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Investigation of Dielectric Response Measurement as a Tool to Detect Copper Sulphide on Insulation Paper

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Master of Science in Energy and Environment
Submission date: June 2008
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Problem Description

Copper sulphide has caused several transformers to break down during the last decade. Until now there are no reliable methods to detect this problem without taking the transformer apart. Dielectric response measured on the transformer terminals is used to characterise the insulation material in a nondestructive way. Traditionally this method has been used to detect the water content of the insulation materials. The task for this master thesis is to investigate if dielectric response measurement can be a suitable method to detect copper sulphide before the transformer breaks down. Two types of instruments with some differences in frequency range are available. The measurements are performed on paper samples contaminated with copper sulphide. The samples are either grown in the laboratory or taken from a transformer with proven copper sulphide contamination.

The student's tasks:

Investigate if copper sulphide can be detected with the use of dielectric response measurements.

Do measurements on paper with copper sulphide and see how the response varies with degree of contamination.

Test different quantification methods for classification of contamination degree.

Investigate how the dielectric response depends on temperature.

Make an evaluation of how the method investigated can be taken futher so the test eventually can be carried out on a transformer in its entirety.

Assignment given: 15. January 2008
Supervisor: Hans Kristian Høidalen, ELKRAFT

Abstract

Several power transformers has, during the last two decades, broken down due to the copper sulphide problem. Corrosive sulphur reacts with the copper conductor contaminating the surrounding insulating paper. To this day there are no reliable nondestructive methods to detect copper sulphide contamination in a power transformer.

Dielectric frequency measurement is a nondestructive tool used in the transformer industry. The measurements can give important information about the condition of the transformer insulation. In this master thesis the usage of dielectric frequency measurement as a tool to detect copper sulphide on insulation paper has been investigated.

The task has been divided into two subtasks:

- **Subtask I:** Develop and test different methods to create and classify copper sulphide contamination on paper samples.
- **Subtask II:** Examine the dielectric frequency response of the contaminated samples as a function of copper sulphide

Solution and results for Subtask I
For sample creation two methods were used:

- Creating samples out of paper strips taken from existing transformers
- Growing copper sulphide on paper in the laboratory for three, five, eleven and fourteen days

For copper sulphide classification three methods were used:

- Resistance measurement
- Area percentage calculation
- Classification by the number of days grown.

For sample creation the growth of copper sulphide samples seemed to be the better choice as the gaps between the samples of strips gave large uncertainties in the dielectric frequency response. The growth varied in some extent and days of growth some times became insufficient in classifying degree of copper sulphide. Good connection between the copper and paper may be an important factor, and the size of the samples is recommended to be kept small. For all samples, contamination got heaviest at the edge. For the samples grown for fourteen days the paper became too fragile for dielectric frequency testing.

The resistance measurement classification method seemed to be the best choice, as it could cope with large variations in the contamination. The area percentage calculation method worked under very limiting conditions as the contamination had to be of a certain brightness level and with low variations in colour. The classification due to days of growth came too short as the contamination between five and seven days were not that significant. Larger time intervals can be a solution.

Solutions and Results from Subtask II

Dielectric frequency measurements were tested with two separate equipments:

- The Insulation Diagnostic Analyser: Frequency range: 1mHz–1kHz
- The Alpha-Analyser: Frequency range: 100Hz–1MHz

All tests where performed in equilibrium with air humidity at the time of testing.

The gaps between the strips, used in the samples, gave a substantial effect to the dielectric frequency response. This effect was clearest at the lower frequencies, but some effect can also be seen in the higher region. At the lower frequencies the results gave no indication of any effect in the dielectric response due to copper sulphide. However, the gap effect may have concealed the effect, and testing without gaps is recommended.

At the higher frequencies the grown copper sulphide samples gave some variations in the dielectric frequency response. The variations in results also occurred between samples with the same creation conditions. This may still indicate a relation between copper sulphide and dielectric response at higher frequencies. Even with the large deviations there were, at frequencies above 100kHz, a steeper increase in losses for the contaminated samples. Of the grown samples created only samples of five and seven days growth were tested. This gives a limited research foundation and further investigation is needed

Conclusions and Future Work

In general there might be a correlation between copper sulphide and dielectric frequency measurement. Growing samples has turned out to work well as the test object provider. By combining days of growth with resistance measurements the classification of the contamination should be sufficient. However, more research is absolutely needed and some suggestions for further work are as follows:

- Dry the samples to minimise the water effect on the dielectric frequency measurement.
- Creating a new smaller sample cell for the Insulation Diagnostic Analyser, so the sample size can be reduced and sample without gaps can be run in the lower frequency range.
- Increase the time gap between the samples grown to get larger differences in contamination, and reduce the effect of variations.
- Reducing the temperature may be a way to increase days of growth. However, in this case more research on the temperature dependencies of copper sulphide growth is needed.

Preface

This Master Thesis, dealing with the influence between copper sulphide and dielectric frequency response, is carried out at the Norwegian University of Science and Technology in cooperation with SINTEF, during the spring of 2008.

I would like to thank my supervisor Professor Hans Kristian Høidalen for his guidance and feedback during the semester. I would also like to thank Senior Researcher Lars Lundgaard for his help with result presentation and discussion, and Research Dag Linhjell for important information about equipment usage and result analysing.

The report is written in the typesetting program LaTeX

During the work some problems with the Insulation Diagnostic Analyser test cell arose. This resulted in a remake and rerun of five test performed with this equipment. As a function to this the discovery of the strip influence was discovered at a late time. This is mainly the reason for testing of samples created by strips were given such a large amount of attention.

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LIST OF FIGURES

Figure 2.1, 3.1, 3.4, 3.8, 4.1, 4.2, 5.12, 6.1, 6.3 and 6.4 are drawn by the author.

CHAPTER

ONE

INTRODUCTION

1.1 Background for the Project

In recent years several transformers have failed due to copper sulphide. Even though the phenomenon has been known since the late 40s, there is little documentation and research on this topic. In high voltage transformers, copper sulphide contamination is a fairly small problem, with only about 100 failed units worldwide[8]. However the majority of these units had only been in service for 5–10 years. Since power transformers have an expected service time of at least 30 years, the problem became far more urgent. The main portion of failures were on transformers with high load, and transformers with limited access to oxygen[8]. In addition the infected transformers worked perfectly until breakdown. A lot of questions arose during this time: What caused this problem? How many transformers were in the danger zone? How to detect the problem before breakdown? These and several other questions started a comprehensive research on this phenomenon. At the turn of the millennium the research on copper sulphide increased rapidly with CIGRÉ [1] as one of the leading actors. It turned out that transformer oil had some amount of corrosive sulphur, which had reacted with the copper windings. Sulphur makes many different compounds in oil and not all of them are corrosive. In fact some compounds actually reduces the oil oxidation rate. Oxidation creates acid and water in the oil, reducing the life time of both oil and paper[15]. As a result to this the transformer oil producers have created ways to reduce the “bad” sulphur and keep the “good” sulphur. Several international standards, such as ASTM D 12753[2] and ISO 5662[3], insures that the transformer oils on the market are safe to use. Because of the high security it was quite a shock when transformers started to break down due to copper sulphide. Even though the first failure cases were, in some extent, special cases, there is a general concern that this is only the tip of the iceberg.

Because corrosive oils passed today’s standards, modifications had to be made. A modified version of ASTM D 12753, called ASTM D 12753 B, detects corrosive sulphur by heating the test oil with a copper strip, with one layer of paper, at 150°C for 48 hours[14]. Visual detection of copper sulphide on the copper strip or the paper confirms corrosive sulphur in the oil. However, this test only check whether the oils are corrosive or not, at fairly good conditions for copper sulphide growth. There are still no reliable method to detect copper sulphide in an existing transformer. This is of big concerns since copper sulphide rarely is the sole reason for breakdown. As copper sulphide grows the insulation properties of the paper reduces. However the insulation usually still has the strength to cope with normal load conditions. It is in abnormal conditions, such as overvoltages or transients, that usually gives the stress that causes the breakdown. Because of this a “sick” transformer can work perfectly

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[1] International Council on Large Electric Systems

[2] International Organization for Standardization

[3] American Society for Testing and Materials

for several years before it suddenly breaks down. As a reliable electricity supply gets more and more important this is a problem the electrical power industry can not ignore.

1.2 The Main Purpose of the Master's Thesis

This master thesis investigates the usage of dielectric frequency measurement as a tool to detect copper sulphide on insulation paper. The task has been divided into the two subtasks:

- **Subtask I:** Develop and test different methods to create and classify copper sulphide contamination on paper samples
- **Subtask II:** Examine the dielectric frequency response of the contaminated samples as a function of copper sulphide

1.3 Report Structure

For this report the theoretical background about both dielectric frequency measurement and the copper sulphide contamination problem will be explained. Then the equipment used for both sample creation and documentation, and dielectric measurements are introduced in chapter. Walk through of the different experiments performed are stated in chapter 4. The results with comments are represented in chapter 5 followed by the discussion chapter where the results are more thoroughly investigated and discussed. The master thesis is closed with the conclusion in chapter 7. Suggestions to future work are also included in the conclusion chapter.

CHAPTER

TWO

THEORY

This chapter contains the theoretical information used and needed for the work represented in this thesis. Dielectric frequency measurement basics are explained as this is important for later discussion.

2.1 Dielectric Frequency Measurement

Dielectric frequency measurement has been shown applicable with transformers to detect water percentage in the paper[6]. The basic understanding of dielectric frequency measurement consist mainly of permittivity and polarisation, and their frequency depended behaviors. These phenomenons will be theoretically explained here.

2.1.1 Polarisation Basics

Considering an energised parallel plate capacitor in vacuum. The electrons will displace on the area of the capacitor creating a displacement field, D_0,$[C/m^2]$. This generates an electric field, E,$[V/m]$. In vacuum the correlation between E and D_0 is:

$$D_0 = \varepsilon_0 E \tag{2.1}$$

where ε_0 is the vacuum permittivity, and gives an indication of charge storage per volt-meter, $[C/Vm]$, for the *free charges* on the plates. The vacuum permittivity, or the permittivity of free space, has a constant value of $\varepsilon_0 = 8.85 \cdot 10^{-12} C/Vm$ i.e. the charge withstand strength of vacuum. In other words the permittivity gives an indication of insulation strength. From equation 2.1 it is important to notice that D_0 indicates the total amount of free charges a capacitor in vacuum can withstand, when applied an electric field E.

All insulation materials there have electrical charges within the material. These charges are exposed to a force equal to:

$$F = qE \tag{2.2}$$

where F is the force, q is the charge and E is the electric field. In most cases the charges in a material are ,in some degree, bounded. This prevents the charges to move freely. Instead they will realign or relocate to counteract the applied field. As shown in figure 2.1 this will weaken the total charge amount at the plates. This gives the capacitor the possibility to store more free charges and the displacement field rises. The new field is named D and can be written as:

$$D = D_0 + P \tag{2.3}$$

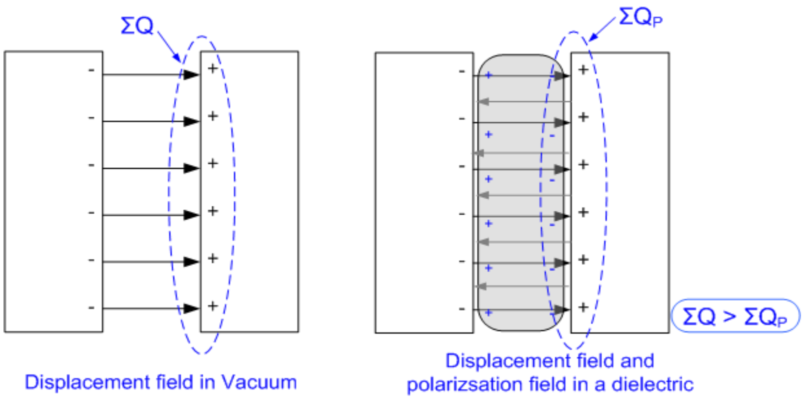

Figure 2.1: Capacitor with and without polarisation contribution. ΣQ indicates the total charge with no polarisation contribution. ΣQ_P indicates the net charge when polarisation contribution is included

where D_0 is the displacement field from the free charges and P is polarisation, or the supplement from the bounded charges. More specific explanation of the phenomenons causing polarisation are explained in chapter 2.1.2. By considering the polarisation happening momentarily, the polarisation can be rewritten as:

$$P = D_0\chi = E\epsilon_0\chi \tag{2.4}$$

χ is called electrical susceptibility and indicates how easily a material polarises when applied an electric field. By using equation 2.1 more generally and introducing relative permittivity, the total displacement field can be expressed as:

$$D = \varepsilon E = \varepsilon_0\varepsilon_r E \tag{2.5}$$

The relative permittivity is the ratio between the permittivity for an insulation material and permittivity in vacuum. By using equation 2.3 and 2.5 the polarisation becomes:

$$\begin{aligned} P &= D - D_0 \\ &= \varepsilon_0\varepsilon_r E - \epsilon_0 E \\ &= \varepsilon_0 E(\varepsilon_r - 1) \end{aligned} \tag{2.6}$$

Comparing equation 2.4 and 2.6 shows that the electrical susceptibility equals to $\chi = \varepsilon_r - 1$.

2.1.2 Dielectric Relaxation

In chapther 2.1.1 it is assumed that the permittivity is constant, or that polarisation happens instantaneously. In real life this is rarely the case. In a physical dielectric, the polarisations are induced by several different phenomenons within the material. The different polarisation mechanisms have different polarisation time, which can be categorised into the four groups[9]:

- Electronic polarisation
- Ionic polarisation
- Orientation (Molecular) polarisation
- Interfacial (Adsorption of charge) polarisation

Electronic polarisation: When an atom is exposed to an electric field the electrons and the atomic nucleus are exposed by the electric force explained in equation 2.2. Since the electrons and the atomic nucleus have charges of reverse signs the forces will work in opposite directions. This results in a slight displacement of charge, creating a dipole. This effect causes a polarisation contribution and the total charge is reduced

Ionic polarisation: Materials consisting of ions will organise the ions so the net charge is zero.

When applied an electric field, the ions rearrange and create dipoles to limit the field strength. The rearrangement will gi a polarisation contribution.
Orientation polarisation: Molecules of different types of atoms will in some cases be natural dipoles, like water. However, in natural environment, these will align so the net charge is zero. When an electric field is applied, the atoms turns to work against the field. The net charge for the molecules will no longer be zero and give a polarsation contribution.
Interfacial polarisation:A solid material is never totally homogeneous. It will always be some areas with different dielectric properties. If there are free electrons present and an electric field is applied, these electrons will move to the local area surfaces creating charge changes within the material. This type of polarisation will also occur between the layers of an insulation systems containing several materials, such as paper-oil-insulation.

Both electronic and ionic polarisation respond fast and are considered as momentarily. Orientation and interfacial polarisation are slower mechanisms and need time to fully polarise, hence it is time dependent. By assuming that all slow polarsation mechanisms have the same time dependence, a general polarisation can be expressed as:

$$P(t) = P_m + (P_s - P_m)g(t - t_0) \tag{2.7}$$

Where P_m is the momentarily polarisation and P_s is the saturation value. $g(t)$ is a function with the behavior:

$$g(t) = \begin{cases} 0, & \text{if } t < 0 \\ 1, & \text{if } t \to \infty \end{cases} \tag{2.8}$$

And with the restrictions:

$$g(t) \geq 0, \quad \tfrac{\delta g(t)}{\delta t} \geq 0 \tag{2.9}$$

This phenomenon is the foundation in dielectric relaxation and can be expressed in both time and frequency domain.

Time domain

From equation 2.6 and 2.9 the polarisation can be described as:

$$P(t) = [(\varepsilon_\infty - 1) + (\varepsilon_s - \varepsilon_\infty)g(t - t_0)]\varepsilon_0 E \tag{2.10}$$

This is however with a constant electrical field. When applying an alternating field, and assuming a linear, homogeneous and isotropic dielectric, convolution can be use to express the polarisation as:

$$P(t) = (\varepsilon_\infty - 1)\varepsilon_0 E(t) + \varepsilon_0 \int_{-\infty}^{t} f(t - t_0)E(t_0)dt_0 \tag{2.11}$$

where $f(t)$ is the response function:

$$f(t) = (\varepsilon_s - \varepsilon_\infty)\frac{\delta g}{\delta t} \tag{2.12}$$

Introducing this to equation 2.3 and 2.5 the displacement field can be expressed as:

$$D(t) = \varepsilon_\infty \varepsilon_0 E(t) + \varepsilon_0 \int_{-\infty}^{t} f(t - t_0)E(t_0)dt_0 \tag{2.13}$$

According to Maxwell's equations the current density generated by an alternation electric field is:

$$J(t) = \sigma E(t) + \frac{\delta D}{\delta t} \tag{2.14}$$

where σ is the current contribution due to condutance in the material, and $\frac{\delta D}{\delta t}$ is the current contribution due to variations in the displacement field [9].
By combining 2.13 and 2.14 and assuming $t_0 = 0$, the current density, with both dielectric relaxation and variating electrical field considered, becomes:

$$\begin{aligned} J(t) &= \sigma_0 E(t) + \varepsilon_0 \left[\frac{\delta E}{\delta t} + \frac{\delta P}{\delta t} \right] \\ &= \sigma_0 E(t) + \varepsilon_0 [\varepsilon_\infty \delta(t) + f(t)E(t)] \end{aligned} \tag{2.15}$$

Here $\sigma_0 E(t)$ represents the direct current contribution, $\varepsilon_\infty \delta(t)$ is the momentarily polarisation part, and $f(t)E(t)$ is the current caused by dielectric relaxation.

Frequency Domain

To get from time domain to frequency domain Fourier transform is used. The relation between time and frequency is stated as:

$$k^*(\omega) = \int_{-\infty}^{\infty} e^{-j\omega t} k(t) dt \tag{2.16}$$

where * detonates a complex quantity
It turns out that the Fourier transform of the dielectric response function $f(t)$(see equation 2.12) equals to the complex electric susceptibility [10],[17]:

$$\chi^*(\omega) = \chi'(\omega) + j\chi''(\omega) = \int_{-\infty}^{\infty} e^{-j\omega t} f(t) dt \tag{2.17}$$

where $j = \sqrt{-1}$
By applying this to equation 2.13 the displacement field in frequency domain becomes:

$$D^*(\omega) = \varepsilon_0 (\varepsilon_\infty + \chi^*) E^*(\omega) \tag{2.18}$$

Combining this with Maxwell's current density equation 2.14, the complex frequency dependent current density can be written as:

$$J^*(\omega) = j\omega\varepsilon_0 \left[\varepsilon_\infty + \chi'(\omega) - j \left(\frac{\sigma}{\varepsilon_0 \omega} + \chi''(\omega) \right) \right] E^*(\omega) \tag{2.19}$$

By physically current density measurements the permittivity can be calculated. However the measurement system can not see the difference between the contribution from conductivity and the contribution from slow polarisation [17]. This means that the measured permittivity differ from the theoretical defined in equation 2.5. Instead the measured dielectric relative permittivity ε^*_{rm} is defined from:

$$J^*(\omega) = j\omega\varepsilon_0 \varepsilon^*_{rm}(\omega) E^*(\omega) \quad \text{where} \quad \varepsilon^*_{rm}(\omega) = \varepsilon'_{rm}(\omega) + j\varepsilon''_{rm}(\omega) \tag{2.20}$$

By combining equation 2.19 and 2.20 the real and imaginary parts of the measured permittivity ε^*_{rm}, can be written as:

$$\varepsilon'_{rm}(\omega) = \varepsilon_\infty + \chi'(\omega) \tag{2.21}$$

$$\varepsilon''_{rm}(\omega) = \frac{\sigma}{\varepsilon_0 \omega} + \chi''(\omega) \tag{2.22}$$

Since equation 2.20 it can be seen that the real part of the measured permittivity represents the imaginary part of the current density and the imaginary part of ε^*_{rm} is real in the current density equation. This means that ε''_{rm} re the represent the losses of the material, while ε'_{rm} is the capasitive part. As seen in equation 2.21 the loss parameter is dependent on both the conductivity and the imaginary susceptibility. However the conductivity will reduce with increasing frequncy. This states that the conductivity of a material is give a significant loss contribution for lower frequencies.

