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Electric field mapping inside metallized film capacitors

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Key Words: failure mode and effect analysis, film capacitors, micro-sectioning, Kelvin probe force microscopy

SUMMARY & CONCLUSIONS

Failure mode and effect analysis (FMEA) is an important step in the reliability assessment process of electric components. It provides knowledge of the physics of failure of a component that has been subjected to a given stress profile. This knowledge enables improvement of the component robustness and durability and serves as verification that failure- and degradation mechanisms remain the same at different stress levels during accelerated testing.

In this work we have used Kelvin probe force microscopy (KPFM) to analyze metallized film capacitors with the purpose of determining the degradation mechanism(s) they suffered from accelerated testing. We have prepared film capacitors for analysis by micro-sectioning and verified the quality of the preparation procedure using optical and atomic force microscopy.

The potential distribution in the layer structure (alternating 7 µm thick dielectric and 50-100 nm thick metal) of a new capacitor was used as reference. KPFM measurements on the degraded capacitors showed a change in contact potential difference from -0.61V on the reference capacitor to 3.2V on the degraded ones, indicating that corrosion of the metallization had happened. Studies also showed that some of the metallization stripes had lost contact to the end-spray.

In conclusion we have shown that the surface electric potential distributions on micro-sectioned film capacitors can be obtained through KPFM analysis. We have, from KPFM measurements, shown that the degraded capacitors under investigation had suffered from metallization corrosion and some degree of end-spray detachment. The results obtained in this work, along with results from the literature, demonstrate the usefulness of KPFM as a tool for FMEA.

1 INTRODUCTION

Capacitors are widely used in power electronic applications and thus their reliability is of great importance. According to Yang et. al. capacitors are, by the industry, regarded as some of the more fragile components in power electronic applications [1]. They are therefore frequently subjected to accelerated testing for robustness evaluation, component selection, and lifetime estimation.

Even though a lot of valuable information can be obtained through accelerated testing, it can sometimes be misinforming. One main purpose of accelerated lifetime testing is to accelerate the degradation/failure type(s) that the given component suffers from during normal use, and extrapolate an estimated lifetime from the stress-lifetime data. Hence, one issue with accelerated lifetime testing is the possible change in failure mode that follows subjecting the component to much higher stresses than it was designed to withstand [2]. This makes failure mode and effect analysis (FMEA) an important step in the process of component lifetime estimation in order to verify the usability of the obtained data.

Failure analysis of capacitors can be challenging, since the active parts are encapsulated. One solution is to measure the standard electric parameters, such as capacitance, equivalent series resistance, and loss factor. These parameters may, however, be difficult to link to a specific failure or degradation mechanism. Hence, the next step is the physical characterization of the materials that the component is composed of.

A technique that has been used to measure electric potential distributions for characterization of electric components is Kelvin probe force microscopy (KPFM). This technique utilizes the electric forces between a metal coated probe and the sample under investigation to map the electric potential over a given surface [3]. It has been used on components, such as solar cells, organic field-effect transistors, silicon pn-junctions, and multi-layer ceramic capacitors [4-7].

Some of the most common degradation types in metallized film capacitors, such as metallization corrosion, end-spray detachment, and partial discharges [8], are detectable by KPFM. Corrosion of the metallization will increase the measured contact potential difference (CPD) at the metallization sites, due to the increased work function of the oxides [9]. If the electrical contact between metallization layers and end-spray is lost, any applied voltage across the capacitor will not be measurable on the detached metallization layers. Finally, if a partial discharge has occurred, the metallization will be cleared at the discharge site, which will be seen as a sudden change in surface potential at the metallization site.

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2 EXPERIMENTAL

The capacitors that were investigated in this work had undergone accelerated testing performed by Danfoss Silicon Power. They were subjected to 85°C, 85% relative humidity, and 230V AC for 541 hours. During that time all capacitors under test had lost 90-99.9% of their original capacitance. An un-stressed capacitor was included in the studies as reference.

