other insulating materials, a contrastable experiment using an Al_2O_3 target instead of SiO_2 was carried out. Besides a higher sputtering power of 400 W, other parameters remained the same. We were able to achieve a sputtering rate of up to 1,6 µm/h. The layer thickness was varied in five steps from 400 nm to 2700 nm. The results of the insulation rating plotted against the layer thickness are shown in figure 5 (right).

Similar to the results with SiO_2 thin-films, it seems that there is a dependency between the layer thickness and the insulating capability, recognisable for the polished Ti6Al4V sample. However, this is not clearly visible for the 1.3505 samples, sticking to a poor level of insulation, regardless of layer thickness and surface roughness in the investigation range. Further experiments are necessary to ensure the results with Al_2O_3 and for the evaluation of thicker layers.

6. Conclusions and Outlook

Prior investigations revealed that the electrical insulation of common engineering materials by thin-films was scarcely reproducible. Therefore, thorough research on sputtered insulation layers, especially on SiO_2 was carried out and presented.

From influencing factors on the substrate-layer system, ten of the most important ones were selected. A screening design of experiment was introduced to reduce test runs and for the identification of the main influence factors by means of systematic evaluation.

These experiments showed that the major influence on the quality of insulation is given by the substrate material and its surface roughness. Thus, further investigations were carried out in terms of layer thickness variation on different substrates with turned and polished surfaces. The results strengthen the outcome of the screening experiment and point to a dependency between insulation quality and film thickness. Furthermore, the insulation does not depend on the surface roughness alone, but also on the surface structure itself.

Finally, the first experiments utilising Al_2O_3 as an insulation layer are presented and compared. Further investigations are needed in terms of insulating capability and to evaluate the applicability and possible advantages or disadvantages of Al_2O_3 against SiO_2.

Layers, thicker than about 3 µm could be investigated for their insulation capabilities in the future. Nevertheless, for the intended use of the layer system, e.g. for high precision sensors, thinner layers are preferred as they potentially have less influence on the measurand and better adhesion.

The use of other sputtering techniques, as in pulsed sputtering systems, with potentially denser thin-films is projected.

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