2.1.3 Temperature Dependence

The dielectric frequency response of solid insulation materials is highly depended on temperature. However, for several solid materials, change in temperature gives shift along the frequency axis only. The slope of the permittivity readings stays the same. This gives the possibility to recalculate measurements from one temperature to another, increasing the frequency range beyond the actual range of measurement. These calculated curves are called “master curves” and can increase the information amount from the measurements. It is important to note that this method only works if the slope of the reading are unchanged for all temperatures.

2.2 Copper Sulphide

2.2.1 Basic Description

Copper sulfide in General

All compounds with a chemical formula Cu_xS_y belongs to the copper sulphide family. In a transformer the copper sulphide created is primarily Cu_2S (Chalcocite), with a small percentage of CuS(Covellite). Through this thesis, however, Cu_2S will describe cobber sulphide in general, as this is the main component. The colour of copper sulphide has a wide specter spanning from bluish gray to black and in some cases even green or violet. This makes it difficult to detect and confirm copper sulphide visually. Chalcocite contains a large amount of copper giving the mineral high conductivity [16].

Sulphur Compounds in Transformer Oil

Transformer oils contain sulphur in the rage of 0.01% - 0.5,%[14]. Sulphur can appear in several different chemical compounds. The different types and there reactiveness are shown in table 2.1. The table shows that elemental sulphur and mercaptans are the most corrosive and are therefore most likely to attack the copper conductor. This is no new discovery for the transformer oil producers. As a matter of fact, as mentioned in chapter 1.1, some sulphur compounds actually reduce the oxidation speed of the oil. Therefore oil producers distinct between corrosive [1] and noncorrosive sulphur. Because of the oxidation benefit the transformer oil producers have found ways to keep the stable sulphur, while reducing the reactive. This is mainly done by chemically transforming corrosive sulphur compounds into noncorrosive compounds[11]. As a result of this there are several international tests, to check the corrosiveness of oils, such as ISO 5662 and ASTM D 1275. In the last few decades it has turned out that even though oils have passed these tests, in some cases, corrosive sulphur are still present. Whether the corrosive part has been there from the start or been created over time is not certain. Still this has started a comprehensive research on this problem such as CIGRÉ’s WGA2.32 [2].

Type	*Formula*[a]	*Reactivity*
Elemental Sulphur	S	very reactive
Mercaptans	$R-SH$	very reactive
Suphides	$R-S-R_1$	reactive
Disulhides	$R-S-S-R$	stable
Thiophides	5-membered ring with S	very stable

Table 2.1: Corrosiveness for Different Sulphur Molecules, [11]

[a] R and R_1 are strings of hydrocarbons

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[1] Oils with corrosive sulphur is defined as oil that gives gray or brown contamination on a silver strip after 18 hours of heating at 100°C. Unlimited access to oxygen

[2] http://www.cigre-a2.org/Site/What/download/WG%20a2.32%20scopeid15ver42.pdf

CHAPTER

THREE

EQUIPMENT

3.1 Dielectric Measurement Setup

For the dielectric frequency readings, two different instruments are used. These are:

- Insulation Diagnostic Analyser - for frequencies from 1mHz to 1kHz
- Alpha-Analyser - for frequencies from 100Hz to 1MHz

The need for different instruments are mainly because of the physical effects, in the sample cell materials, when applied variating fields. However the basic measurement concept is the same for both instruments and is explained solely. The specific differences between the two instruments are explained in subsection 3.1.2 and 3.1.3.

3.1.1 Measurement Basics

Basically the permittivity and conductivity of a test object is calculated from the impedance. The impedance is found by measuring the voltage across and the current through the test object. See figure 3.1. The voltage frequency is regulated by the computer and the voltage source. The measured voltage and current can be written as:

$$\begin{aligned} U(t) &= U_0 cos(\omega t) = Re(U^* e^{i\omega t}) && (3.1) \\ I(t) &= I_0 cos(\omega t + \varphi) = Re(I^* e^{i\omega t}) && (3.2) \end{aligned}$$

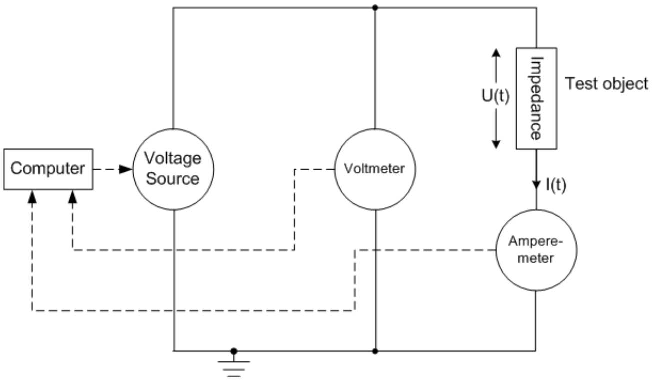

Figure 3.1: Measurement circuit

where:

$$\begin{aligned}&U^* = U_0\\&I^* = I' + jI'' \quad I_0 = \sqrt{I'^2 + I''^2} \quad \tan(\varphi) = \tfrac{I''}{I'}\end{aligned}$$

φ is the phase angle between voltage and current.

Given that the electromagnetic response is linear, i.e. the impedance is constant for this specific frequency, the test object impedance can then be calculated by Ohms law:

$$Z^* = Z' + jZ'' = \frac{U^*}{I^*} \tag{3.3}$$

The complex capacitance model is defined as:

$$Z^* = \frac{1}{j\omega C^*}; \quad \text{where} \quad C^* = C' - jC'' \tag{3.4}$$

The complex relative permittivity can be derived by:

$$\varepsilon_r^* = \varepsilon_r' + j\varepsilon_r'' = \frac{C^*}{C_0} \tag{3.5}$$

C_0 is the capacitance of the empty measurement cell and can be calculated using the relation:

$$C_0 = \frac{\varepsilon_0 A}{l} \tag{3.6}$$

where ε_0 is the permittivity in vacuum, A is the area of the plates and l is the gap length between. It is important to remember that the complex permittivity in this case is the same as in equation 2.22, which include the conductivity.

3.1.2 Insulation Diagnostic Analyser

The Insulation Diagnostic Analyser is used for low frequencies. The system have been proved to work well on transformer paper samples in a frequency range of 1 mHz to 1 kHz [6].
The IDA system consist of the items[1]:

- The IDA mainframe
- Termination box
- Specially designed sample cell, with guard system
- Oil tank
- Circulator, HAAKE Phoenix II P1
- The IDA software

The mainframe is the heart of the system and provide the necessary amplitudes and frequencies. From the mainframe the connection goes to **the termination box** which has the outlets to the high, low, guard and ground connections. As shown in figure 3.3 the system can be connected in four different ways.

The sample cell for the Insulation Diagnostic Analyser is shown in figure 3.2. The outer structure consist of two discs put together as a capacitor. For insulation a disc of non-conductive plastic is placed between the capacitor discs. The plastic disc has a center hole for test sample placement. The top metal disc has an adjustable part in the center. A spring system press the adjustable part to the test object giving the necessary pressure. The bottom metal disc is divided into a measurement area with a diameter of 60mm. The area can be seen as the area within the red circle in picture 3.2. The steel area outside the red circle is the electrode for the **guard system**. The basics of the guard system is explained later in this chapter. For temperature control and cooling the test object cell is placed in an **oil chamber**. The temperature in the chamber is controlled by the **Circulator, HAAKE Phoenix II P1**.

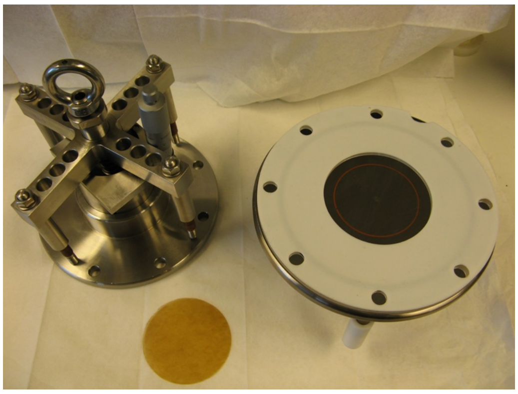

Figure 3.2: Sample cell for the IDA system. Top disc to the left

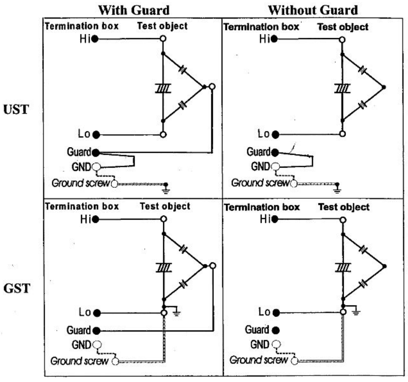

Figure 3.3: Different connection possibilities for the IDA system

General Understanding of the Guard System

The principle of the guard system is shown in figure 3.4. The divided low potential plate cuts out the stray field and gives a more homogeneous electric field to measure. If the system is not grounded the voltage on the lower disc can vary. This creates the possibility of stray field between the low side and guard. Therefore the potential between guard and the low side should be as small as possible.

The guard system works best at relatively low frequencies. If the frequency gets to high the stray field between the low side electrode and the guard electrode gives too much error. In addition the sample cell for the Insulation Diagnostic Analyser is made of steel, which gives too large losses to neglect. Due to these reasons the maximum frequency applied for the Insulation Diagnostic Analyser is limited to 1kHz.

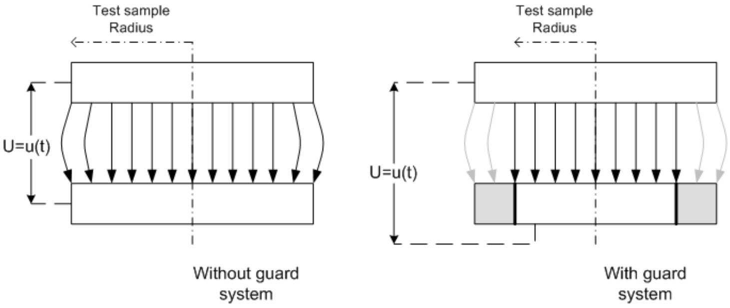

Figure 3.4: Basic principle of the guard electrode system

3.1.3 Alpha-Analyser

The Alpha-Analyser is used for higher frequencies. According to Jan Trygve Borlaug's master's thesis [2] the readings are most exact in a frequency range of 100Hz to 1MHz.

- The Alpha mainframe
- 4-wire Impedance Interface ZG4
- Specially designed sample cell in teflon, no guard system
- Oil chamber
- Circulator, HAAKE Phoenix II P1
- Labview software program

The Alpha mainframe consists of a frequency analyser and power supply for active and direct current. Hence a dielectric frequency spectroscopy can be run with this instrument alone. However, for wider impedance range and high sensitivity measurement, this connection often gets insufficient. Mainly because of the neglection of cable and contact impedances. **The 4-wire Impedance Interface ZG4** solves this problem by a more advanced measurement sequence. Figure 3.6 shows how the impedance interface is built up. GEN is the the port for the applied sine wave, generated by the mainframe. V_1 and V_2 are measurement ports and calculates amplitudes and phase shifts over the test object. I_{high} and V_{high} measures on the high potential side of the test object while I_{low} and V_{low} measures on the low side. By combining for instance I_{high} and V_{high} the Alpha Mainframe can cancel out the cable and contact impedances. Same applies for the low side. However, according to [3], the I_{low} can create some additional currents which can give measurement errors. Due to this the use of 4-wire mode should be limited to measurements in specific need for this connection. Figure 3.5 shows the **sample cell** for the Alpha-Analyser, and has a diameter of 25mm. The cell is made of teflon, which has low losses at high frequencies[2].

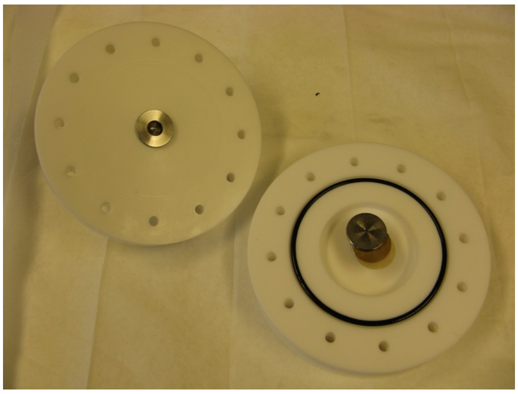

Figure 3.5: Sample cell for the Alpha-Analyser system

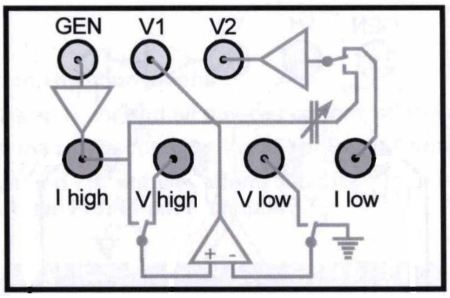

Figure 3.6: 4-wire Imedance Interface

3.2 Water Content Measuring Equipment

3.2.1 737 Karl-Fisher Coulometer

The 737 KF Coulometer with special sample chamber is used to measure water by the Karl-Fisher titration method[13]. The basic behind the Karl-Fisher detection method is based on several chemical reactions which can be summarised in the overall equation 3.7. The water in the paper sample is evaporated and guided into a chamber where it will react according to the chemical reaction. By applying an alternating electrical voltage between to electrodes in the chamber the current flowing is strongly dependent on the free iodine, I_2, present. As more iodine reacts with the water the current between the electrodes will drop. By analysing the voltage signal the water content can be derived.

$$H_2O + I_2 + [RNH]^{1}SO_3CH_3 + SO_3CH_3 + 2RN \rightarrow [RNH]SO_4CH_3 + 2[RNH]I \tag{3.7}$$

3.3 Resistance Measurement Equipment

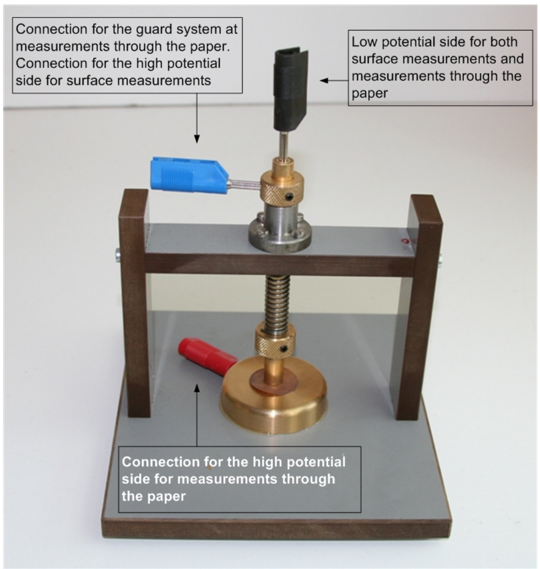

Figure 3.7: Resistance Measurement Equipment. The figure shows a sample ready for measurements through the paper. For surface measurements a plate of teflon was placed between the paper and the lower, large electrode.

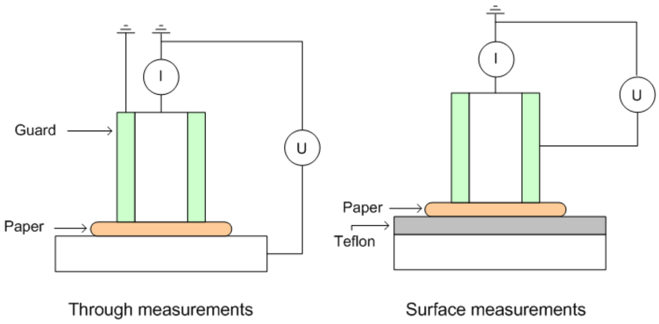

Figure 3.8: Connections for resistance measurements through the sample and on the surface

Resistance measurements were performed on all paper samples used for research. For resistances in the range of ohms to mega ohms a FLUKE112 Multimeter was used, while a Megger S1-5010 was used for resistances up to several hundred giga ohms. The paper samples were placed on the measurement platform as shown in figure 3.7. Both resistance measurements through the paper and on the surface were performed. The connections for the different resistance measurements are shown in figure 3.8. The guard system has the same potential as the low side[2], but does not constitute a part of the measurement. A generalised understanding of the guard system is stated in section 3.1.2.

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[1] RN is the buffer base and helps to stabilise the pH-value

[2] The Megger has an isolated ground, with connection possibilities for high, low and ground.

3.4 Software

3.4.1 ImageJ

To calculate the area contamination of copper sulphide, the image processing program ImageJ was used. The program operates by counting the amount of pixels over a given brightness degree. Coloured pictures of the test samples were loaded into the program. Then the brightness were adjusted to give maximum differences between the paper and the copper sulphide brightness. The pictures were converted to black and white for the area percentage calculation tool to work. The brightness degree of a pixel is divided into 255 levels with 0 as totally black and 255 as entirely white. The program colours all pixels above a given brightness level. By marking a wanted area the program calculates the percentage amount of red pixels within that area.

3.4.2 ABB MasterCurves

To calculate the master curves for the Alpha-Analyser readings, a computer program created by ABB was used. The program recalculate the readings from the actual test temperature to the decided master curve temperature. The program uses the principles explained in section 2.1.3.

CHAPTER

FOUR

EXPERIMENTS

4.1 Test Sample Creation

All test samples contained of three layers of paper. In most cases the two outer layers were of clean and whole Termo70 paper. The test samples were created in two ways; with paper from two retired transformers, and paper samples created in the laboratory.

4.1.1 Sample Creation Materials

The paper used for sample creation was taken from the transformers: The Skagerrak HVDC-transformer PT32 and the Sira-Kvina transformer.

The Skagerrak HVDC-transformer PT32
The Skagerrak transformer was put into service in 1993 in the HVDC connection between Norway and Denmark. In 2003 the transformer broke down due to flashover in one of the phases. The phase windings were replaced and the transformer were put back into service. The cause of failure was not found. Two years later the transformer broke down again and it was discovered that the transformer was heavily affected by copper sulphide. The transformer windings were isolated with paper strips wounded around the conductor. The paper strips has a width of 15mm.

The Sira-Kvina Transformer
The Sira-Kvina Transformer were taken out of service in 2008 after 38 years of service. The transformer has no signs of copper sulphide and is used as a reference of old paper with good insulation qualities. The insulation paper wounded around the conductor has a width of 12mm.

For the samples with grown contamination clean and new Termo70 insulation paper was used. The corrosive oil used for growth was Nytro10X. Data sheet for this oil is placed in appendix A.34.

4.1.2 Samples Created from Paper Strips

The paper strips were taken from the two transformers described above. As the test cells used in this research are circular, the strips were flattened and cut to fit the respective cells. The illustration in figure 4.1 shows the strips between the two clean and whole layers. To get the paper strips flatten, the paper was moistened and placed between two teflon plates. The plates were heated in a heat chamber at 50°C for 72 hours. To give some pressure effect, 4kg of weights were placed on top of the plates. The Alpha-Analyser has a test cell diameter of 25mm. The Insulation Diagnostic Analyser has a electrode diameter of 60mm. However, since the analyser has a guard system, the test samples was created with a diameter of 75mm. See chapter 3.1.2 for more information.

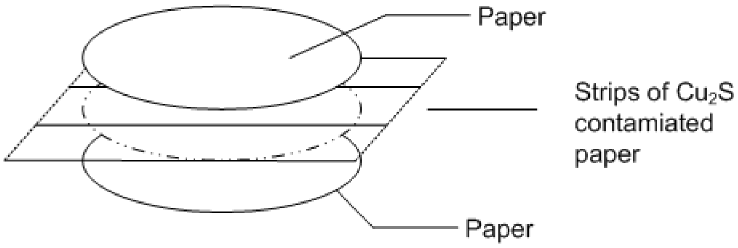

Figure 4.1: Sample made from transformer paper strips

Gap Consequences

As seen in figure 4.1 in chapter 4.1.2 this sample creation method will create gaps between the strips. Whether this effects the measurements or not is not known and must be investigated. The method used is to first run a test with 3 layers of whole papers. In the next test the paper in the middle is cut in strips with the same width as the paper from the existing transformer. A possible difference may then be detected by comparing the two results.