2.1 *Micro-sectioning*

The capacitors were prepared for investigation by a standard micro-sectioning procedure [10]. The capacitors were cast into epoxy for structural support and cut using a diamond saw. The cut plane was chosen in order to obtain a surface in a way that a voltage could still be applied across the capacitor through the original connectors. The exposed surface was grinded on a series of sanding pads ranging from grain 240 (coarse) to grain 4000 (extremely fine). The surface was not polished, because initial attempts showed that the polishing agent would lodge itself into the soft dielectric. The resulting quality of the grinding procedure was examined by optical microscopy using a Leica DMI3000M inverted microscope.

After approval of the grinding procedure a 2 mm slice, containing the grinded surface and the connectors, was sawed off from the component in order to fit into the atomic force microscopy (AFM) setup. This procedure caused small displacements in the capacitor layer structure in some of the components. These displacements were, however, small enough to assume that it would not affect the measurements. Finally, wires were soldered onto the connectors enabling application of voltage across the capacitor during KPFM measurement. An image of one of the prepared film capacitors is shown in figure 1.

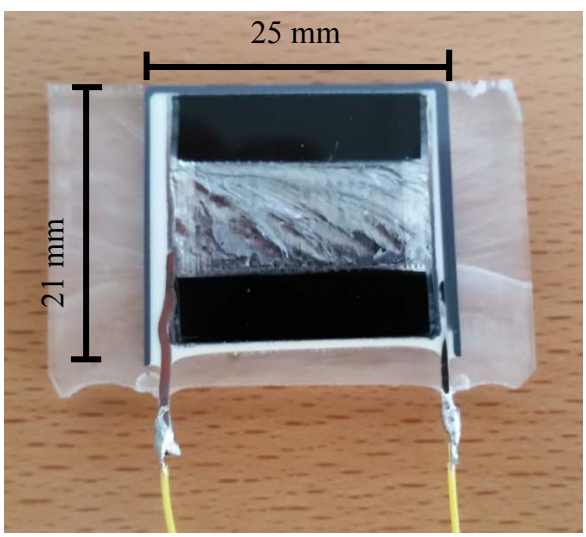

Figure 1: Micro-sectioned film capacitor prepared for KPFM analysis. The yellow wires are soldered onto the connectors.

2.2 *Atomic and Kelvin probe force microscopy*

KPFM is a scan mode of AFM and is used to measure the surface electric potential distribution across a given area. It requires a cantilever with a metal coated tip to which a voltage can be applied. A potential difference arises between the tip and the sample due to the difference in work functions, also known as CPD, between the metal coating on the tip and the metallization layers in the capacitor. In KPFM mode the cantilever is oscillated electrically by applying an AC voltage between the tip and the sample at frequency ω. An additional DC voltage is also applied. This gives rise to a force on the cantilever, which can be divided into three contributions; one static, one at frequency ω, and one at frequency 2ω. The force at frequency ω is given by

$$F_\omega = -\frac{dC}{dz}(V_s - V_{DC})V_{AC}\sin(\omega t), \qquad (1)$$

where C is the capacitance of the tip/sample system. This force can be nullified by adjusting V_{DC} so that it matches the surface potential V_S. This is done in every scan point and thereby a two-dimensional map of the surface potential is obtained [3].

The interpretation of this measured surface potential on metal/dielectric heterostructures, such as film capacitors, is rather complicated and will therefore not be discussed in depth, since it is not the primary scope of this work.

Identifying corrosion of the metallization from KPFM measurements requires knowledge of the work functions of all the involved compounds. In this work the cantilevers are coated with platinum and iridium, which are among the metals with the highest work functions. The metallization layers consist of zinc and aluminum and thereby a corroded metallization layer would consist of zinc oxide and aluminum oxide. The work functions of these compounds are listed in table 1.

