METHODS OF PRODUCTION OF SAMPLES FOR DIELECTRIC SPECTROSCOPY

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Abstract: One of the important fields in dielectric spectroscopy is a production of quality samples needed for dielectric measurements. Many methods are currently used for these purposes and they are integrated into the actual measurement. On the basis of an experiment I have explored these methods and found out their advantages and disadvantages. Materials used to manufacture moulds for production of epoxy sample with nanofillers had to satisfy these conditions: high temperature resistance, low surface friction and ductility. On the basis of these requirements, various materials such as metal, glass, Teflon and different types of foils were studied. In the conclusion, the most suitable method for production of samples for dielectric spectroscopy measurement was chosen.

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

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.

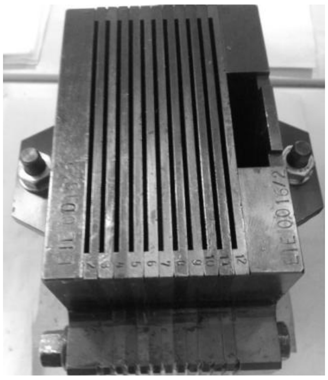

Figure 1: The original mould from ABB Comp.

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. GLASS AND WIRE

In Fig. 3 we can see three glasses between which two wires are inserted in the shape of a circle, and at the top they are open for air venting when being filled with epoxy from the top. The thickness of the wire determines the thickness of the sample, and wire with a thickness of 510 μm was used for this production. Metal clips were used to secure the glass together to prevent leakage of epoxy with nanofillers.

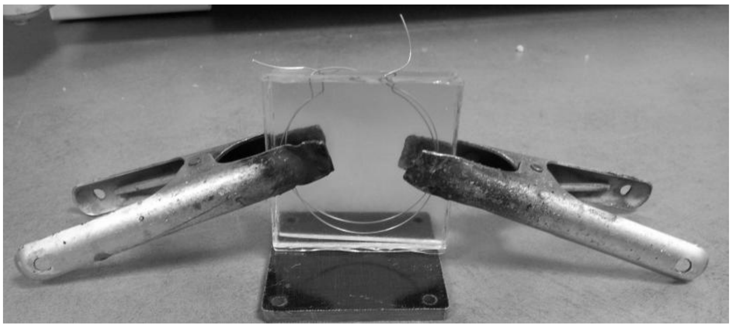

Figure 3: Glass and wire.

The disadvantage of this mould is a problem with the dismantling of it and the collection of the samples because hardening epoxy binds to the glass and creates a very rigid structure. We managed to disassemble the mould by heating it to 100 °C. The problem is then the cracking of the mould caused by uneven heating.

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 4: 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 FOIL ON GLASS AND WIRE

Teflon was proven in anti-adhesion. Therefore an experiment was carried out on a sample consisting of two Teflon foils with a wire inserted between them, in order to fix their distance. So as to achieve constant pressure, the Teflon foil was loaded with a glass pane and an additional weight, see Fig. 5, so as to keep the samples even. However, in spite of it, produced samples still exhibited problems with an uneven distribution of the material that arose when they were heated to 120 °C. This caused curling of the foil and so had an adverse effect on the samples. The advantage was ability to obtain a thickness of samples ranging from 100 – 510 μm.

The disadvantage of this method is the uneven distribution of epoxy in the production of samples and it would cause problems when measuring the LCR bridge.

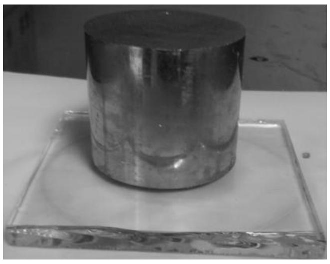

Figure 5: Teflon foil with wire loaded on glass and load.

3.5. 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. 6. 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 6: 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.

3.6. TEFLON AND GLASS WITH A TEFLON FOIL WITH THE POSSIBILITY OF AIR LEAK

The combination of Teflon and glass turned out to be successful. Samples were thin and showed no signs of bubbles. However, a problem arose during the disassembly of the mould, where Teflon parts could not be separated easily. Also, epoxy resin kept sticking to glass again, see Fig. 7.

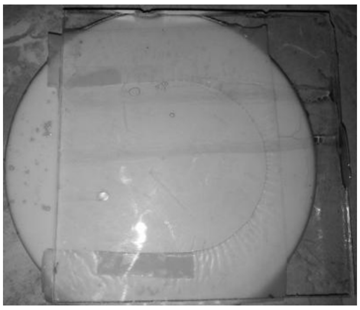

Figure 7: Teflon and glass with a Teflon foil with the possibility of air leak.

The disadvantage of this mould is a problem of samples separation from the glass. Therefore it was necessary to replace the glass with other material.

3.7. 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. 8 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.

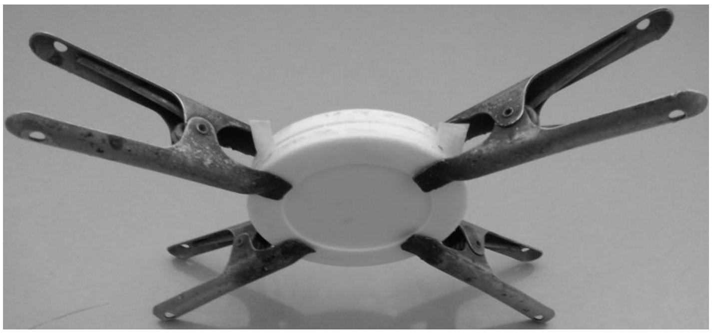

Figure 8: Teflon with a Teflon foil with the possibility of air leak.

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.

4. CONCLUSION

All of the listed methods were used during the sample 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. Both wire and Teflon were suitable as displacement materials but the above mention Teflon foil was used for the Teflon foil thickness of 180 μm. 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 Teflon as a mould was most successful and was subsequently used for the production of samples for dielectric measurements.

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