NEW WAYS OF FABRICATION OF EXPERIMENTAL SAMPLES FOR DIELECTRICAL MEASUREMENTS

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Abstract: It has been proven that most of problems with samples, made for measurement, are encountered during manufacturing process. Samples can be produced by several manufacturing processes and even a subtle abnormality or defect may render such sample useless. These faults are further discovered during measurement and usage. Scientific sample measurement hereby aids in discovery of errors in production stage. Recently published studies discussing moulds made of different materials and its impact on sample manufacturing raised a lot of attention among scientist. The goal was to create samples considerably better than produced nowadays, so they could match specific criteria - most notably nanofiller distribution, sample thickness, dielectrical properties, etc.

Keywords: Sample preparation, Dielectric measurements, Teflon, Metal,

1. INTRODUCTION

The most important part for examining the issue of dielectric spectroscopy is to have quality produced samples to measure. For this purpose there are professional moulds that are used, such as moulds from ABB Company. This company deals with the large-scale production of dielectric materials. The use of the ABB mould, for making epoxy with nanofillers of different types, was found unsuitable. Since the Agilent 4284 LCR bridge could not measure the capacitance of dielectric materials used in the production, a new satisfactory mould for the samples had to be made. On this basis, the research was redirected and focused on the methods of production of samples themselves; so they would comply with the conditions set by the LCR bridge. Various methods with their individual advantages and disadvantages will be explored in this article.

2. PRESENT STATE AND BACKGROUND

Dielectric relaxation spectroscopy (DRS) has been an established tool for the investigation of molecular dynamics of materials for a long time [1]. Developments in instrumentation and in measurement technique allow the study of dielectric properties in wide frequency and temperature ranges (10^{-6} Hz – 10^{15} Hz, 10 K – 1000 K) and for very different materials, ranging from materials for biomedical engineering up to insulations for HV rotating electric engines and power grids [2].

At the present time there are many devices and methods used for the measurement. In this research, the sample was inserted into a parallel-plate electrode system, and that was connected to the LCR bridge, and the data were further processed by the computer. In spite of corrections that have been enabled on the device, the data was reflecting large errors in the samples produced in the original ABB mould, see Fig. 1, and they could not be used. Therefore, the research had to focus to the

sample production process. The original mould satisfied important requirements such as virtually no roughness of the surface material of the mould, high durability and simple disassembly. However, the thickness of samples produced in this original mould was approximately 2 mm. The capacitance of these samples was of the order of 3 pF, which was below the measurement capabilities of the LCR bridge. It was, therefore, important to decrease the sample thickness to as low as possible, to approximately 100 – 500 μm. This was experimentally achieved by grinding original samples off. However, in the grinding process we experienced large material losses, and samples also suffered from susceptibility to cracking.

Figure 1: The original mould from ABB Comp.

Therefore, another method was experimentally examined for the production of samples with similar thickness, as has been achieved with grinding. For the purposes of production, the method which was reported in the laboratory course in Spain [3] was used. It is a method where sample material is placed between two electrodes. To achieve that the space matches the required dimension, a distance strip is then inserted between the electrodes and that is placed on the sample with either of the subsequent two methods. For example, the method of two parallel strips placed between the electrodes; or the method in which there are three distance points placed in the gaps between the electrodes, in the way to avoid connection of the electrodes but at the same time preventing complete displacement of the measured samples. The last method is locating distance strip all around the perimeter of the electrode, and this has led to the following experiment.

3. EXPERIMENT AND RESULTS

The need to experiment in the production of samples for measurement of dielectric spectroscopy was due to the thickness of the samples and their inaccessibility to the measurement of dielectric properties.

3.1. ORIGINAL MOULD

In Fig. 2 we can see a sheet from the original mould, from which a sample is removed in a state where it can be easily pulled from the mould and allowed to bake in oven at 140 °C for 12 hours.

Figure 2: Original mould.

This mould is made of steel, and its walls are perfectly cut to maximum smoothness to prevent sticking of the sample to the mould. To decrease the adhesion, a microfilm of silicone grease is

applied to all walls of moulds to prevent the binding of samples to the mould at the points of slight damage. The disadvantage of this mould is the inability to adjust the thickness of the produced samples and therefore it could not to be used for the production itself.

3.2.   TEFLON WITHOUT THE POSSIBILITY OF AIR LEAK

Based on the good experience with Teflon new moulds of Teflon were made. Precisely lathed parts of mould were used to determine a gap see fig. 3. Problem has arisen during production of the first sample, because the air could not escape. On the sample appeared large clusters of bubbles, and therefore these samples were not suitable for the measurement.

Figure 3: Teflon without the possibility of air leak.