Material	Work function (eV)
Platinum	5.65
Iridium	5.3
Zinc	4.24
Aluminum	4.3
Zinc oxide	5.3
Aluminum oxide	7.3

Table 1: List of work functions of the cantilever coating metals, metallization metals and their oxides. [12-15]

Zinc and aluminum have lower work functions than platinum and iridium, meaning that the measured CPD should be negative if measuring on non-corroded metallization layers. The work functions of the oxides are higher than those for zinc and aluminum. Especially that of aluminum oxide is considerably higher than all the metals. Hence, a measured positive CPD would indicate that the metallization layers are corroded.

In AFM mode the surface topography was obtained using an NTEGRA-Aura nanolaboratory (NT-MDT) and standard silicon cantilevers [11]. The topography was mainly used to determine the root-mean-square (RMS) roughness of the grinded surfaces.

For KPFM the same instrument as for AFM was used.

The cantilevers were of the Platinum-Iridium coated NSG01/Pt type from NT-MDT. These cantilevers have a tip curvature radius around 35 nm, which is sufficiently small for this type of investigations. The KPFM measurements were carried out in two-pass mode, meaning that the same line was scanned twice. In the first pass the topography of the sample was recorded and used to maintain a fixed tip/sample distance of 10 nm in the second pass. The magnitude of the applied AC voltage between tip and sample was fixed at 0.3V. The measurements were performed twice on each sample. In the first measurement both leads were connected to the AFM ground, meaning that no voltage was applied across the capacitor. In the second measurement 5V DC was applied across the capacitor by an external power supply, while still connected to the AFM ground. After the measurements the obtained data was treated in WSxM 5.0 [16].

3 RESULTS

3.1 *Quality Assessment of the Micro-Sectioning Procedure*

After the grinding procedure the quality of the grinded surface was inspected by optical microscopy. The requirements were low surface roughness, visible layer structure, and low concentration of grinding tracks. A microscopy image of a grinded capacitor surface is shown in figure 2, where it is seen that the requirements are fulfilled. The layer structure is clearly visible and there is only a few grinding tracks in the surface.

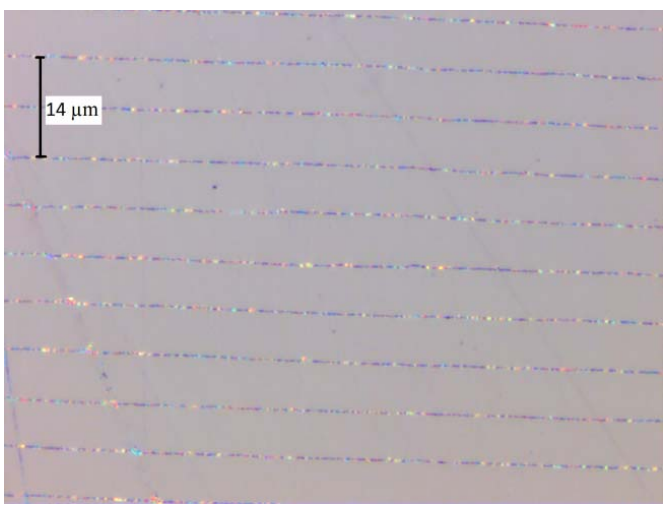

Figure 2: Optical microscope image at 1000X magnification of the micro-sectioned reference capacitor.

The surface roughness was further examined by AFM. From the measurements it was determined that the sample shown in figure 2 had an RMS surface roughness in the range of 20-30 nm, which also demonstrates the quality level of the grinding procedure.

3.2 *KPFM Measurements on New Capacitor*

KPFM measurements were initially performed on the un-stressed reference capacitor with the purpose of determining the CPD at the metallization sites and obtaining a reference surface potential distribution. The results from the measurements without applied voltage and with 5V DC are shown in figure 3. The measured surface potential distribution on the capacitor with no applied voltage reveals a periodic surface potential profile despite the fact that no electric field should be present in the capacitor structure. This effect is associated with KPFM measurements on metal/dielectric heterostructures and arises due to polarization of the dielectric caused by the electric potential applied to the cantilever. The length of the period is around 7 µm, corresponding to the dielectric film thickness as shown in figure 2. The measured surface potential on the biased capacitor also reveals a periodic surface potential distribution. As expected, the period length is 14 µm, since every second metallization layer had 5V applied and those in between were grounded.