4.1.3 Growing Paper Samples with Different Cu_2S Contamination

Until now the most reliable way to diagnose copper sulphide is to check whether the oil i corrosive or not. CIGRÉ has one way of doing this by putting a strip of clean transformer winding, with paper, in an oil bath of transformer oil [4]. The container is then sealed and heated up. Depending on the corrosiveness of the oil sulphur will react with the copper. The contamination can be detected on either the strip, the paper or both. The controll of the results is done by visual detection of copper sulphide contamination. The test has two outcomes, noncorrosive or corrosive. This means that the amount of copper sulphide contamination does not effect the result as long as there is contamination. However, the method shows that growing of Cu_2S is feasible. Whether the amount of contamination can be controlled has been tested out in this thesis.

For the paper samples to fit the test cell they need to be circular shaped with a diameter of 25mm. To get the best possible connection surface between paper and copper, the paper sample was fixed to a copper cylinder as shown i figure 4.2. The cylinder has a diameter of 8mm and a high of 30mm. Cotton thread was used to fasten the paper to the cylinder. Then the test object was placed in a test tube filled with 15 ml of corrosive oil, in this case Nytro 10X[1]. The test tube was then placed in a heat chamber and heated up to 150 degrees. The contamination degree of copper sulphide was set by the number of days inside the heat chamber. Due to the sample size needed for the Insulation Diagnostic Analyser, the growing of samples has only been done for the Alpha-Analyser.

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[1] Nytro10X oil is a commercial oil which turned corrosive. The oil was taken off the marked and replaced with the new Nytro10XN oil.

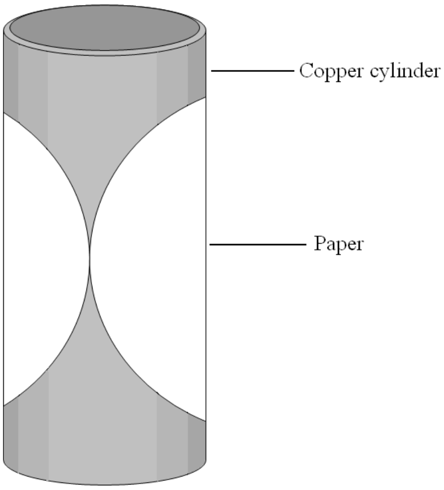

Figure 4.2: Way of making Cu_2S samples

Figure 4.3: Example of created sample ready for copper sulphide growth. The glass tube contains corrosive Nytro10X oil

4.1.4 Documentation of Contamination Degree

Classification of the copper contamination was performed and tested with the three methods, Resistance measurement, area percentage calculation and classification after number of days grown in the laboratory.

To classify the grade for contamination the resistance was measured for surface resistance and resistance through the paper samples. The equipments used were a Megger-5010, a FLUKE112 Multimeter together with a specially designed measurement set-up. See chapter 3.3, for more information. To get a general understanding of the insulation properties of the paper samples, measurements were done at several locations on the samples. Pictures of both sides of the test samples were taken and resistance measurements were documented in the pictures.

The area percentage calculation were performed with the ImageJ computer program, explained in section 3.4.1. The program marked the pixels in the picture over a given brightness level with a red colour. By comparing the red areas with the contaminated areas on the picture originally loaded into the program, the validation of the percentage calculation could be made. Only samples where this method worked has the percentage value documented.

The grown samples were grown for three days, five days, seven days and fourteen days. By comparing with the results from the resistance measurements validity of this classification method could be checked.

4.2 Dielectric Response Measurements

4.2.1 Dielectric Response Measurements in the Frequency Range of 1mHz–1kHz

For the lower frequency range the Insulation Diagnostic Analyser was used. The samples used are created from the existing transformer windings. Paper strips from both Skagerrak and Sira-Kvina were tested. As shown in picture 4.1 that for the samples created of strips, the strips were cut to fit the sample cell. For the lower frequency range the diameter was 75mm. The strips were placed between two circular pieces of clean paper of same size. Nytro10XN [2] transformer oil were used for impregnation of the test samples. The oil had been placed in open air for at least 24 hours to get in equilibrium with the air humidity. During testing the sample cells were placed in an oil bath. For the lower frequency range the oil bath had a constant temperature of 20°C.

For all experiments with the Insulation Diagnostic Analyser the system was isolated from ground, and the guard system had the same potential as the low side. See the UST connection in figure 3.3 for more information.

Tests with the Insulation Diagnostic Analyser system used the following parameters:

Frequency range: 1kHz – 1mHz

Frequency step length: 50% reduction from previous frequency

Voltage levels: 200V, 100V, 50V and 25V.

Temperature level: Room temperature

Total running time: 6:30h

The samples were cut by hand with and diameter between 65mm and 75mm

4.2.2 Dielectric Response Measurements in the Frequency Range of 100Hz–1MHz

The Alpha-Analyzer were tested on samples created from paper strips taken from both of the existing transformers and on samples grown in the lab. Nytro10XN transformer oil were used as impregnation, and given the same treatment as the oil used for the Insulation Diagnostic Analyser samples, explained in section 4.2.1.

Tests with the Alpha-Analyser system used the following parameters:

Frequency range: 1MHz – 100Hz

Frequency step length: 30% reduction from previous frequency

Voltage level: 3V

Temperature levels: 20°C, 40°C, 60°C, 80°C, 20°C, 0°C, -10°C, 20°C

Time between temperature measurements: 30min

Load-short calibation: Always activated

Total running time: 40h

The samples were made with a 25 mm hollow cylinder

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[2] The replacer for the corrosive Nytro10X. Nytro10XN is not corrosive after the renewed standards. Data sheet for Nytro10XN is placed in appendixA.35

4.3 Water Percentage Calculation

As the dielectric response is highly influenced by the water content in the test object, the water percentage is needed. The physical amount of water, in micro grams, in each test sample was found by the use of the 737 KF[3] Coulometer. Then the samples were cleaned in heptane, toluene and isopropanol. The samples were dried in a vacuum oven at 60°C for 72 hours. Then the samples were taken out one by one and the weight was measured. The time of air humidity exposure were reduced to a minimum. For each time a sample was taken out, an additional half hour in vacuum were given to the remaining samples in the oven. The water amount in percent was calculated by the equation 4.1, where M_{water} is the water amount found by the coulometer and M_{dry} is the dry wight.

$$P_{water}[\%] = \frac{M_{water}[\mu g]}{M_{dry}[\mu g] + M_{water}[\mu g]} \cdot 100\% \tag{4.1}$$

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[3] Karl-Fisher

CHAPTER

FIVE

RESULTS

The result chapter contains the comparison between the different tests and the most significant results from sample creation and documentation. Detailed documentation for all of the test samples, with dielectric frequency measurement, are placed in the appendix chapter.

5.1 Test Summary

Table 5.1 shows the different tests performed with the Insulation Diagnostic Analyser. The test samples were placed between two layers of whole and clean pieces of Termo70 insulation paper. All samples where in equilibrium with the air humidity at the time of testing.

No.	*Test sample specification*	*Comments*	*Water amount*
IDA1	Whole and clean paper	Verification of the system	5.4 %
IDA2	Strips of clean paper	See the effect of gaps between the strips	5.2 %
IDA3	Highly contaminated paper strips	See the effect of copper sulphide	4.6 %
IDA4	Paper from old, clean transformer	Reference sample	4.3 %
IDA5	Paper with moderate contamination	See the effect of copper sulphide	4.2 %

Table 5.1: Tests performed with the Insulation Diagnostic Analyser.

Table 5.2 shows the tests performed with the Alpha-Analyser. All test were performed with three layers of paper. In all tests, except number 5, the two outer layers were new, clean and whole pieces of Termo70 paper. For test 5 all layers where highly contaminated with Cu_2S. All test were performed at air humidity equilibrium at the time of testing. Test 6 and 8 were grown in the laboratory for five days, while test 7 was grown for seven days.

No.	*Test sample specification*	*Comments*	*Water amount*
AA1	Whole and clean paper	Verification of the system	4.6 %
AA2	Strips of clean paper	Influence of gaps between the strips	5.3 %
AA3	Highly contaminated strips	Influence of copper sulphide	3.5 %
AA4	Paper from old, clean transformer	Reference sample	4.4 %
AA5	Highly contaminated strips	All layers contaminated	3.4 %
AA6	Grown sample, 5 days	No gaps, air humidity	3.8 %
AA7	Grown sample, 7 days	No gaps, air humidity	4.0 %
AA8	Grown sample, 5 days - Test 2	No gaps, air humidity	4.3 %

Table 5.2: Tests performed with the Alpha-Analyser

5.2 Sample Creation and Documentation

5.2.1 Samples Created with Paper Strips

Sample IDA3, IDA5, AA3 and AA5 were created with paper samples taken from the, copper sulphide contained, transformer Skagerrak. Figure 5.1 shows the test sample for the IDA3 test. As the measurement methods and results are fairly similar for the three other samples, the results for the these test are not included in the result chapter. All information about the other samples are placed in the appendices. The area percentage calculation is also represented, in figure 5.2. Considering the resistance measurements in figure 5.1. The paper used for sample creation had been in direct contact with the copper conductor, when still in service. The toward and away side indicate the side of the paper that respectively was facing against and facing away from the conductor. The toward side shows a layer of several different colours and resistance readings in the kilo ohm area. The through measurements are somewhat higher than the surface readings. The away side shows dark paper with spots of silver coloured contamination. Resistance measurements on the silver areas gave much lower resistance than at the dark areas. One of the area measurements through the paper has been done on both sides. These areas are pointed out by the green arrows. The reading taken on the toward side shows a resistance value that is almost five times lower than on the away side. This phenomenon is more thoroughly discussed in chapter 6.1.2.

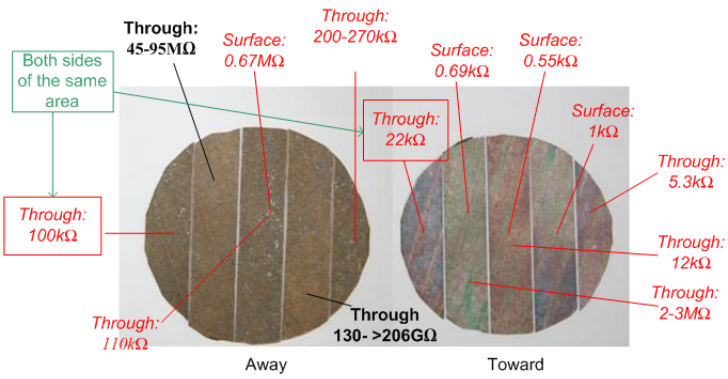

Figure 5.1: Test IDA3: The test sample. Red and italic - Multimeter, Black and bold - Megger at 200V

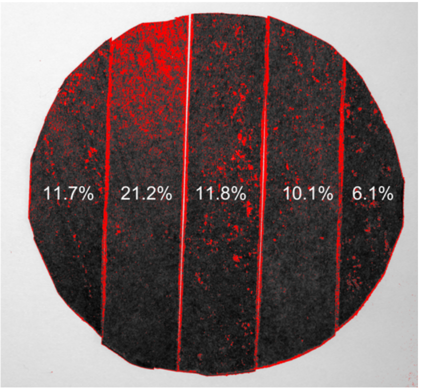

Figure 5.2: Test IDA3: Area percentage measurement and calculations. Red indicates copper sulphide contamination

Figure 5.2 shows the area percentage of copper sulphide on each strip. The calculations were performed with the computer program ImageJ. See section 3.4.1 for the software information. The red colour indicates pixels with a brightness level over a given value, and represents the copper sulphide contamination of the sample. By creating a frame around one of the strips ImageJ calculated the percentage amount of red pixels within that area. The calculations are stated in the figure with white text. The area percentage calculation worked well for the away side of sample IDA3, AA3 and AA5. For the rest of the contaminated samples the copper sulphide colours and brightness levels differed too much. This will be discussed more thoroughly in chapter 6.1.2.

5.2.2 Samples Created by Controlled Copper Sulphide Growth

Figure 5.3 shows two samples grown for seven days[1], while figure 5.4 shows the AA8 test, grown for five days. All three tests were grown at a temperature of 150°C. For the samples in figure 5.3 only the toward side is shown. Visual inspection shows that the sample to the left has dark areas around almost the entire edge, which in some cases also had a bluish silver colour. Dark areas are also located at the inner part of the sample. The sample to the right, in figure 5.3, has just some areas at the edge

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[1] These tests were never tested for dielectric frequency measurement but are included as general examples of growth

with dark colour. The bluish silver colour is also present in some few areas. In addition the sample seems to have a layer of light contamination over most of the surface. The sample grown for five days, shown in figure 5.4, looks fairly similar to the sample at the right in figure 5.3. Also at this sample the bluish silver is present in some areas. The resistance measurements show that the reduction of resistance is heaviest at the edge of the samples. Most of the contamination had grown at the surface facing toward the copper cylinder. At some areas around the edge the copper sulphide growth has also reduced the surface resistance on the away side. This can be seen in figure 5.4 and is pointed out with the green arrows. See appendix figure A.25, A.27 for more examples of this phenomenon. The resistance measurements stretches from a value of $206G\Omega$ to below $50k\Omega$ at the area with heaviest contamination. Due to large electrodes, measurements through the paper were not possible at the edge.

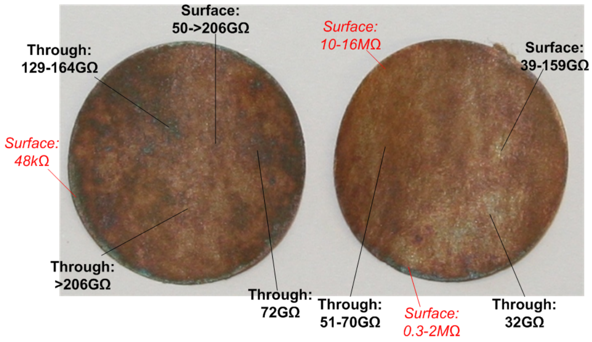

Figure 5.3: Two paper samples with copper sulphide contamination grown for seven days. Only the side that faced toward the copper during growth is shown.

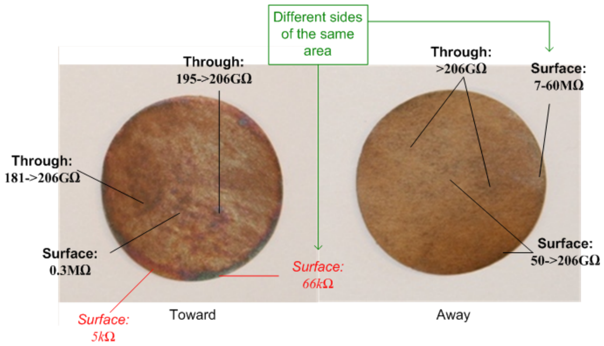

Figure 5.4: The toward and away side for the AA8 test with resistance measurements. The test was grown for five days.

Samples Grown, but not Tested for Dielectric Frequency Response

Samples grown for three days, eleven days and fourteen days were also created. However the permittivity response was not tested. Resistance measurement for these tests are placed in appendix A.15. The samples grown for three days, had a thin silver coloured layer on the side in contact with the copper cylinder. The resistance measurements indicated some surface reduction, around the edge of the paper, on the side facing toward the copper. No reduction through the paper or at the surface on the away side was found. These tests were not prioritised as the differences compared to clean paper were little. For the eleven days test the contamination is heavy and the measurements through the paper indicated high insulation degradation. Due to time limitations the test grown for three and eleven days were not run. The tests grown for fourteen days were not run due to the paper quality became too fragile. This can be seen in appendix figure A.33.

5.3 Dielectric Frequency Measurement Results

5.3.1 Master Curve Creation

Master curves were calculated by the computer program supplied by ABB, see section 3.4.2. As measurements with the Insulation Diagnostic Analyser were carried out at just one single temperature, master curve calculations were not possible. For the Alpha-Analyser measurements master curves were generated for 20°C. Figure 5.5 shows the master curve for the AA1 test, whole and clean paper layers. The graphs for the different temperatures have rather large variations. Especially for the permittivity loss readings, ε''. This means that the slope of the dielectric frequency response changes with temperature. As constant slope is one of the assumptions behind the master curve theory, master curve creation is not possible for these measurements. This applies for all readings done with the Alpha-Analyser, and further master curve calculations will not be carried out.

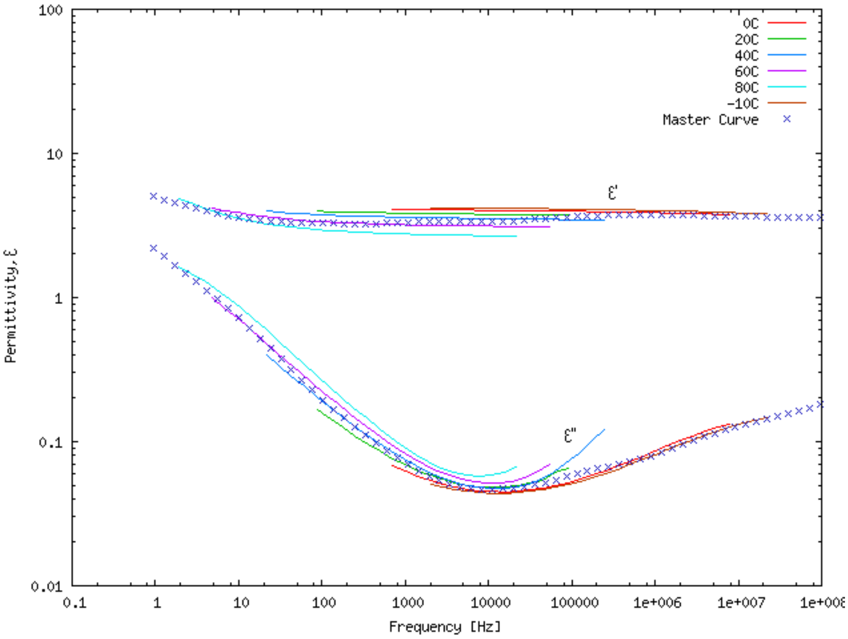

Figure 5.5: Master curve calculated for 20°C for the AA1 test.

5.3.2 Comparison of Dielectric Frequency Measurements at the Lower Frequency Range

Figure 5.6 and Figure 5.7 show the dielectric frequency response at $25V$ and $50V$ for all the Insulation Diagnostic Analyser tests. In figure 5.7 the $200V$ reading for the IDA1 test is also included and represented with dots. The figures contain both permittivity and permittivity loss readings for all test samples. These are represented as ε' and ε'' respectively. The number of gaps between the strips, for the middle layer, is also stated. IDA1 has a of whole middle layer, hence no gaps. IDA2, IDA3 and IDA5 were all made of 15mm paper strips and have four gaps. The IDA4 test was created of paper strips of 12mm width and has an additional gap giving a total of five gaps in the middle layer. The graphs show that the tests with four gaps follow the same slope with small variations. Considering frequencies above 10Hz, the IDA4 test has the highest permittivity losses. The IDA1 test, with no gaps, has the lowest losses in this frequency range. For frequencies below 10Hz the IDA4 readings coincide with the other test samples containing gaps. For the IDA1 test the losses increase and exceed the other test readings at a frequency of approximately 8Hz. For frequencies below 0.1Hz all loss readings have the same value.
Comparing the two figures 5.6 and 5.7 the readings show that the the difference between the samples increase with voltage.
From the appendix figures A.3, A.5, A.8, A.10 and A.12 is can be seen that samples containing gaps are much more influcened by the voltage level when it comes to permittivity losses. The readings for the IDA1 test is fairly the same for all voltages, while for the samples containing gaps the losses differ with voltage.
Considering the $200V$ reading, included in figure 5.7, the measurement shows a much lower loss characteristic at frequencies above 0.1Hz. This will be discussed in chapter 6.
For the ε' all test has the same slope with some deviations at a frequency range of 1Hz to 10Hz. The variations increase some with increasing voltage.