The disadvantage is the inability of the air to leak and this damaging the sample. The advantage is to easily determine the thickness of the sample with a wire.

3.3.   TEFLON AND WIRE

Teflon has very good adhesion properties to material. With the help of the wire as a stabilizing circuit, the use of Teflon allowed a manufacture of small samples. However, the thickness of the sample could not be reduced by varying the thickness of the wire.
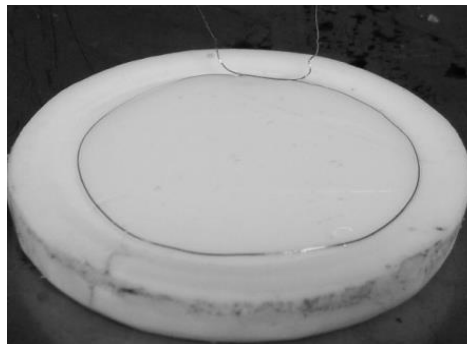
Figure 3: Teflon and wire.

A disadvantage of this method is the impossibility of reducing the thickness of the sample. Even when loading the sample with 300 kg, we achieved a size of 610 μm.

3.4.   TEFLON WITH A TEFLON FOIL WITH THE POSSIBILITY OF AIR LEAK

By examining the previous study we found out that the usage of Teflon will be most suitable for the sample production because of problems with removing the samples from the mould see fig. 5 did not occur. Two thick Teflon moulds are used between which an 180μm thick Teflon foil is placed in a way to create a displacement gap that serves the production of thin samples. With the help of this method samples of a thickness of 360 μm were successfully created. In order to attach the solid Teflon parts to each other it was necessary to use metal clamps.
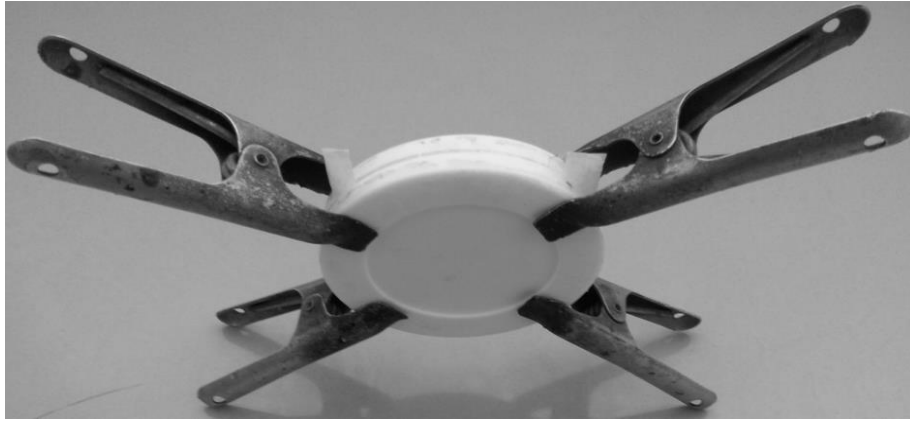
Figure 5: Teflon with a Teflon foil with the possibility of air leak.

The disadvantage of this method is a very thin gap and the mould then has to be heated up so that the fluid can pass through more easily. All the other characteristics of this mould are only positive, such as: the simplicity of disassembly and the production of very thin samples. The method is very suitable for the production of sample for dielectric spectroscopy measurements.

3.5. NEW METALLIC DESIGN

Form with the silicon sides is more than suitable for production of thin high-quality samples. The main disadvantage of this mold is number of samples, which can be created from one casting. The core advantages of sample are thin structure, easy measurement and easily observable dielectric characteristics. It was necessary to design a new larger mold, which could produce a set of samples from one casting, so we could easily measure error rate.

Figure 6: New metallic form with four special parts

This mold has a specially refined shape, as can be seen in the Fig. 6. New shape allows air bubbles, which are created while pouring liquid mixture to the form, to spread easily.

4. CONCLUSION

The samples production out of epoxy resins with nanofillers that serve the purpose for dielectric spectroscopy measurements. The differences between the methods were often inconspicuous as far as the utilized materials are concerned, and in terms of the shape/distance; however, they were of great relevance for the sample production. Teflon of a thickness from 1 to 2 cm, which fulfilled the requirements of a smooth surface and heat stability at the same time, was most suitable for the sample production with regards to the desired characteristics of the material. Metal clamps which would resist the temperature of 140°C were used in order to attach the coarse parts together in a way that no leakage through the mould or the foil would be possible. The method of using new Metallic design as a mould was most successful and was subsequently used for the production of samples for dielectric measurements.

ACKNOWLEDGEMENT

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