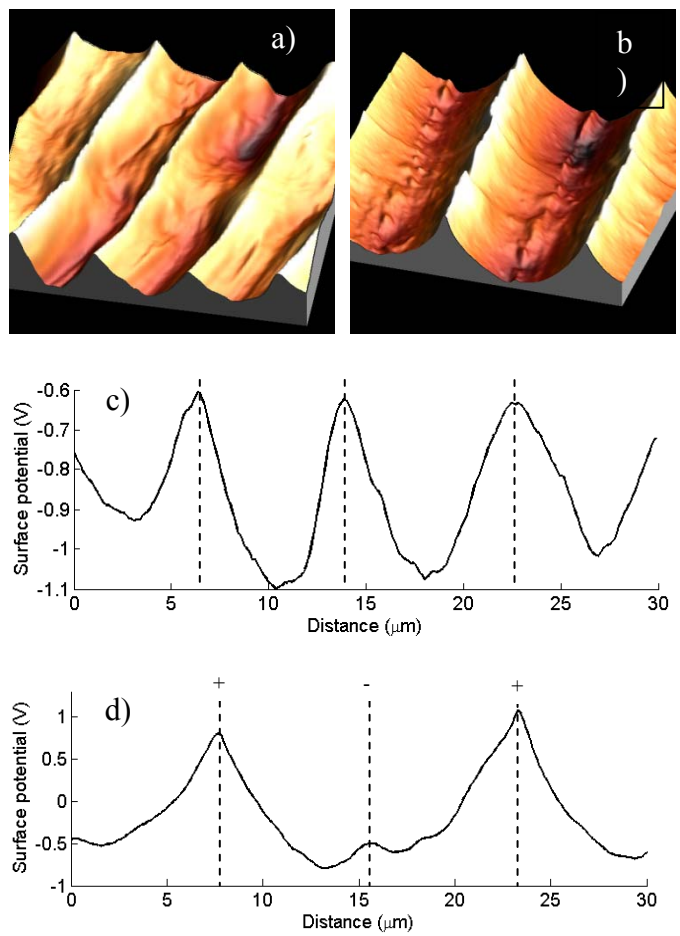

Figure 3: Results from the KPFM measurements on the un-stressed film capacitor. a) Surface potential distribution of the capacitor, while grounded and b) while biased. c)-d) Cross-section profiles obtained from the surface potential measurements presented in panels a) and b), respectively. The dashed lines indicate the position of the metallization. The plus and minus signs indicate whether the metallization layers are connected to the biased or grounded end-spray, respectively.

The measured potential difference between alternating metallization layers does, however, not correspond to the 5V that was applied across the capacitor. There are multiple

possible explanations to this phenomenon, where one is due to the point spread function (PSF) of the cantilever tip. Since electrostatic forces are long range forces, the measurement in each scan point is affected by the surface potentials in the surrounding area. Thus, the measured surface potential does not correspond to the surface potential in the scan point, but rather weighted contributions from the surrounding scan points as well [17-18].

The surface potential on the metallization sites, corresponding to the CPD between the cantilever and metallization, was measured on the grounded and biased capacitor to be -0.61V and -0.50V, respectively. This means that the metallization is not corroded on the reference capacitor. The magnitude of the CPD does not correspond to the expected CPD, which would be around -1V, according to table 1. This could also be explained by the point spread function of the cantilever, but also the fact that the work function varies with crystallinity and crystal direction in the surface [19]. It is also affected by the presence of a thin water adsorbate layer on the surface and possible contaminations from the surrounding environment, since the measurements were carried out in ambient air.

3.3 *KPFM Measurements on Stressed Capacitor*

Measurements were also performed on the degraded capacitors and the results are shown in figure 4. The measurements on the grounded capacitor in figure 4a demonstrate a similarly periodic structure as on the reference capacitor, but with a periodicity of 5 µm corresponding to the dielectric layer thickness in this capacitor.