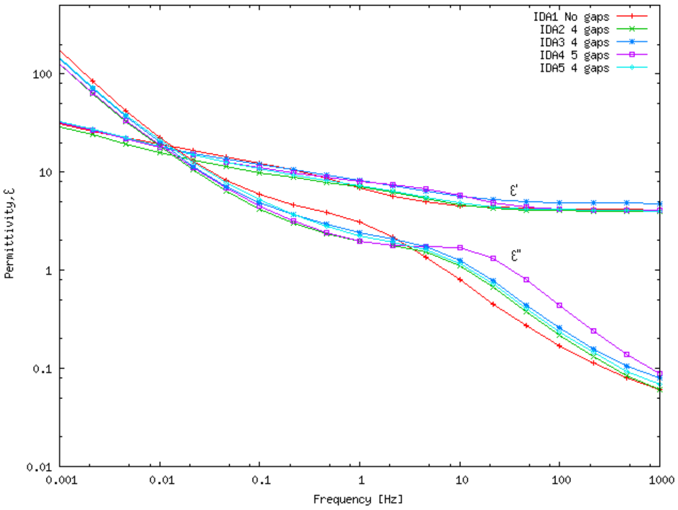

Figure 5.6: Comparison of the IDA1, IDA2, IDA3, IDA4 and IDA5 test, at 25V

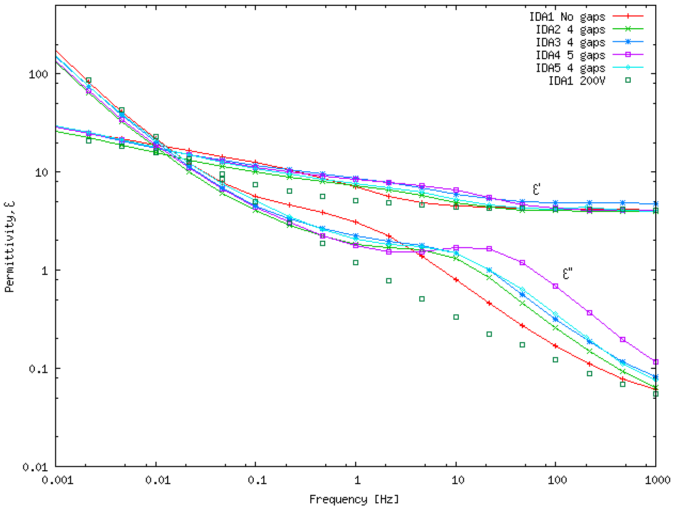

Figure 5.7: Comparison of the IDA1, IDA2, IDA3, IDA4 and IDA5 test, at 50V. The 200V reading for IDA1 is also included

5.3.3 Comparison of Dielectric Frequency Measurements at the Higher Frequency Range

Comparison of the Samples with Gaps

Figure 5.8 shows the results from the Alpha-Analyser measurements for samples *with gaps*. The test result for the whole and clean test, AA1, is included for comparison. The graph contains both permittivity and permittivity loss readings for all samples, shown as ε' and ε'' in the figure. The graph shows that, for frequencies below 10^4Hz, samples containing gaps has lower permittiviy losses than the sample with no gaps. For higher frequencies the samples with one gap only, has an increase in losses which exceeds the AA1 test sample. For the AA4 test the losses continues to decrease to a minimum at a frequency of around $2.5 \cdot 10^4$Hz, continued by a slow increase.

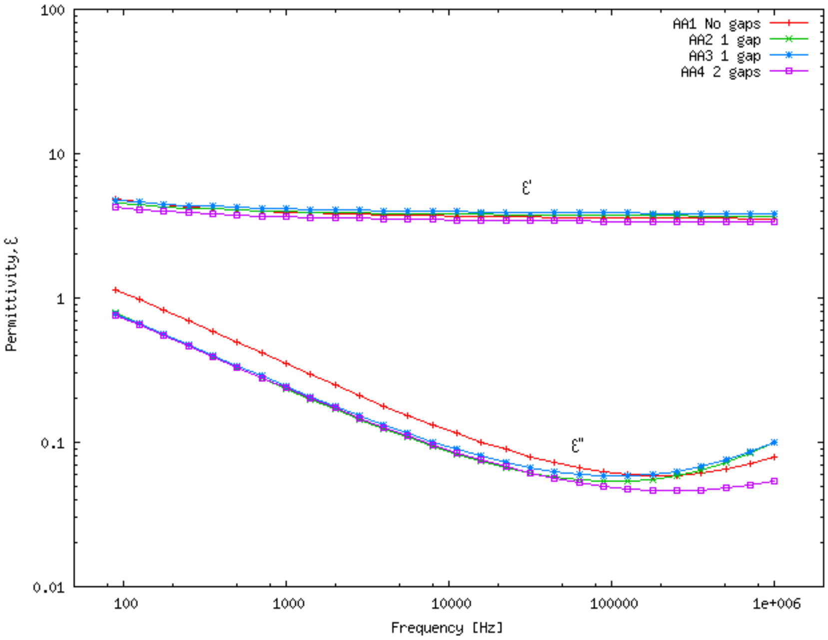

Figure 5.8: Comparison of samples created of paper strips at 60°C. The whole paper test is also included

Comparison of the Samples Without Gaps

Figure 5.9 show dielectric frequency measurements for the Alpha-Analyser for samples *without gaps*. Considering the middle layer of the test samples. The AA1 test has a new piece of clean paper, AA6 and AA8s middle layer had been grown for five days, while AA7 had a middle layer grown for seven days. At frequencies below 10^4Hz test AA1, AA7 and AA8 follow the same curve, while the AA6 test has some lower loss characteristic. For higher frequencies the AA7 and AA8 sample has a rapid increase while the two remaining samples continue to decrease up to around $2.5 \cdot 10^4$Hz. At this point the AA8 loss plot coincides with the AA1 and AA7 plots. The two samples grown for five days,AA6 and AA8, shows some large deviations between each other. These test where grown for the same time periode. AA8 was created three weeks after AA6.

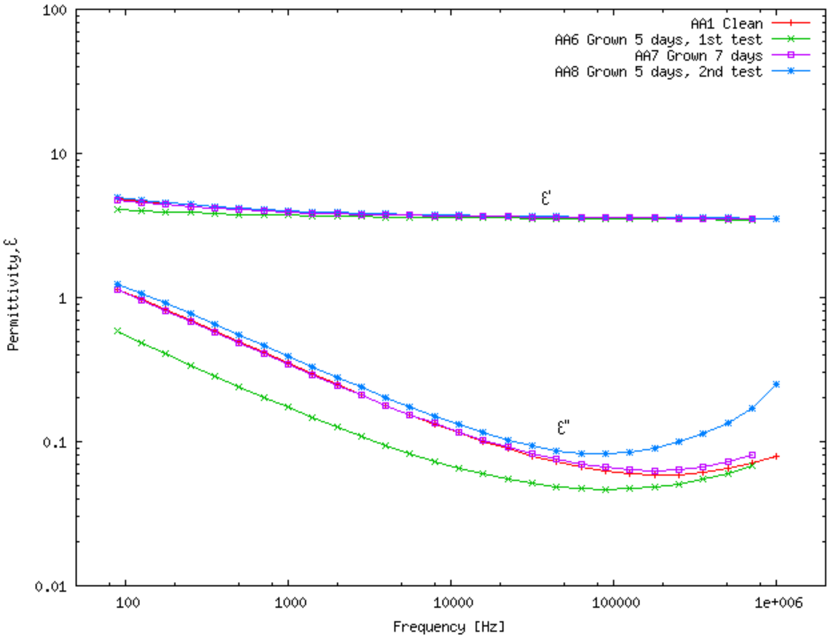

Figure 5.9: Comparison of paper samples grown in the laboratory. The dielectric frequency measurement were performed at 60°C for all samples in the figure. The test with clean paper layers, AA1, is also included

5.3.4 Comparison Between High and Low Frequency Range

The comparisons are divided into two graphs to give a better presentation of the results. Figure 5.10 show the varations between no gaps and gaps for the entire frequency range. None of the samples in this figure is contaminated with copper sulphide. In figure 5.11 the comparison between samples of strips with and without contamination is shown. The IDA2 test is represented in both figures to give the possibility of comparison between the two cases.

Figure 5.10 shows the readings for the whole and clean samples, IDA1 and AA1, and for clean samples of strips, IDA2 and AA2. For the IDA1 test the results for both the 200V and 100V measurement are shown. As seen, the IDA1 and AA1 reading coincide well, for both ε' and ε''. Comparison between the IDA1 and IDA2 shows that the gap influence causes an increase in losses for the lower frequency range. At the higher frequency range the effect is the opposite as AA2, with gaps, has a lower loss value compared to the AA1 sample. This results in a mismatch between the IDA2 and AA2 loss readings.

In figure 5.11 all test samples containing gaps are shown for both frequency ranges. As also seen in figure 5.10 there is a gap between the two frequency ranges. IDA3 and AA3 are the only samples contaminated with copper sulphide.

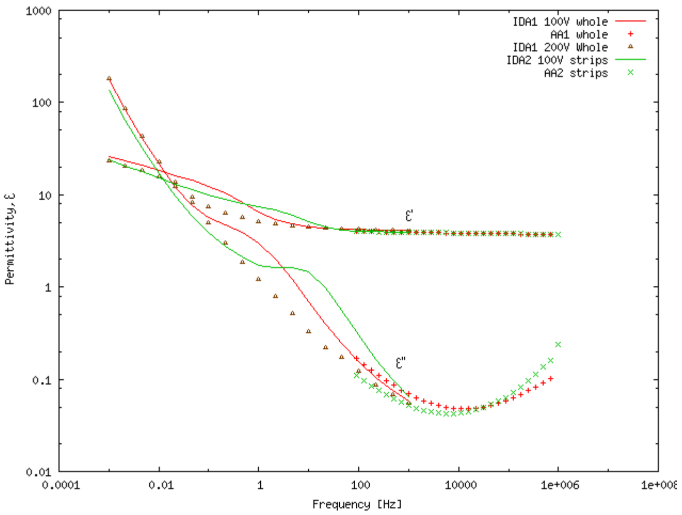

Figure 5.10: Comparison of the whole paper and the stripped paper tests, performed with both Insulation Diagnostic Analyser and Alpha-Analyser. Test IDA1, IDA2, AA1 and AA2.

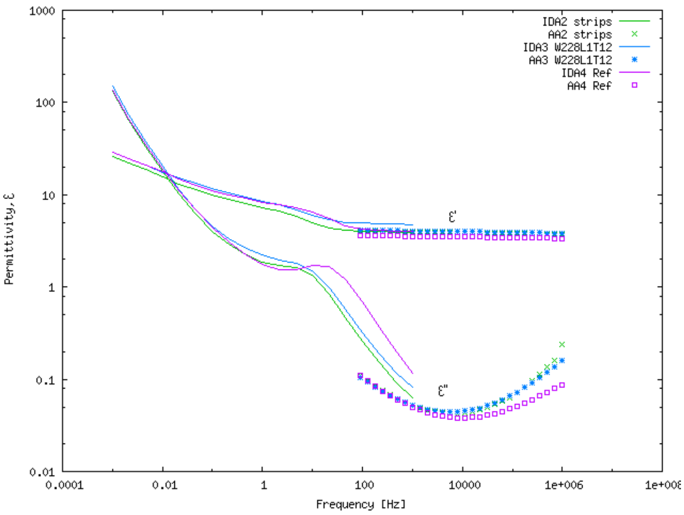

Figure 5.11: Comparison of all tests created of strips, performed with both Insulation Diagnostic Analyser and Alpha-Analyser. Test IDA2–4 and AA2–4.

5.3.5 Test AA5: Three Layers of Strips with High Contaminated of Copper Sulphide

The AA5 test had all three layers highly contaminated with copper sulphide. All information about this sample is to be found in appendix A.11. Since the paper used was taken from the Skagerrak transformer all layers had one gap. To remove the possibility of direct contact between the electrodes the layers were created with the gaps as far from the center as possible. Figure 5.12 shows the build up of the sample in question. The buildup shows that there are only two gaps crossing each other in the same point. As this is the only sample where all layers were contaminated, direct comparison with the rest of the tests is not performed. Considering the dielectric frequency reading in figure 5.13. The permittivity loss curve is linear and stretches from a value of 2–8 at 1MHz to a value of 20000–50000 at 100Hz. The real permittivity has a constant value of around 10. There is a clear increase in losses with increasing temperature, however the slope stays the same.

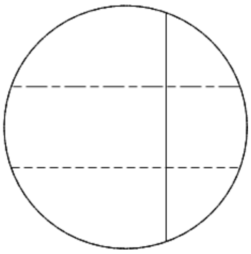

Figure 5.12: The buildup for the sample with three layers of contaminated paper. The lines indicate the position of the gaps for the three layers

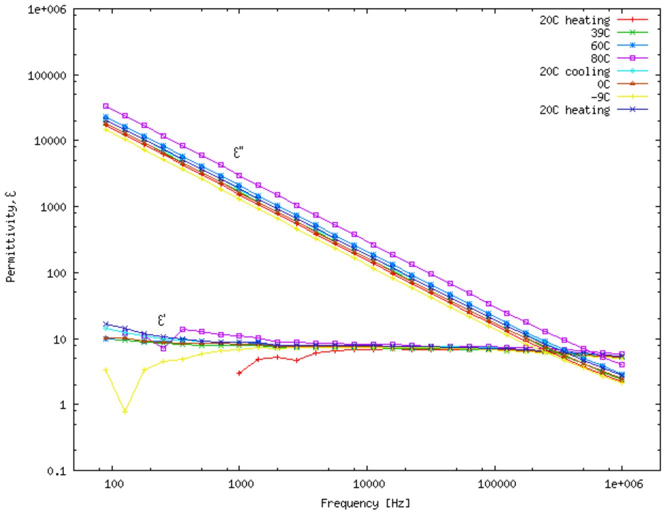

Figure 5.13: The dielectric frequency measurement for the AA5 test

CHAPTER

SIX

DISCUSSION

The discussion chapter is divided into Sample Discussion and Dielectric Frequency Measurement Discussion. The Sample Discussion section covers the discussion around the sample creation and documentation. The Dielectric Frequency Measurement Discussion section discusses the differences in dielectric frequency response, and cover the discussion of the two frequency ranges individually and together. Significant results for single test samples are also included.

6.1 Sample Discussion

6.1.1 Sample Creation

Creation from paper strips

Sample created of strips was a pretty easy way of making samples. Large amount of samples could be made in a short time period. The main problem came with the remaking of additional samples with fairly the same contamination amount. From transformer winding inspections the contamination showed substantial differences along the windings. Finding new samples with fairly the same contamination became difficult and time consuming.

Sample with grown copper sulphide contamination

As shown in figure 5.3 in the result chapter, there are a great deal of visual differences of the contamination, but the variations are smaller from the resistance measurements. Additional examples are to be found in appendix A.15. The general growth of copper sulphide turned out to work well. Also controlling of the contamination by days of growth worked for most of the cases. The samples grown for three days had just some insulation reduction at the surface. This was only seen at the side facing toward the copper cylinder. With two days of additional growth the contamination had increased a lot and reductions in withstand strength were on both sides of the sample. From five days to seven days the contamination degree seemed to stagnate some, with almost the same degree of contamination. For the sample grown for eleven days the contamination had increased and given strong reductions in both surface resistance and resistance through the paper. Comparison between this sample and the one grown for fourteen days shows that the eleven days test actually has a heavier contamination than the one grown for three more days.
From these results there seem to be a clear correlation between time and degree of contamination. However the correlation varies and the number of days between the samples should be large enough

to cope with these variations. In addition, due to the large variations, conclusions should not be done by this method alone.

6.1.2 Sample Documentation

Resistance measurements

For the samples seen in figure A.6, A.17,and A.22, the resistance measurements through the paper gave different values depending on the side of measurement. A cause for this phenomenon can be in the measurement platform, shown in figure 3.7, in combination with the Cu_2S contamination characteristics. The measurement platform in figure 3.7 shows a fairly small low potential electrode only covering a fraction of the test sample. The high potential electrode, however, is large and cover the most of the test objects. Considering the samples mentioned above, all have one side of fully covered copper sulphide. Hence has a high surface conductance[1]. The other side had a more spotted Cu_2S layer, and lower surface conductance. Figure 6.1 shows an illustrations of resistance measurements through the paper, for one of the discussed samples. The black lines indicates conductive copper sulphide, meaning that current can move freely on these lines. From the measurements on the evenly contaminated side of the samples, it can be assumed that the entire side has a fairly high conductance. Taken this to account an electrode in connection with this side, has connection to the total sample surface, not just there area in contact with the electrode. The illustration to the left in figure 6.1 shows the evenly contaminated side in connection with the small electrode. Assuming that the entire side is conductive, the whole side will be connected to this electrode. The measurement area then becomes the entire surface area of the sample tested. The side of the paper facing downwards is in contact with the larger electrode. Since this electrode is much larger than the paper sample, the entire sample has connection. This means that all areas were copper sulphide have grown through the paper have contact with both electrodes.
Considering the illustration to the right, the side with spotted contamination is turned upwards. In this case some of the copper sulphide areas are not in connection with the small electrode. The areas outside are not able to conduct any current. In this case the measurement area becomes the small electrode. Combining the two cases the connection illustrated to the left has a larger cross-section of contaminated paper and gives a lower resistance. As the test cells have contact with the entire surface on both sides of the paper the total resistance of the sample can be even lower.

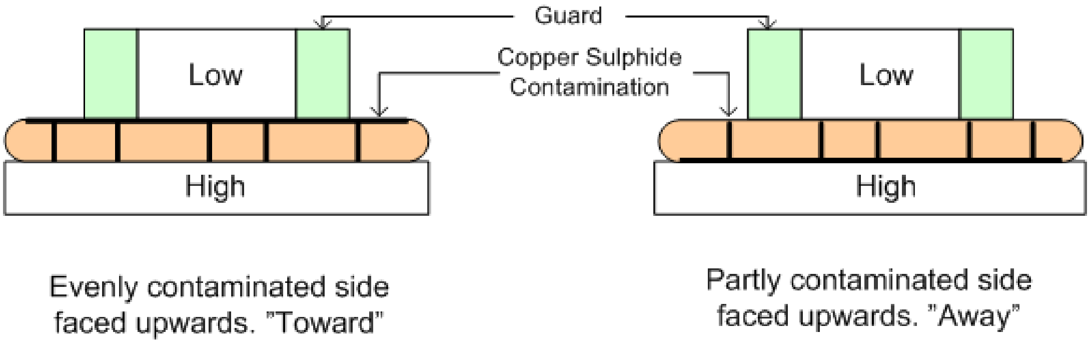

Figure 6.1: Illustration of resistance measurements through a contaminated sample. The sample has one side entirely covered with copper sulphide and some areas with contamination grown through the sample. The figure shows testing on both sides of the sample

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[1] Conductance equals to Resistance^{-1} and has the denomination Siemens,$[S] = [\Omega^{-1}]$

Area percentage documentation

The area percentage documentation was carried out with the processing program ImageJ. The program used differences in brightness to differ copper sulphide from paper. This method worked best for the away side of the samples: IDA3, AA3 and AA5. These three samples all have copper sulphide grown through the paper, with the same silver colour. The area percentage calculations are represented in appendix figure A.7, A.18 and A.23. As the program used the brightness level of pixels to calculate the amount of copper sulphide the contamination needed to be fairly similar over the area. In addition the brightness level between the paper and contamination had to be large enough for ImageJ to detect it. Seen in figure 5.3 the copper contamination can come in both dark colour, bluish silver and light, almost transparent, layers. These types of contamination are far to complex for the ImageJ software, and the program will not be able to separate the copper sulphide form the paper.

Water percentage measurement and calculation

The water percentage has been calculated by using the equation 4.1 and the measured weights in appendix A.1. As all tests have been exposed to air humidity at the time of testing, some variations are normal. However, as seen in the appendix A.1[2], most of the tests contaminated with copper sulphide have a lower water percentage, than the samples with no contamination. Considering the most contaminates samples, IDA3, AA3 and AA5, these samples also have a relative high weight compared to the clean samples.The middle layer for all the Alpha-Analyser samples were of the same size. In addition the entire layer were measured for both the water amount and the weight of the dry sample. Hence there should be little weight differences between these samples. However, as stated earlier, the weight for the AA3 and AA5 tests are considerably higher than the rest of the samples. The AA3 and AA5 weights are respectively 13% and 30% higher than for the clean test sample AA1. There are reasons to belive that the acctual water amount is fairly similar for all samples, but since the weight is higher for the most contaminated samples, the percent amount of water gets reduced. As a function to this the water percentage has different starting point and should just be used as a guidence.

Also for the reference samples, IDA4 and AA4, the water percentage is a little lower than expected. According to article[7] moistened pressboard samples had lower water percentage after aging than before, even when seal the entire time. The article suggests that during the aging process of the cellulose, some amount of water have been bounded to the paper and cannot be titrated out. However this amount of water may still give a contribution to the dielectric frequency measurement. Since pressboard is made of compressed paper strips this phenomenon may also apply for old paper strips. As the reference samples are almost 40 years old, there might be some amount of bound water in the paper.

6.2 Dielectric Frequency Measurement Discussion

6.2.1 Verification of the Systems

Refer to the IDA1 test in figure A.3. The dielectric frequency response for the whole and clean papers coincide well with previous work. Figure 6.2 shows dielectric responses for moistened paper from earlier work done with the Insulation Diagnostic Analyser[6]. The graph shows responses for paper with different humidity degrees. As seen in the figure the responses shifts to higher frequencies with increasing water content. In addition a hump starts to show at a higher degree of humidity. For the voltage levels $100V$, $50V$, and $25V$, in figure A.3, the response slopes coincide best with the 4% humidity paper measurement. The graphs are shifted in some degree toward higher frequencies and

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[2] Samples in bold are contaminated by copper sulphide

the hump is wider for the IDA1 test results. This correlates well with the previous work as the IDA1 test has a water amount of 5.5%.

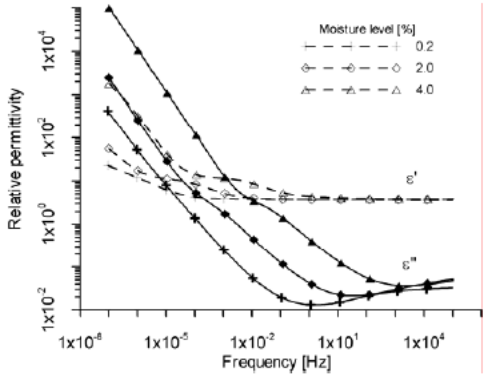

Figure 6.2: Earlier dielectric frequency measurements done with the Insulation Diagnostic Analyser. The graphs show the responses for clean paper with different humidity. The figure is taken from Article [6]

6.2.2 Lower Frequency Range: 1mHz–1kHz

Considering figure 5.6 and 5.7. The graphs indicate an increase of permittivity losses with increasing number of gaps. The sample with five gaps has the highest permittivity losses, and has its peak at around 50Hz. It is important to notice that the permittivity loss graphs also contains the conductivity losses. See chapter 2.1.2 for a deeper theoretical explanation. Due to this the permittivity loss peak is defined as the point of the graph where the slope is at its lowest. For the samples with four gaps the peak is at about the same value and has its peak at around 8Hz. For the whole paper sample, with no gaps, the peak occurs at around 1Hz. The value is a little higher than for the other cases, but this could be due to increasing conductivity losses. The $200V$ reading for the whole paper sample is also considered. The slope is significantly lower than for the other samples and do not have a peak. This might actually be the most exact permittivity reading for this test sample. According to Ekanayake's PhD Thesis [5], dielectric frequency responses for paper have been given the same slope as the $200V$ reading for the IDA1 test. The humps present for the other test might be due to interfacial polarisation, as explained in chapter 2.1.2. Considering the $200V$ reading for the IDA1 test and assuming that the connection between the paper layers and the electrode is good. The interfacial polarisation contribution is most likely small, as the surface area between paper and oil is small. If there is poor connection between the layers and electrodes, congestions of oil may appear in the insulation. This gives larger surface areas for free charges to follow along when the electrical field varies. If one of the layers contains of strips the area may get even larger.

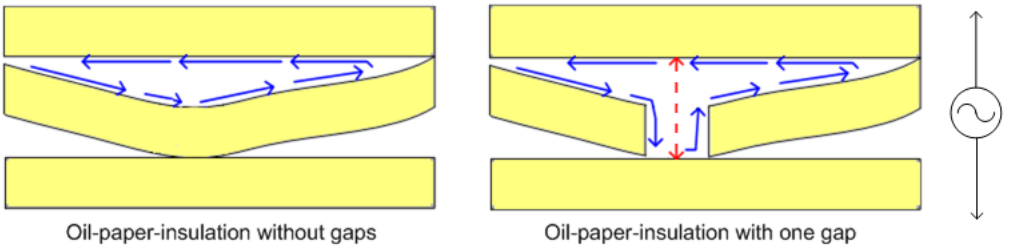

Figure 6.3: Interfacial polarisation in paper-oil-insulation with and without gaps. Solid-drawn arrows indicate the free charges following the paper surface. The dotted arrow shows the interfacial polarisation contribution that skips the paper layer due to the gap.

Figure 6.3 considers the two cases with poor connection between the paper layers. The electrodes are not included in the illustrations. The illustration to the right shows the insulation without gaps, while the illustration to the left has on gap in the middle layer. Considering the left illustration; the solid arrows indicate the interfacial contribution of the oil as the electric field alternates. The interfacial polarisation in the paper not included. For the figure to the right, there is a gap in the paper sample. The interfacial polarisation between paper and oil gives a contribution, shown with solid arrows. In addition the dotted arrow indicates a contribution to interfacial polarisation that skips the paper layer with the gap and goes directly to the next paper layer. The interfacial polarisation will contribute to the real permittivity as long as the frequency is low enough for the free charges to follow. With increasing frequency there will come a point where the free charges no longer can follow the changes. This gives permittivity losses as the interfacial movement is lagging the field. At high enough frequencies the free charges do not have time to move at all and will not contribute to any permittivity losses.

6.2.3 Higher Frequency Range: 100Hz–1MHz

The tests performer with the Alpha-Analyser are compared in figure 5.8 and 5.9 in the result chapter. As in the result chapter the samples with and without gaps are discussed separately.

6.2.4 Discussion and comparison of samples with gaps

Considering the figure 5.8, the AA2 and AA3 tests follow the same curve over the entire frequency range. Both of these had one gap in the middle paper layer. While AA2 contained of new and clean paper, the AA3 was highly contaminated with copper sulphide. From this there seems to be a fairly little contribution to the permittivity response from copper sulphide.

6.2.5 Discussion and comparison samples without gaps

The gaps turned out to give a significant effect in the permittivity response. The samples without gaps then become the most reliable source for finding a possible copper sulphide effect to the dielectric frequency response. The grown samples are the only samples without gaps and with copper sulphide contamination. The comparison between these samples are shown in figure 5.9 stated in the result chapter. The samples compared are: AA1–clean paper, AA6 and AA8–samples grown for five day, and AA7–sample grown for seven days. The resistance measurement for the contaminated samples, placed in appendix figure A.25, A.27 and A.29, showed small variations in contamination due to days of growth. Still, however, the dielectric frequency response for the grown samples differed a great deal. The sample grown for seven days followed the clean samples in the entire frequency range, and indicates that copper sulphide gives no effect to the dielectric frequency response. The AA8 test, grown for five days, shows the same progress for frequencies up to 500kHz, and then has a rapid increase in permittivity losses. The last sample gave a much lower loss characteristic from 100Hz to its minimum value at 100kHz. Then the permittivity losses increased and coincide with the graphs for the whole papers. The low loss characteristic for the AA6 sample is the main reason for the running of an additional five days test. Still the AA6 test has the same rapid increase as the AA8 test.

All grown samples has the heaviest contamination around the edges. This creates a rim of fairly good conductivity. It is important to state that the insulation degradation also happened at the inner part of the samples. In addition the contamination has significant variations around the edge. Either way, the weakest point for the insulation papers seems to be at the edge of the samples. At the edge a stray field will most likely occur, giving an electrical potential in the radial direction. As the surface is more conductive this may give rise for more freely moving charges at the edge.

As the results differs a lot, a solid conclusion can not be drawn at this point. However the variations can be an indication that there may be a correlation between the copper sulphide contamination and the dielectric frequency response. There is certainly a need for more research to get a stronger picture of the relation between copper sulphide and permittivity response at higher frequencies.

6.2.6 The Whole Frequency Range

The comparison between the high and low frequency range are stated in figure 5.10 and 5.11 stated in the result chapter. For the sample containing whole paper layers only the frequency ranges overlaps good and this applies for both of the voltage reading in the low frequency range. The 100V and 200V level for the IDA1 test connects good to the AA1 test giving an solid-drawn curve for the entire frequency range. As for the stripped samples the matching is not that good. The samples containing gaps at the lower frequencies had a higher permittivity loss compared to the whole paper samples. At the higher frequencies the gaps seems to cause a loss drop in respect to the AA1 sample. The result was a distinct difference between the curves for the low and high frequency range. This is not that unexpected as the sample preparation for the two frequency ranges are quite different. As the stripped sample at the lower frequencies contains four gaps, the stripped samples in the higher frequency range only contains one. Since it has been observed that a gaps give an effect to the permittivity response the samples may be too different for results comparison.

6.2.7 Test AA5: Three Layers of Strips with High Contamination of Copper Sulphide

The dielectric frequency measurement for the test with three contaminated layers are shown in figure A.24. As stated in chapter 5.3.5 the permittivity loss is linear and reaches a value of around 50000 which is more than ten thousand times higher than for any of the other Alpha-Analyser test readings. There is a strong reason to believe that there has been conductive connection between the electrodes. However, as the resistance measurement in figure A.22 shows, the resistance through the paper is quite high. Despite the heavy contamination on the one side. Still, the gaps between the paper might be enough as the surface of all three layers has a fairly good conductance. Figure 6.4 shows an illustration of a current path made possible because of the surface layer of conductive copper sulphide. The current can travel relatively easy on the surface, neglecting the insulation contribution of the paper. The electrical withstand strength between the electrodes is, in this example, reduced to the areas of oil between the paper layers, shown as dotted circles in the figure. This give the current an almost resistance less route and the conductive part of the permittivity losses dominates.

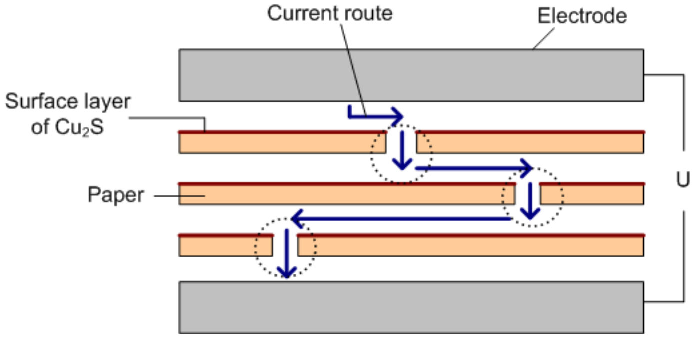

Figure 6.4: The current path created due to gaps and surface contamination

CHAPTER

SEVEN

CONCLUSION

The conclusion chapter sums up the most important discoveries found during the work of this master thesis. In addition suggestions to future work in this field is included.

- For sample creation the growth of copper sulphide samples seemed to be the better choice as the gap influence is canceled. However the growth may vary in some extent and days of growth can be insufficient in classifying the degree of copper sulphide. The contamination was heaviest at the edge of the samples. Good connection between the copper and paper may be an important factor, and the size of the samples is recommended to be kept small.

- For contamination classification the resistance measurement method seemed to work best. The method is precise and can cope with large variations in contamination. The area percentage calculation method worked under very limiting conditions as the contamination had to be of a certain brightness level and low variations in colour. The classification due to days of growth worked well for most cases. However, the degree of contamination did not always correlate with days of growth. Larger time intervals can be a solution.

- The gaps between the strips used in the samples, gave a substantial effect to the dielectric frequency response. This effect was clearest at the lower frequencies, but some effect is also detectable in the higher region. At the lower frequencies the results gave no indication of any effect in the dielectric response due to copper sulphide. However, the gap effect may have concealed the effect, and testing without gaps is recommended.

- At the higher frequencies the grown copper sulphide samples gave some variations in the dielectric frequency response. The variations in results also occurred between samples with the same creation conditions. This may still indicate a relation between copper sulphide and dielectric response at higher frequencies. Even with the large deviations there were, at frequencies above 100kHz, a steeper increase in losses for the contaminated samples. Only tests grown for five and seven days were tested. The discoveries has given a path to follow, but, as the research foundation is low, more research is needed.

Future Work

In general there might be a correlation between copper sulphide and dielectric frequency measurement. Growing samples as turned out to work well as the test object provider. By combining days of growth with resistance measurements the classification of the contamination should be sufficient enough. However, more research is absolutely needed and some suggestions for further work are as follows:

- Dry the samples to minimise the water effect on the dielectric frequency measurement. This may also give the possibilities of master curves in a lower frequency range. Hence a new sample cell for the low frequency range may not be necessary.
- Creating a new smaller sample cell for the Insulation Diagnostic Analyser, so the sample size can be reduced for the lower frequency range. This might also give a better permittivity readings in general.
- Increase the time gap between the samples grown to get larger differences in contamination, and reduce the effect of variations. For instance: Clean paper–three days–six days–ten days. As fourteen days resulted in very high degradation, longer growing time may be difficult.
- Reducing the temperature may be a way to increase days of growth. However, in this case more research on the temperature dependencies of copper sulphide growth is needed.

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APPENDIX

A

APPENDICES

A.1 Data for Water Percentage Calculations

	Weight measurements,[mg]			
Test No.	*Water*	*Paper and Container*	*Container*	*Water percentage*
IDA1	37.9	2.299	0.142	5.4 %
IDA2	45.9	2.663	0.162	5.2 %
IDA3	**55.3**	**2.804**	**0.162**	**4.6 %**
IDA4	**28.6**	**1.459**	**0.162**	**4.3 %**
IDA5	**53.5**	**2.496**	**0.165**	**4.2 %**
AA1	22.5	1.189	0.103	4.6 %
AA2	22.0	1.361	0.140	5.3 %
AA3	**25.4**	**1.137**	**0.209**	**3.5 %**
AA4	13.9	0.836	0.192	4.4 %
AA5	**29.3**	**1.288**	**0.250**	**3.4 %**
AA6	**21.8**	**1.012**	**0.157**	**3.8 %**
AA7	**21.8**	**1.059**	**0.161**	**4.0 %**
AA8	**21.9**	**1.147**	**0.161**	**4.3 %**

Figure A.1: Water measurement documentation. Tests marked with bold text are contaminated with copper sulphide. For the Alpha-Analyser the entire sample was measured, while for the Insulation Diagnostic Analyser only one strip was tested in the Karl Fisher.

A.2 Test IDA1: Verification Sample

Sample name:	Test IDA1
Sample material:	New Termo70 paper
Sample dimensions[a]**:**	Circular. Diameter of 75mm
Equipment used for DFM[b]**:**	Insulation Diagnostic Analyser
Test frequency range:	1mHz – 1kHz
Other tests performed:	Resistance measurements
Water percentage:	

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[a] Only middle layer considered

[b] Dielectric Frequency Measurement

Figure A.2: IDA1: Test sample. The surround layers are also included

The resistance readings gave a value of at least $200 G\Omega$ for all measurements.

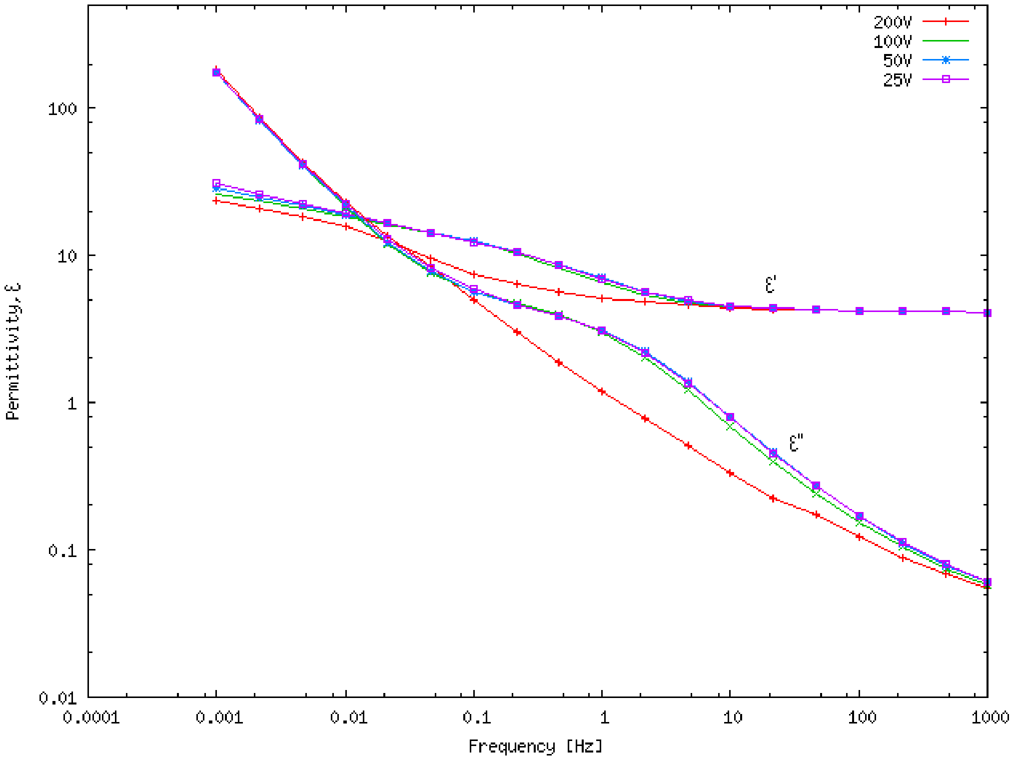

Figure A.3: Test IDA1: Result of the dielectric frequency measurements. Verification of the system

A.3 Test IDA2: Sample Created of Strips

Sample name:	Test IDA2
Sample material:	Strips of new Termo70 paper
Sample dimensions[a]**:**	Five strips forming a circular sample with diameter of 75mm
Equipment used for DFM[b]**:**	Insulation Diagnostic Analyser
Test frequency range:	1mHz – 1kHz
Other tests performed:	Resistance measurements

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[a] Only middle layer considered

[b] Dielectric Frequency Measurement

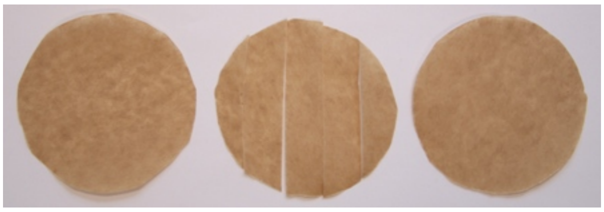


Figure A.4: IDA2: Test sample.

The resistance readings gave a value of at least $200G\Omega$ for all measurements.

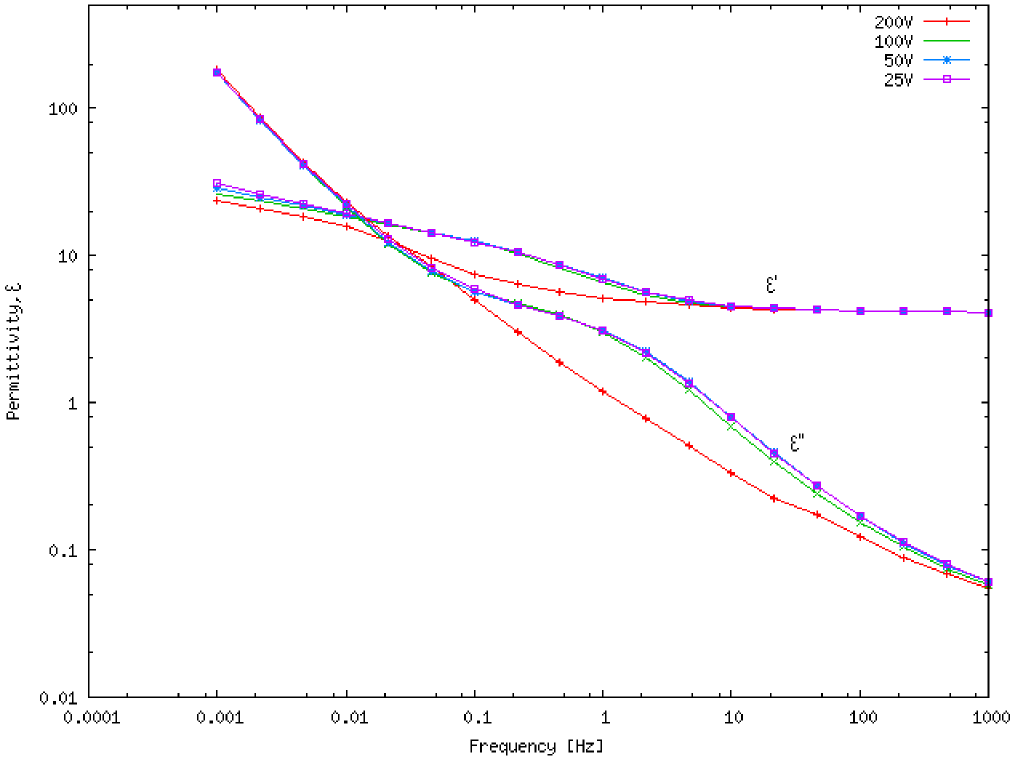


Figure A.5: Test IDA2: Result of the dielectric frequency measurements.