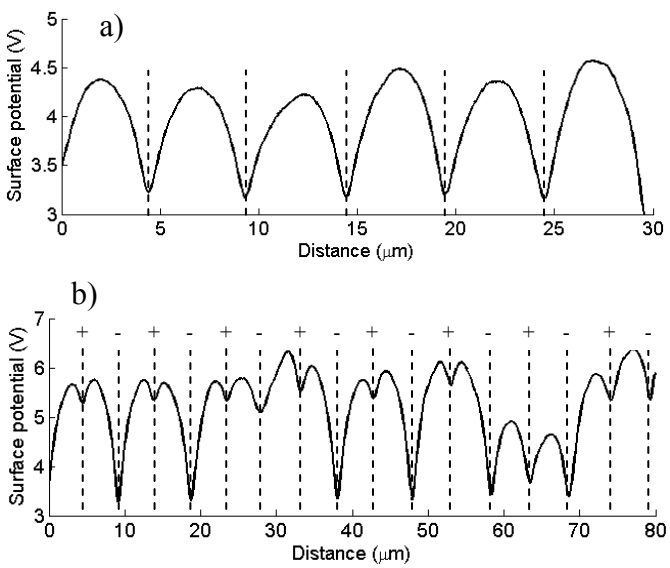

Figure 4: Cross-section profiles of the surface potential distributions a) on the grounded degraded capacitor and b) on the same capacitor with 5V applied across it. The dashed lines indicate the position of the metallization.

It is seen that the magnitude of the CPD at the metallization sites has increased significantly, from -0.6V on the reference capacitor to +3.2V. This is a clear indication that there has been a change in material, and according to table 1 it is caused by corrosion of the metallization layers.

The measurement on the biased capacitor, displayed in figure 4b, shows a similar magnitude level of the CPD at the grounded metallization layers. One can also see that most of the metallization layers have contact to the end-spray. However, the metallization layers at 30 µm, 65 µm, and at 80 µm have lost contact to the end spray, because the measured CPD at these layers are unaffected by the applied potential or ground at the end-spray, which they should be connected to. The metallization layer at 65 µm should be connected to the end-spray with 5V applied, but the measured CPD matches that of the grounded metallization. On the other hand, the CPD measured on the metallization layers at 30 µm and 80 µm corresponds to those measured on the layers with 5V applied, even though they are connected to ground.

The result in figure 4b shows that three out of 16 metallization layers were disconnected from the end-spray. This measurement was performed close to the center of the component. According to Reed et. al. [8] the outer layers are more vulnerable with respect to degradation caused by high humidity. Therefore, measurements similar to those shown in figure 4 were performed roughly 3 mm closer to the edge. The results are shown in figure 5.

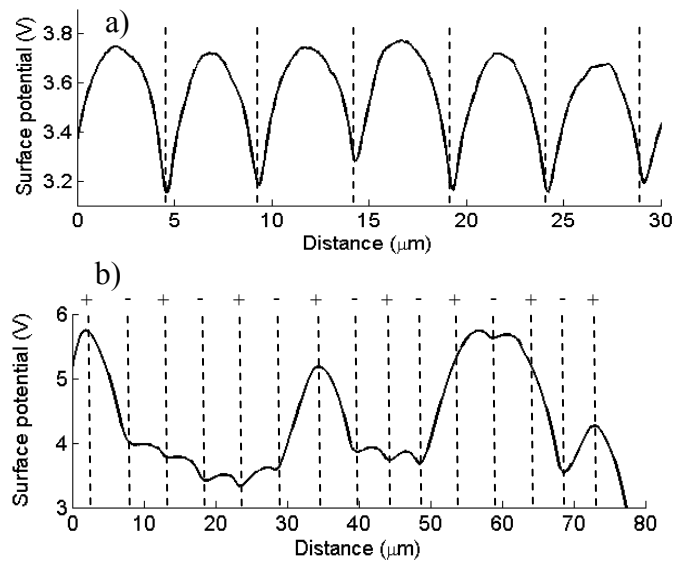

Figure 5: Cross-section profiles of the surface potential distributions measured closer to the edge of the capacitor a) on the grounded degraded capacitor and b) on the same capacitor with 5V applied across it. The dashed lines indicate the expected position of the metallization.