A.4 Test IDA3: Sample Created of Strips with High Copper Sulphide Contamination

Sample name:	Test IDA3
Sample material:	Paper strips taken from the Skagerrak transformer. Winding 228 from the bottom. Turn number 12. Paper layer in contact with the copper conductor.
Sample dimensions[a]:	Five strips forming a circular sample with diameter of 75mm. Total diameter of 75mm. Strip width of 15mm
Equipment used for DFM[b]:	Insulation Diagnostic Analyser
Test frequency range:	1mHz – 1kHz
Other tests performed:	Resistance measurements and area percentage calculations

---

[a] Only middle layer considered

[b] Dielectric Frequency Measurement

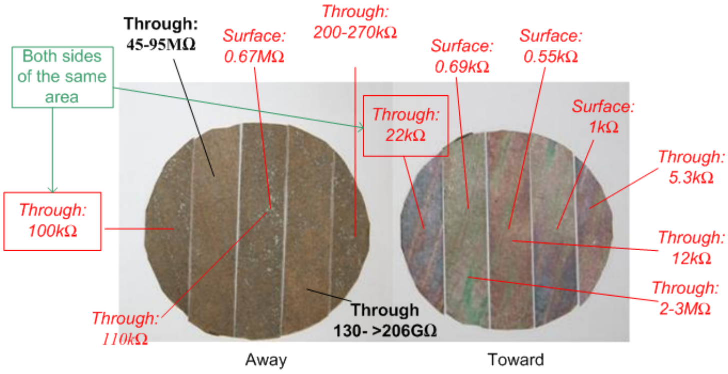


Figure A.6: IDA3: Test sample. Red and italic - Multimeter, Black and bold - Megger at 200V

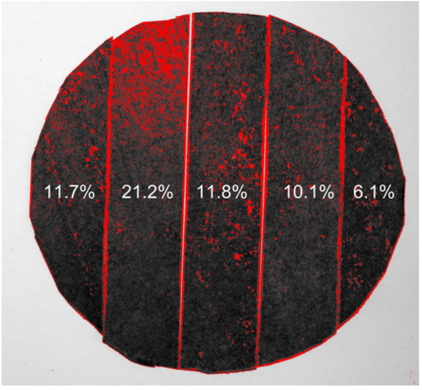


Figure A.7: Test IDA3: Area percentage measurement and calculations

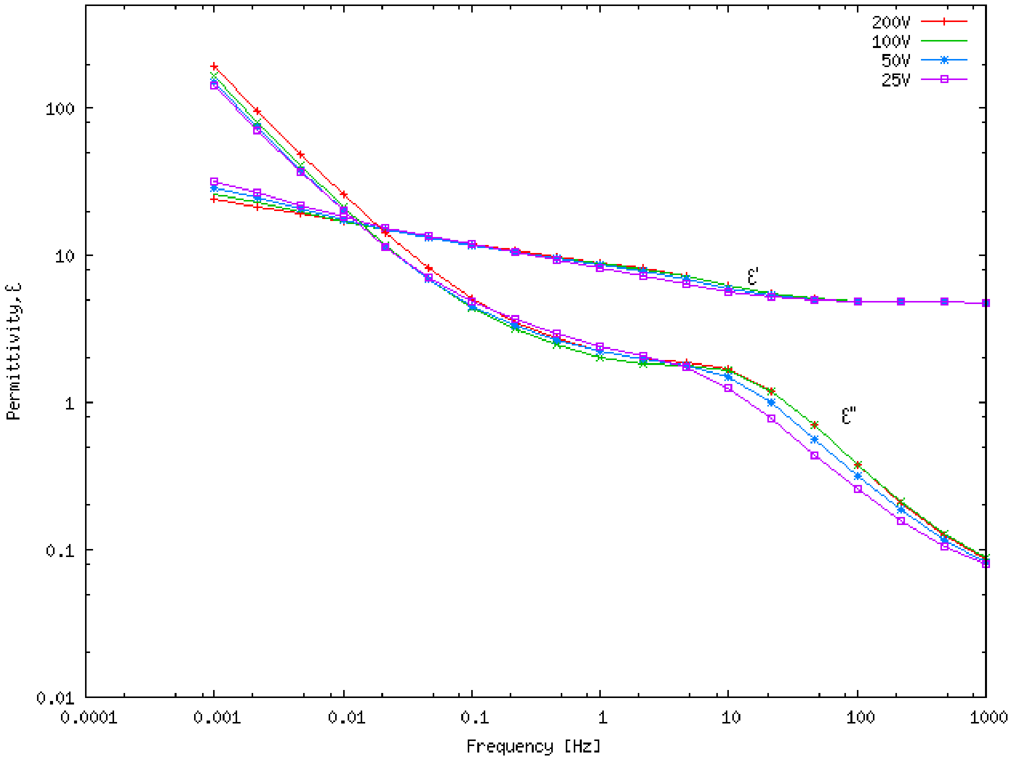


Figure A.8: Test IDA3: Result of the dielectric frequency measurements.

A.5 Test IDA4: Sample Created of 40-Year-Old Paper with no Cu_2S Contamination

Sample name:	Test IDA4
Sample material:	Paper strips taken from Sira-Kvina transformer. Winding 2 from the bottom. Turn 6. Paper layer in contact with the copper conductor.
Sample dimensions[a]**:**	Circular. Diameter of 75mm
Equipment used for DFM[b]**:**	Insulation Diagnostic Analyser
Test frequency range:	1mHz – 1kHz
Other tests performed:	Resistance measurements

---

[a] Only middle layer considered

[b] Dielectric Frequency Measurement

The resistance readings gave a value of at least $200G\Omega$ for all measurements.

Figure A.9: IDA4: Test sample.

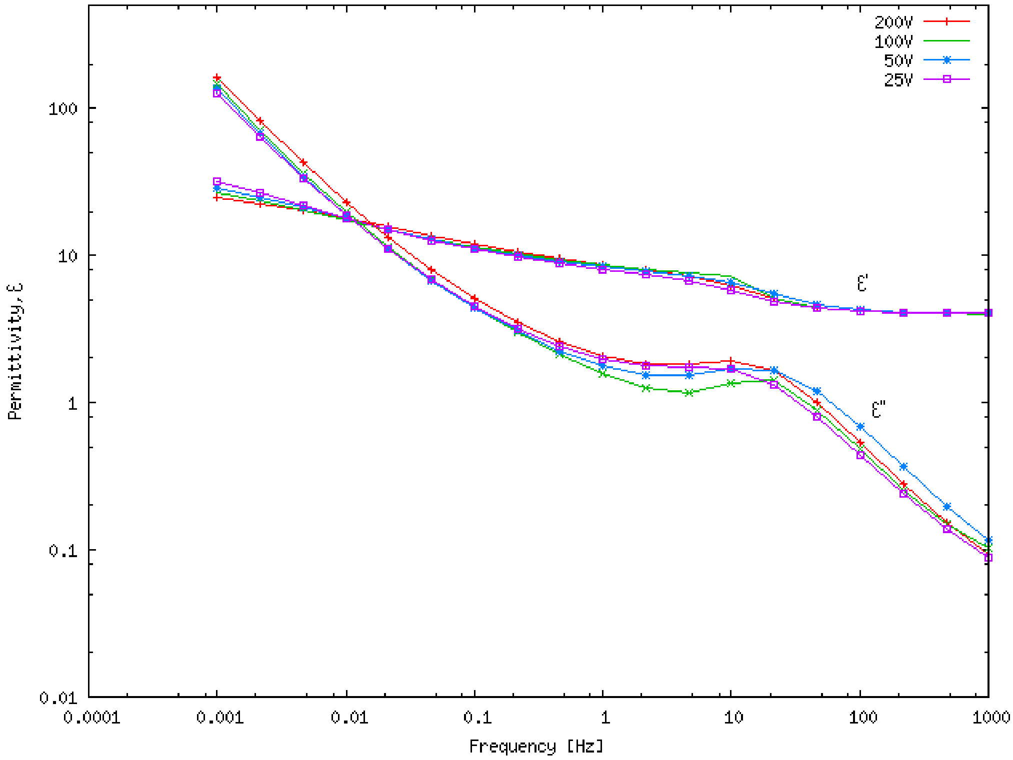


Figure A.10: Test IDA4: Result of the dielectric frequency measurements.

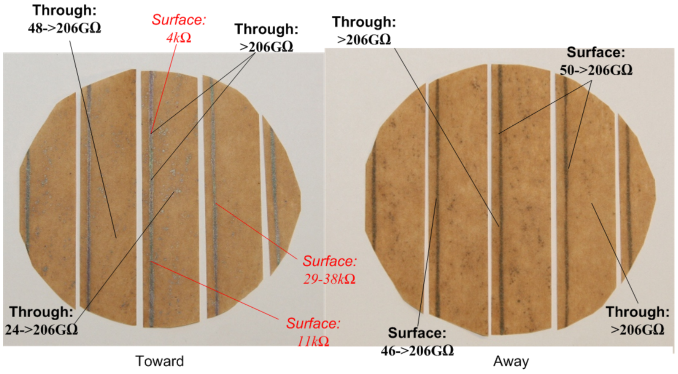
A.6 Test IDA5: Sample Created of Strips with Moderate Copper Sulphide Contamination

Sample name:	Test IDA5
Sample material:	Paper strips taken from the Skagerrak transformer. Winding 228 from the bottom. Turn number 12. Second paper layer from the copper conductor.
Sample dimensions[a]**:**	Five strips forming a circular sample. Total diameter of 75mm. Strip width of 15mm
Equipment used for DFM[b]**:**	Insulation Diagnostic Analyser
Test frequency range:	1mHz – 1kHz
Other tests performed:	Resistance measurements

---

[a] Only middle layer considered

[b] Dielectric Frequency Measurement



Due to large variations in brightness on both paper and Cu_2S contamination, the use of area percentage calculation was not feasible.

Figure A.11: IDA5: Test sample. Red and italic - Multimeter, Black and bold - Megger at 200V

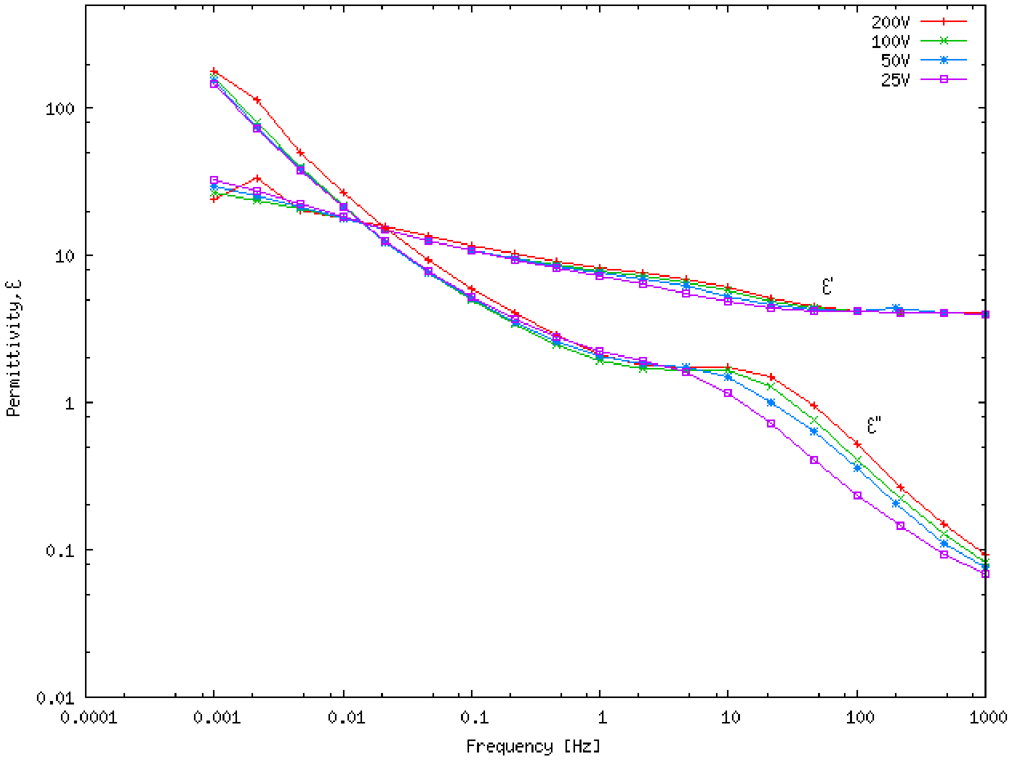


Figure A.12: Test IDA5: Result of the dielectric frequency measurements.

A.7 Test AA1: Verification Sample

Sample name:	Test AA1
Sample material:	New Termo70 paper
Sample dimensions:	Circular. Diameter of 25mm
Equipment used for DFM[a]:	Alpha-Analyser
Test frequency range:	100Hz – 1MHz
Other tests performed	Resistance measurement

---

[a] Dielectric Frequency Measurement

Figure A.13: Test AA1: The test sample

The resistance readings gave a value of at least $200G\Omega$ for all readings

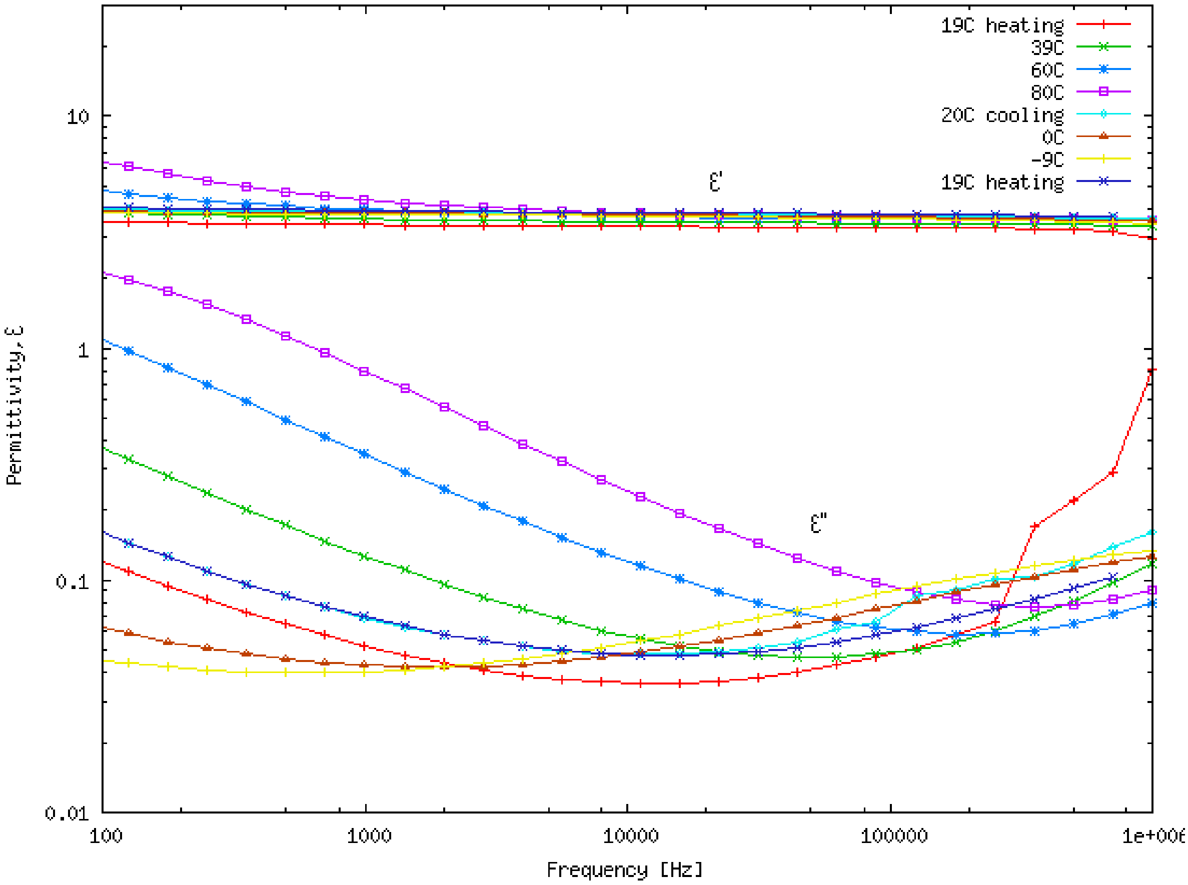


Figure A.14: Test AA1: Result of the dielectric frequency measurements.

A.8 Test AA2: Sample Created of Strips

Sample name:	Test AA2
Sample material:	New Termo70 paper
Sample dimensions:	Two strips forming a circular sample with diameter of 25mm
Equipment used for DFM[a]:	Alpha-Analyser
Test frequency range:	100Hz – 1MHz
Other tests performed	Resistance measurement

---

[a] Dielectric Frequency Measurement

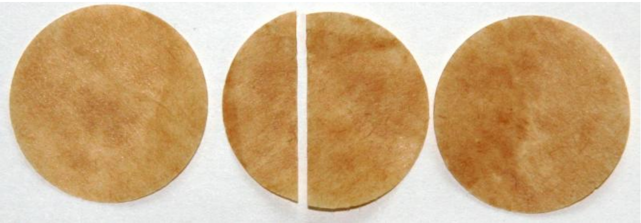


Figure A.15: Test AA2

The resistance readings gave a value of at least 200$G\Omega$ for all readings

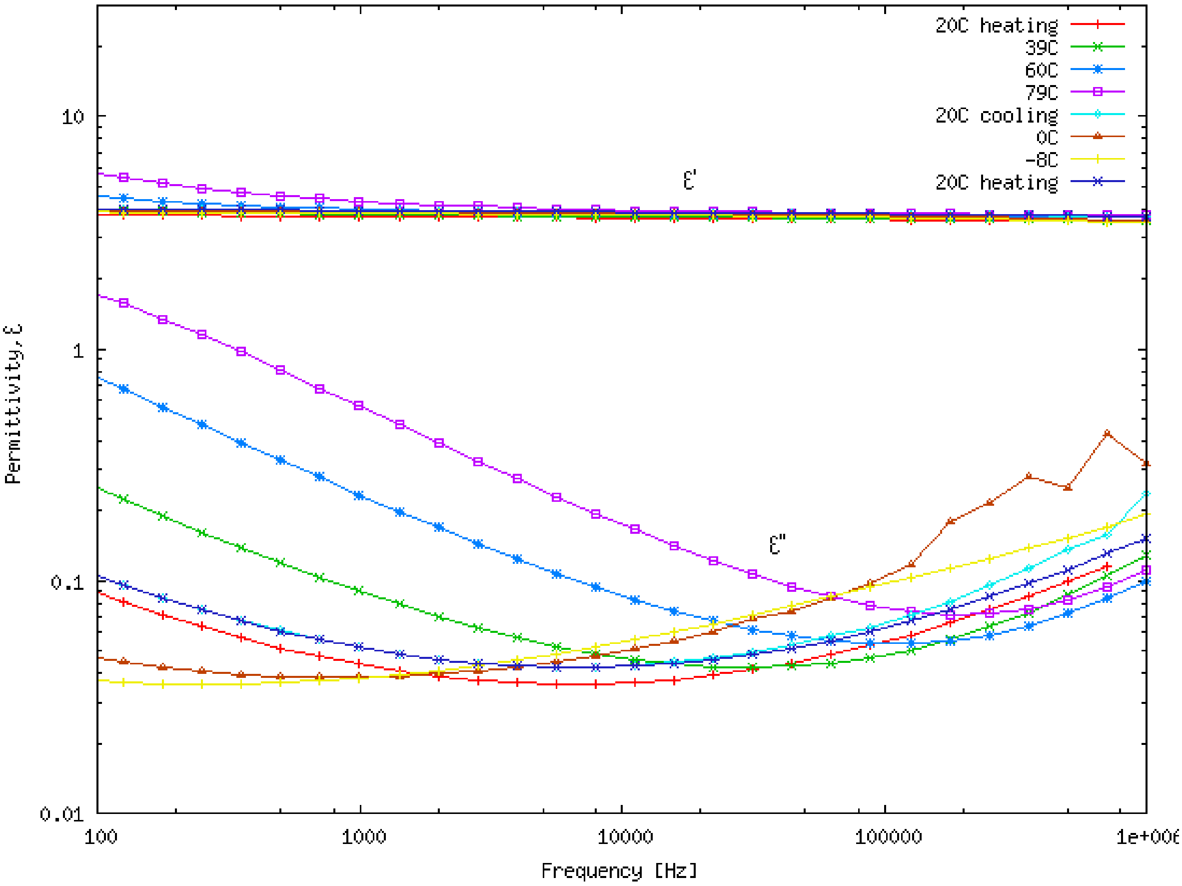


Figure A.16: Test AA2: Result of the dielectric frequency measurements.