The measurement on the grounded capacitor in figure 5a shows a similar pattern as the one shown in figure 4a. The only difference is that the surface potential variations are smaller. The measurement on the biased capacitor (shown in figure 5b), however, is significantly different from the one shown in figure 4b. It appears that only four out of eight metallization layers preserve contact to the end-spray with the 5V applied. Moreover, at least the metallization layer at 60 µm is not connected to the grounded end-spray as it was supposed to. It could, however, also be caused by missing metallization

layers at 55 µm and 65 µm if compared to figure 4b. At this point it can not be concluded if the rest of the metallization layers are still connected to the ground. It is, however, observed that the surface potential variations at 10-30 µm and 40-50 µm are quite small (around 0.2V), which also could indicate poor connection or lack of connection to the end spray.

Based on the results presented in figures 4 and 5 two conclusions on the failure analysis can be drawn. The metallization layers were corroded and some had lost contact to the end-spray as a result of the accelerated testing. The corrosion level was independent of measurement position on the capacitor, whereas the number of metallization layers without contact to the end-spray increased in the outer layers compared to the layers near the center of the capacitor.

4 DISCUSSION

There are some important physics issues associated with KPFM that must be considered in this type of investigation. First of all, when applying KPFM to metal/dielectric heterostructures the measured surface potential will not correspond to the potential distribution only from the applied voltage across the capacitor, but also from the polarization of the dielectric due to the presence of the tip. The shape of the assumed potential distribution from the polarized dielectric was obtained by measuring on an un-biased capacitor. The fact that this potential distribution is only caused by polarization can be verified by a finite element model of the tip/sample system.

Another issue that must be taken into account in KPFM measurements is the point spread function of the tip. Electrostatic forces are long-range and therefore the measured potential in the scan point does not correspond to the actual potential, but rather a weighted sum of potentials surrounding the scan point. The weighing factors are given by the PSF, which depends on the shape of the tip. One important effect of the PSF is that the potential values at sharp peaks and valleys are shifted down and up respectively.

The purpose of this work was to use electric potential mapping by KPFM to determine the degradation type(s) that a group of film capacitors had suffered from accelerated testing. A change in CPD of the metallization layers from -0.6V to +3.2V was detected, which was attributed to corrosion of the metallization. This is based on the sign and distribution of the CPD and not the exact magnitude, due to the uncertainties caused by the PSF and uncertainties regarding to the work functions of the involved materials. Nevertheless, the KPFM technique is proved to be useful in failure analysis of the tested film capacitors.

A lack of connection between the metallization layers and the end-spray was also detected by KPFM. It was done, simply, by applying a voltage across the capacitor and measure if the metallization layers were affected by the applied voltage. It was found that approximately 20% of the metallization layers were disconnected from the end spray in the middle area, while this number significantly increased closer to the capacitor edge indicating more damage in the outer layers caused by environmental effects, as suggested in the literature.

Thus, the KPFM technique was successfully applied in FMEA on metalized film capacitors, where a conclusion on the failure mode was reached. The use of KPFM comes with requirements to the sample preparation and knowledge behind the materials. If, however, these requirements are met, the KPFM technique can prove and has proven to be useful in physical characterization of several different electric components.

5 ACKNOWLEDGEMENTS

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Dennis A. Nielsen is a Ph.D. student at the Department of Physics and Nanotechnology at Aalborg University in Denmark. His work is a part of the Center of Reliable Power Electronics (CORPE) research platform funded by the Danish Strategic Research Council and involves physical characterization of film capacitors by applying measurement techniques used in characterization of nanostructures. He received his B.Sc. in nanotechnology in 2010 and M.Sc. in nanophysics and nanomaterials in 2012 both from Aalborg University. He was one of the recipients of the Grundfos Project Award in November 2012 for his master thesis on anti-fouling surface modifications of ceramic membrane filters.

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