A.9 Test AA3: Sample Created of Strips High Copper Sulphide Contamination

Sample name:	Test AA3
Sample material:	Strips of new Termo70 paper
Sample dimensions:	Two strips forming a circular sample with diameter of 25mm
Equipment used for DFM[a]:	Alpha-Analyser
Test frequency range:	100Hz – 1MHz
Other tests performed	Resistance measurement and area percentage calculation

---

[a] Dielectric Frequency Measurement

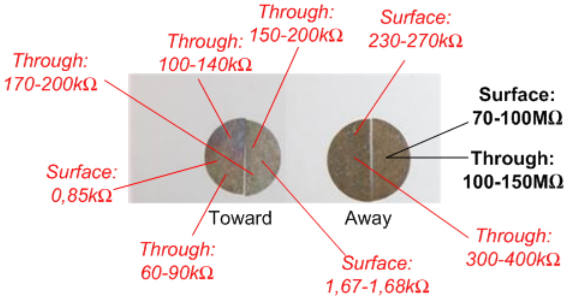


Figure A.17: Test AA3: Test sample. Red and italic - Multimeter,
Black and bold - Megger at 200V

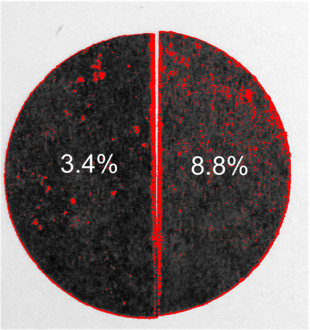


Figure A.18: Test AA3: Area percentage measurement and calculations

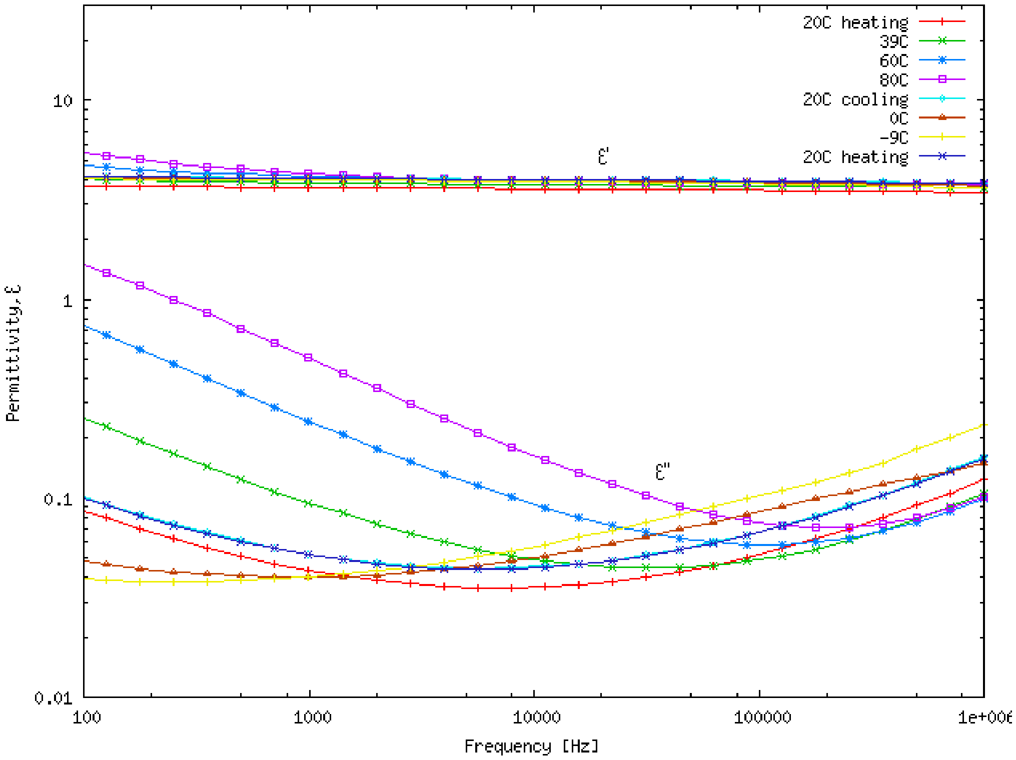


Figure A.19: Test AA3: Result of the dielectric frequency measurements.

A.10 Test AA4: Sample Created of 40-Year-Old Paper with no Cu_2S Contamination

Sample name:	Test AA4
Sample material:	Paper strips taken from Sira-Kvina transformer.
Sample dimensions:	Three strips forming a circular sample with diameter of 25mm
Equipment used for DFM[a]:	Alpha-Analyser
Test frequency range:	100Hz – 1MHz
Other tests performed	Resistance measurement

---

[a] Dielectric Frequency Measurement

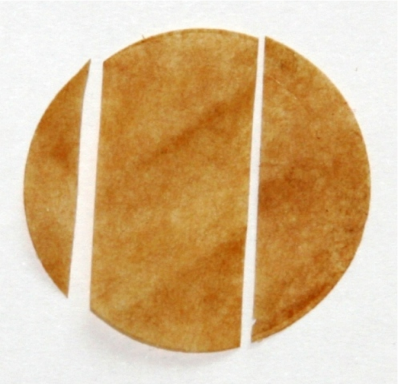


Figure A.20: Test AA4: Test sample

The resistance readings gave a value of at least 200$G\Omega$ for all readings

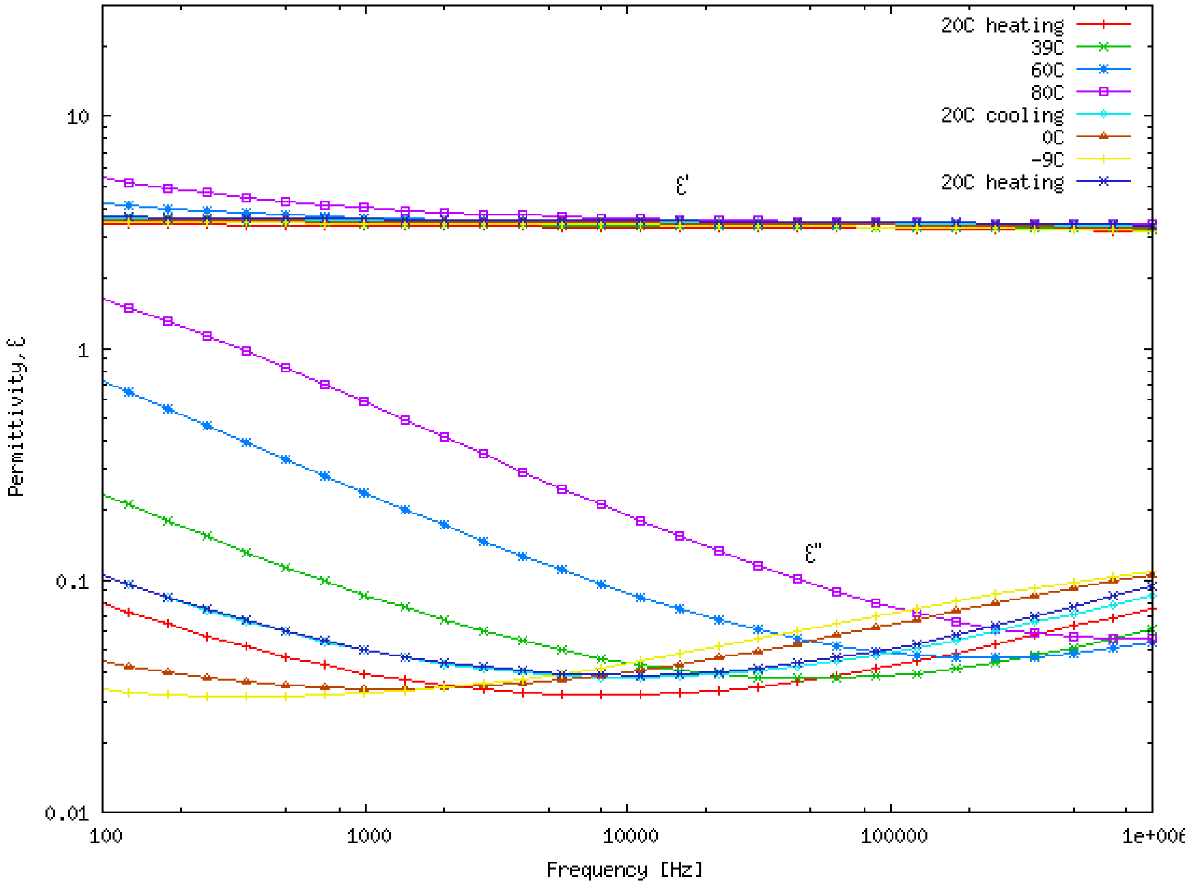


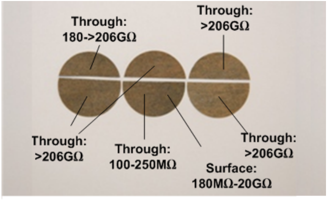
Figure A.21: Test AA4: Result of the dielectric frequency measurements.

A.11 Test AA5: Three Layers of Strips with High Contamination of Copper Sulphide

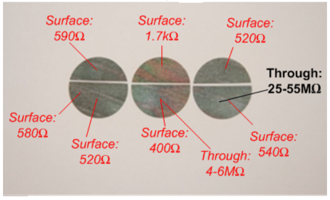
Sample name:	Test AA5
Sample material:	New Termo70 paper
Sample dimensions:	Circular. Diameter of 25mm
Equipment used for DFM[a]:	Alpha-Analyser
Test frequency range:	100Hz – 1MHz
Other tests performed	Resistance measurement and area percentage calculations

---

[a] Dielectric Frequency Measurement



Away



Toward

Figure A.22: Test AA5: Test sample. Red and italic - Multimeter, Black and bold - Megger at 200V

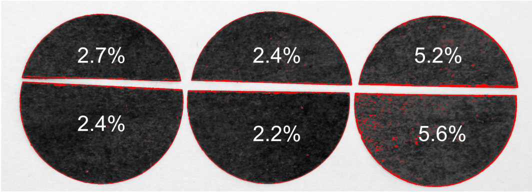


Figure A.23: Test AA5: Area percentage measurement and calculations

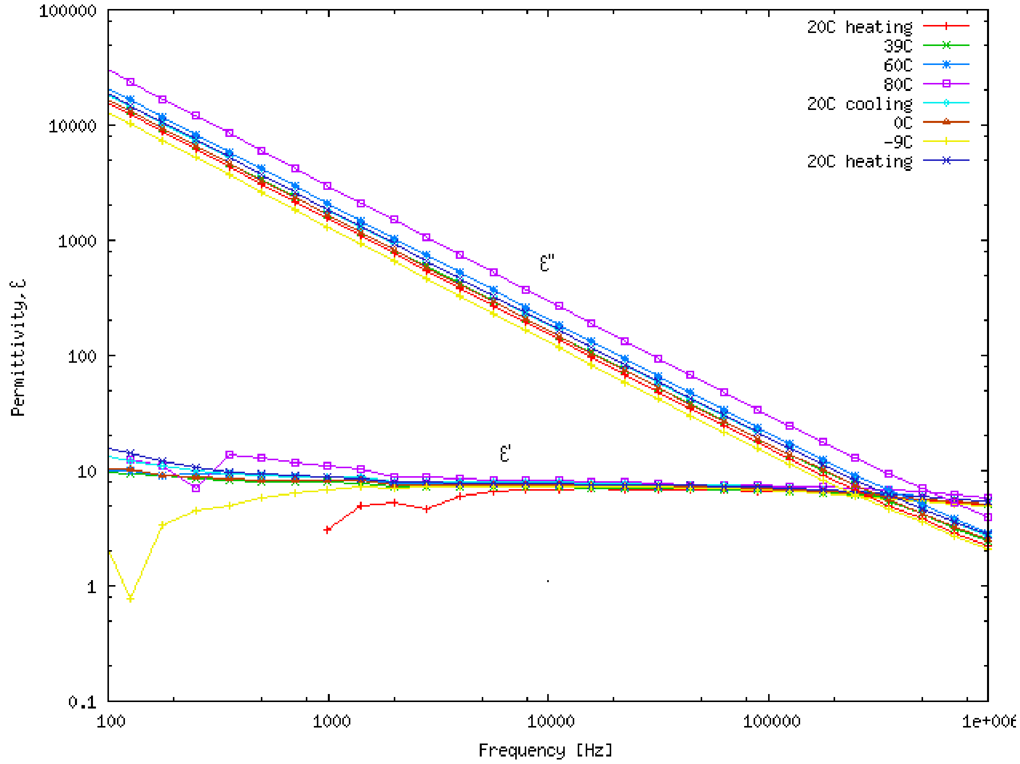


Figure A.24: Test AA5: Result of the dielectric frequency measurements.

A.12 Test AA6: Sample Grown in the Laboratory for Five Days

Sample name:	Test AA6
Sample material:	New Termo70 paper
Sample dimensions:	Circular. Diameter of 25mm
Equipment used for DFM[a]:	Alpha-Analyser
Test frequency range:	100Hz – 1MHz
Other tests performed	Resistance measurement

---

[a] Dielectric Frequency Measurement

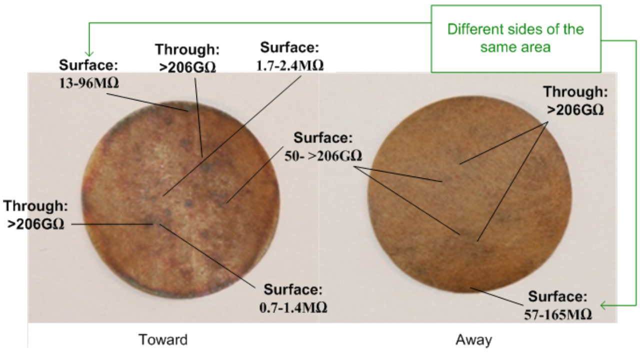


Figure A.25: Test AA6

Due to large variations in brightness on both paper and Cu_2S contamination, the use of area percentage calculation was not feasible.

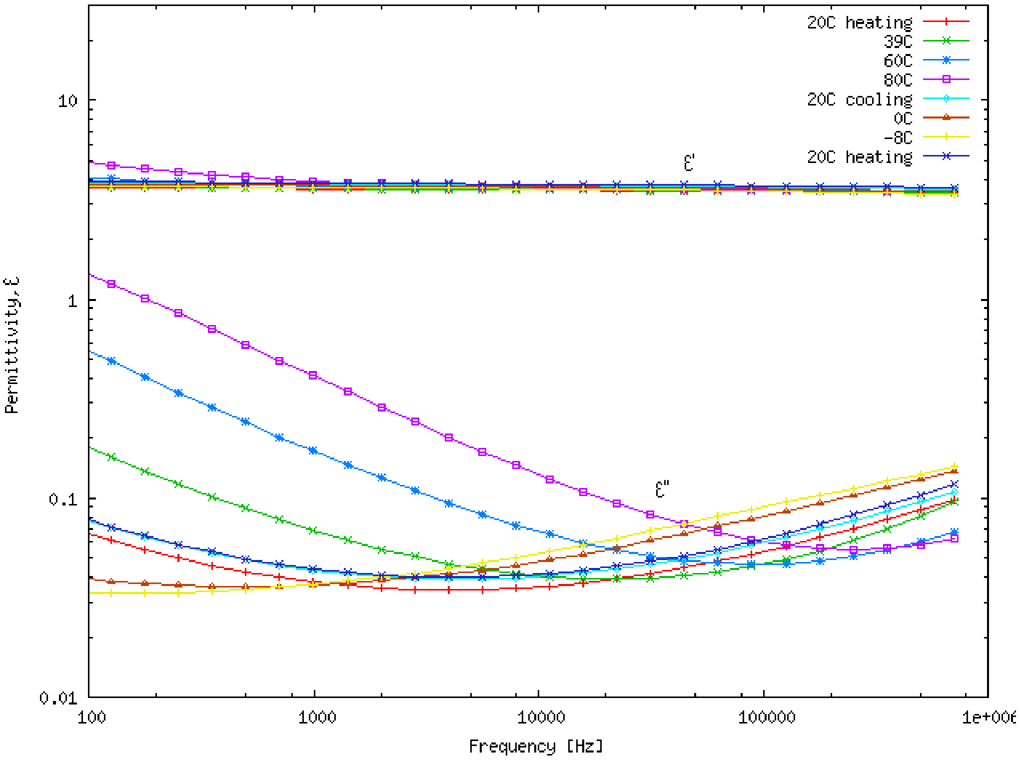


Figure A.26: Test AA6: Result of the dielectric frequency measurements.

A.13 Test AA7: Sample Grown in the Laboratory for Seven Days

Sample name:	Test AA7
Sample material:	New Termo70 paper
Sample dimensions:	Circular. Diameter of 25mm
Equipment used for DFM[a]:	Alpha-Analyser
Test frequency range:	100Hz – 1MHz
Other tests performed	Resistance measurement

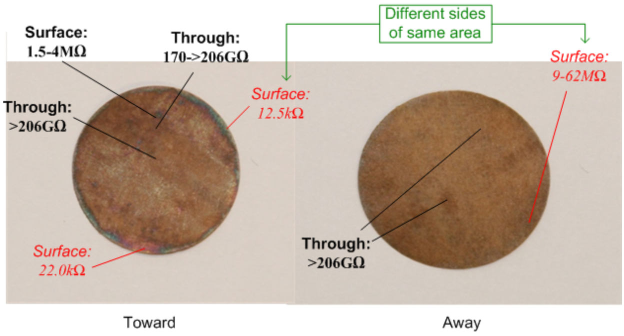


Figure A.27: Test AA7

Due to large variations in brightness on both paper and Cu_2S contamination, the use of area percentage calculation was not feasible.

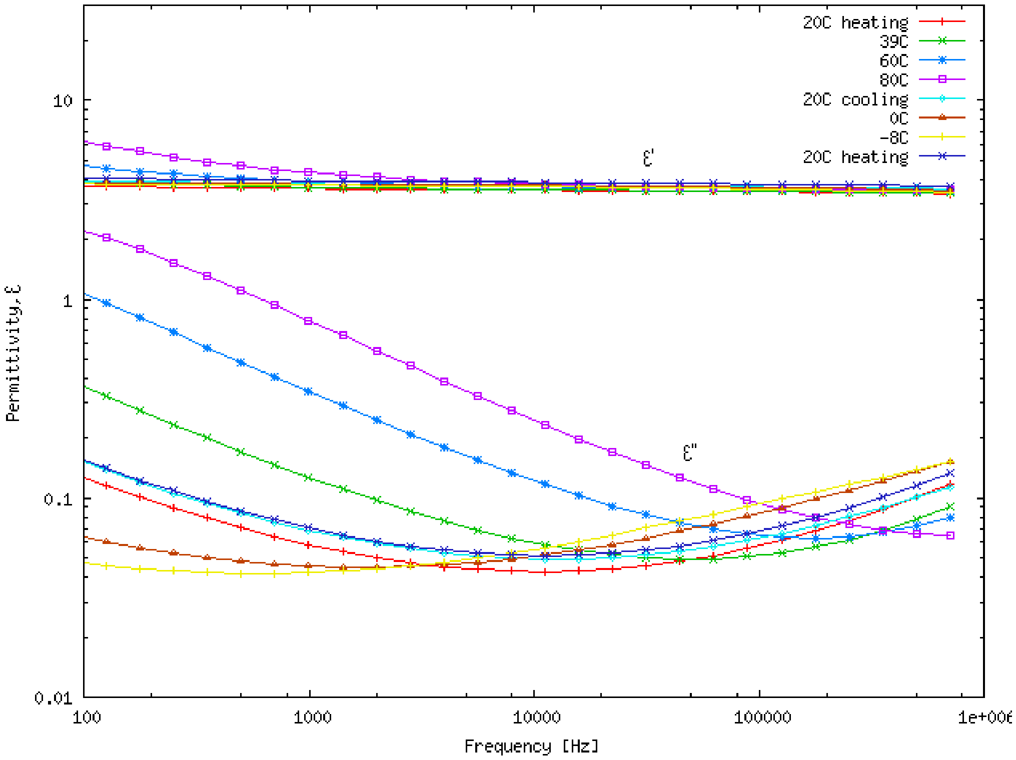


Figure A.28: Test AA7: Result of the dielectric frequency measurements.

---

[a] Dielectric Frequency Measurement

A.14 Test AA8: Sample Grown in the Laboratory for Five Days, Second Test Sample

Sample name:	Test AA8
Sample material:	New Termo70 paper
Sample dimensions:	Circular. Diameter of 25mm
Equipment used for DFM[a]:	Alpha-Analyser
Test frequency range:	100Hz – 1MHz
Other tests performed	Resistance measurement

---

[a] Dielectric Frequency Measurement

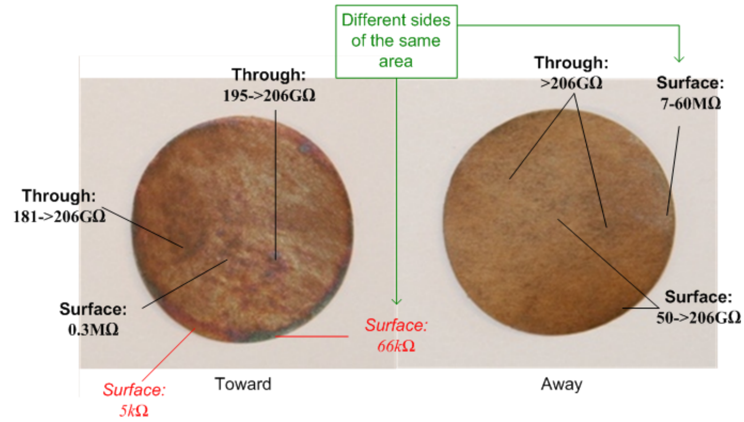


Figure A.29: Test AA8

Due to large variations in brightness on both paper and Cu_2S contamination, the use of area percentage calculation was not feasible.

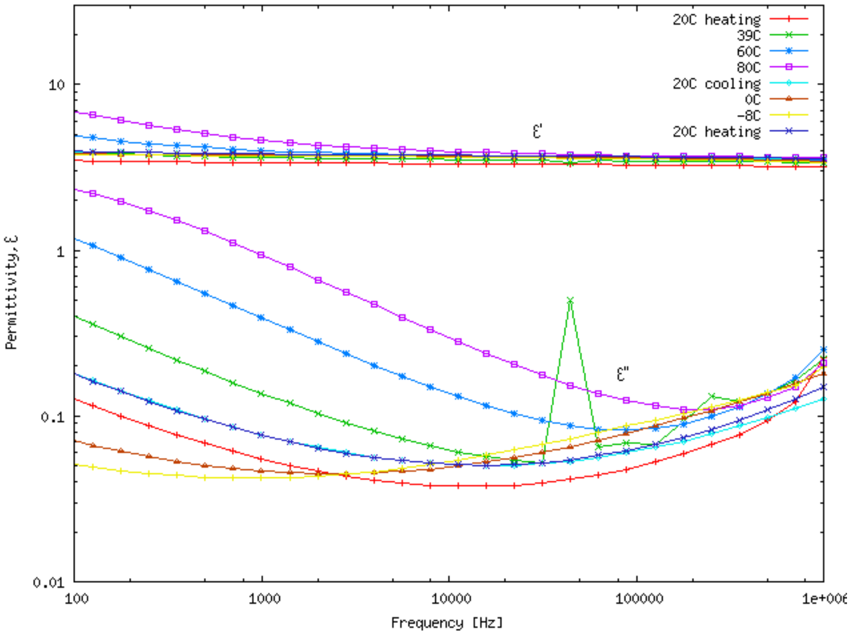


Figure A.30: Test AA8: Result of the dielectric frequency measurements.

A.15 Test Samples Created, but not Tested for Dielectric Frequency Response

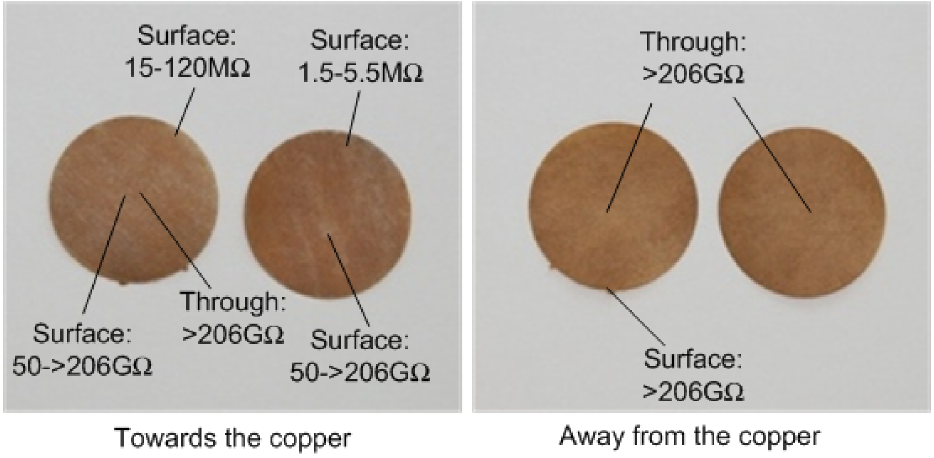


Figure A.31: Samples grown for three days. Red and italic - Multimeter, Black and bold - Megger at 200V

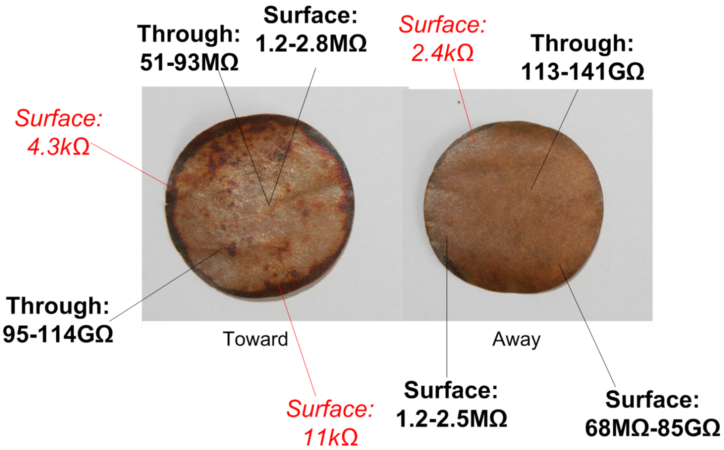


Figure A.32: Sample grown for eleven days. Red and italic - Multimeter, Black and bold - Megger at 200V

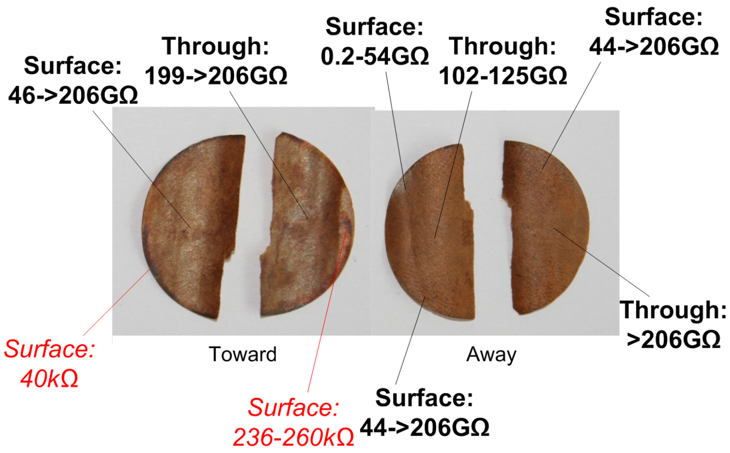
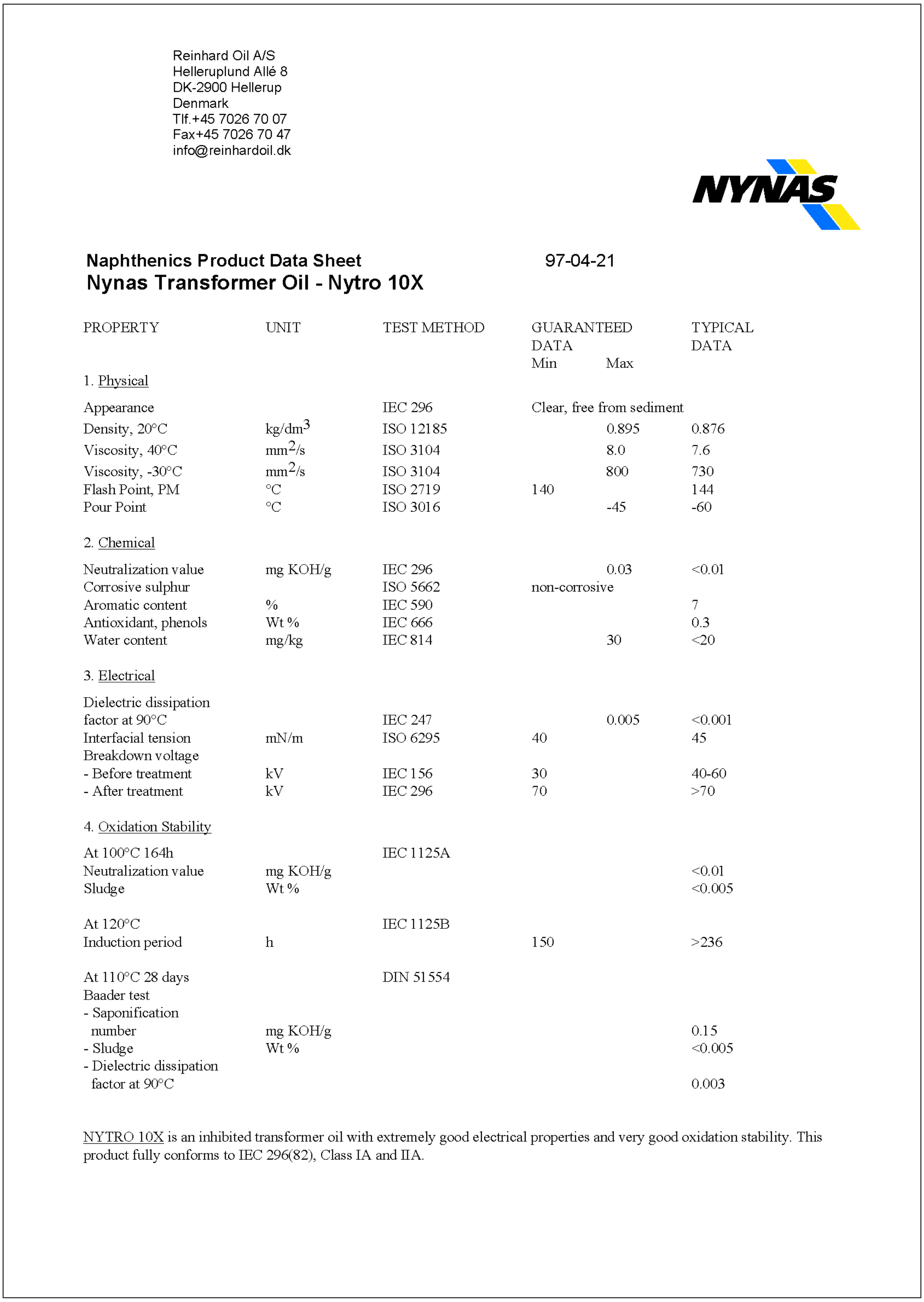


Figure A.33: Sample grown for fourteen days. Red and italic - Multimeter, Black and bold - Megger at 200V

A.16 Nytro 10X Data Sheet



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Naphthenics Product Data Sheet 97-04-21

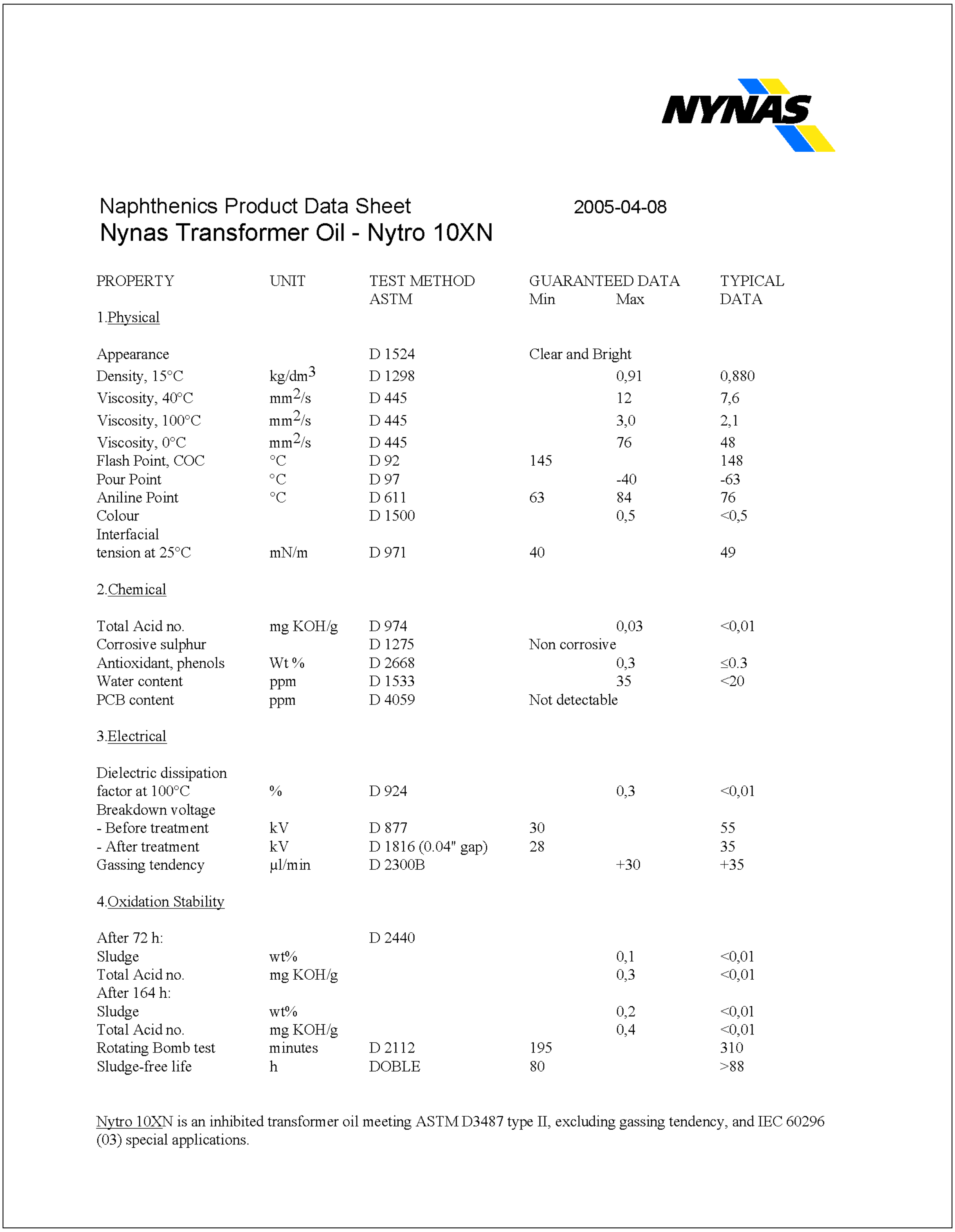
Nynas Transformer Oil - Nytro 10X

PROPERTY	UNIT	TEST METHOD	GUARANTEED DATA		TYPICAL DATA
			Min	Max	
1. Physical					
Appearance		IEC 296	Clear, free from sediment		
Density, 20°C	kg/dm^3	ISO 12185		0.895	0.876
Viscosity, 40°C	mm^2/s	ISO 3104		8.0	7.6
Viscosity, -30°C	mm^2/s	ISO 3104		800	730
Flash Point, PM	°C	ISO 2719	140		144
Pour Point	°C	ISO 3016		-45	-60
2. Chemical					
Neutralization value	mg KOH/g	IEC 296		0.03	<0.01
Corrosive sulphur		ISO 5662	non-corrosive		
Aromatic content	%	IEC 590			7
Antioxidant, phenols	Wt %	IEC 666			0.3
Water content	mg/kg	IEC 814		30	<20
3. Electrical					
Dielectric dissipation factor at 90°C		IEC 247		0.005	<0.001
Interfacial tension	mN/m	ISO 6295	40		45
Breakdown voltage					
- Before treatment	kV	IEC 156	30		40-60
- After treatment	kV	IEC 296	70		>70
4. Oxidation Stability					
At 100°C 164h		IEC 1125A			
Neutralization value	mg KOH/g				<0.01
Sludge	Wt %				<0.005
At 120°C		IEC 1125B			
Induction period	h		150		>236
At 110°C 28 days		DIN 51554			
Baader test					
- Saponification number	mg KOH/g				0.15
- Sludge	Wt %				<0.005
- Dielectric dissipation factor at 90°C					0.003

NYTRO 10X is an inhibited transformer oil with extremely good electrical properties and very good oxidation stability. This product fully conforms to IEC 296(82), Class IA and IIA.

Figure A.34: Data sheet for Nytro10X transformer oil

A.17 Nytro 10XN Data Sheet



Naphthenics Product Data Sheet 2005-04-08

Nynas Transformer Oil - Nytro 10XN

PROPERTY	UNIT	TEST METHOD	GUARANTEED DATA		TYPICAL
		ASTM	Min	Max	DATA
1.Physical					
Appearance		D 1524	Clear and Bright		
Density, 15°C	kg/dm^3	D 1298		0,91	0,880
Viscosity, 40°C	mm^2/s	D 445		12	7,6
Viscosity, 100°C	mm^2/s	D 445		3,0	2,1
Viscosity, 0°C	mm^2/s	D 445		76	48
Flash Point, COC	°C	D 92	145		148
Pour Point	°C	D 97		-40	-63
Aniline Point	°C	D 611	63	84	76
Colour		D 1500		0,5	<0,5
Interfacial tension at 25°C	mN/m	D 971	40		49
2.Chemical					
Total Acid no.	mg KOH/g	D 974		0,03	<0,01
Corrosive sulphur		D 1275	Non corrosive		
Antioxidant, phenols	Wt %	D 2668		0,3	≤0.3
Water content	ppm	D 1533		35	<20
PCB content	ppm	D 4059	Not detectable		
3.Electrical					
Dielectric dissipation factor at 100°C	%	D 924		0,3	<0,01
Breakdown voltage					
- Before treatment	kV	D 877	30		55
- After treatment	kV	D 1816 (0.04" gap)	28		35
Gassing tendency	µl/min	D 2300B		+30	+35
4.Oxidation Stability					
After 72 h:		D 2440			
Sludge	wt%			0,1	<0,01
Total Acid no.	mg KOH/g			0,3	<0,01
After 164 h:					
Sludge	wt%			0,2	<0,01
Total Acid no.	mg KOH/g			0,4	<0,01
Rotating Bomb test	minutes	D 2112	195		310
Sludge-free life	h	DOBLE	80		>88

Nytro 10XN is an inhibited transformer oil meeting ASTM D3487 type II, excluding gassing tendency, and IEC 60296 (03) special applications.

Figure A.35: Data sheet for Nytro10XN transformer oil