Ultramicroscopy 161 (2016) 137–145

Contents lists available at ScienceDirect

Ultramicroscopy

journal homepage: www.elsevier.com/locate/ultramic

Full length article

Measurement of the permittivity and loss of high-loss materials using a Near-Field Scanning Microwave Microscope

A.P. Gregory [a,*], J.F. Blackburn [a], K. Lees [a], R.N. Clarke [a], T.E. Hodgetts [b], S.M. Hanham [c], N. Klein [c]

[a] National Physical Laboratory (NPL), United Kingdom
[b] Consultant to NPL, United Kingdom
[c] Imperial College London, United Kingdom

ARTICLE INFO

Article history:
Received 22 September 2015
Received in revised form
25 November 2015
Accepted 28 November 2015
Available online 30 November 2015

Keywords:
Dielectric measurement
Microwave microscope
Complex frequency

ABSTRACT

In this paper improvements to a Near-Field Scanning Microwave Microscope (NSMM) are presented that allow the loss of high loss dielectric materials to be measured accurately at microwave frequencies. This is demonstrated by measuring polar liquids (loss tangent $\tan\delta \approx 1$) for which traceable data is available. The instrument described uses a wire probe that is electromagnetically coupled to a resonant cavity. An optical beam deflection system is incorporated within the instrument to allow contact mode between samples and the probe tip to be obtained. Liquids are contained in a measurement cell with a window of ultrathin glass. The calibration process for the microscope, which is based on image-charge electrostatic models, has been adapted to use the Laplacian 'complex frequency'. Measurements of the loss tangent of polar liquids that are consistent with reference data were obtained following calibration against single-crystal specimens that have very low loss.

1. Introduction

Two main types of Near-Field Scanning Microwave Microscope (NSMM) [1] are described in the literature: Instruments that are based on the perturbation of the resonance of a cavity that is coupled to a probe tip [2–8], and instruments that are modified Atomic Force Microscopes (AFMs). AFM-based instruments [9–13] use a Vector Network Analyser (VNA) to measure reflection coefficient data at a reference plane at a tip attached to an AFM cantilever. They are principally used for measuring the conductivity of semiconductors at the *nanoscale*, but strategies for measurement of permittivity have recently been reported [12,13]. The NSMM which is described in this paper is of the cavity perturbation type. Compared to AFM-based instruments this has a simpler and more calculable geometry as stray fields associated with the cantilever are avoided. It is suitable for measuring the complex permittivity of non-conductive materials, such as ceramics and polymers. The instrument described uses a comparatively large probe (a wire probe with a spherical tip approximately 0.18 mm in diameter), which enables measurement on the *micron scale* (which is significantly smaller than the tip diameter as the field is concentrated in the region closest to specimens, especially when the permittivity is high). Q-factor and resonant frequency are obtained by fitting to complex transmission coefficients measured with the VNA, from which complex permittivity may be calculated. A recent review [1] provides more information on the current state of the art of both types of instrument.

In order to make quantitative measurements, the NSMM must be calibrated prior to use against solid reference specimens that have known and reproducible properties, and are isotropic, polished and uniform on a small scale. Single crystal materials with a permittivity of up to 24 that satisfy these requirements are available, however a suitable high loss solid material has not been identified. Carbon-loaded composite materials, for example, can have high loss but do not have the required uniformity at the micron scale. In this paper new approaches to loss measurement are studied. It will be shown that accurate measurements of loss can be obtained following calibration with low-loss single crystals using an implementation of perturbation theory under the quasi-static limit that uses the concept of a 'complex frequency' (which mathematically combines resonant frequency and Q-factor). This is tested by making measurements on polar liquids [14], which have high loss and make ideal candidates as reference standards as traceable complex permittivity data is available in the literature [15]. Other workers have used NSMMs to obtain images of aqueous and biological samples in which the probe is immersed [16,17]. In the work described here a well-controlled and calculable geometry was needed, so liquids were contained in a cell with an ultrathin glass window, Fig. 1. The main purpose of this paper is to

* Corresponding author.

http://dx.doi.org/10.1016/j.ultramic.2015.11.015
0304-3991/

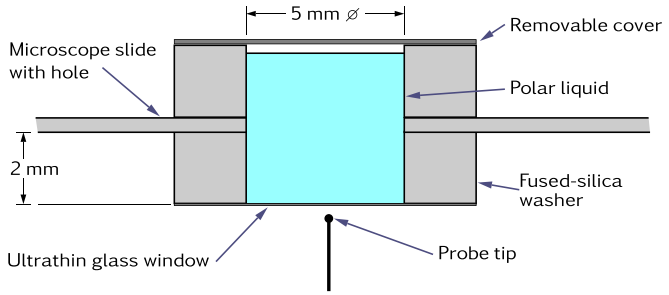

Fig. 1. Cell for measurement of the polar liquids.

study algorithms for calibration and extracting ε' and $\tan\delta$, so measurements of uniform specimens at point locations will only be presented. Raster scans can, however, be made in contact mode to allow images to be obtained.

The paper is arranged as follows: in Section 2, an overview of the design of the microscope is presented. Section 3 describes a subsystem of the instrument that enables contact mode between tip and specimens to be maintained by shear force detection. Calculation and calibration processes are described in Sections 4 and 5. A set of measurements on polar liquids are presented in Section 6. An analysis of sensitivity coefficients to inform evaluation of uncertainties is presented in Section 7. Section 8 is the Conclusion.

2. Overview of the NSMM

The NSMM (Fig. 2) is built into an acoustically-shielded chamber with an active anti-vibration table. The combined power supply and control unit for the anti-vibration table is located externally to reduce temperature rises inside the chamber. Shifts in the resonant frequency of the microwave cavity below 100 kHz are significant in these measurements, so mechanical and thermal stability are important. Physik Instrumente (PI) type M-605 X & Y motorised stages with 0.3 μm resolution allow large area scans (50×25 mm). Coarse control of the separation between the specimen and probe tip is provided by means of a motorised Z stage (range 0–12.5 mm). Fine-scale movement ($100 \times 100 \times 10$ μm) of the specimens is provided by a three-axis closed-loop piezo stage (PI 733.3CD). A subsystem based on beam deflection (see Section 3) can be used as part of a feedback circuit to control the Z axis to obtain contact mode. The movement range of the Z axis of the piezo stage is only 10 μm, so if contact mode is lost it is unlikely that significant damage will occur. The piezo stages have position sensors based on capacitance measurement which can be read via the GPIB interface.

Specimens (including calibration specimens) are attached to a fused silica substrate (a slide for an optical microscope) using a wax or adhesive. Small magnets are used to attach the slide to the piezo XYZ stage. The control software enables measurement positions to be set up manually and stored. This is achieved by using a joystick attached to the motor controller (PI C-848) to set Z-axis height and a CCD camera with a zoom lens to view the tip and specimen. The motorised XYZ stages can be moved to any of the stored positions using buttons on the software interface. This allows measurements to be made in the minimal amount of time (to reduce drift) and with minimal disturbance to the system. The motorised Z stage can be raised in micrometre steps via the software user-interface to allow the user to adjust the specimen/tip separation.

The resonator is a coaxial cavity (Fig. 3) with coupling loops that are adjusted to give weak coupling (insertion loss ≈ 36 dB). The cavity resonant frequency and Q-factor are obtained by the following procedure: A VNA (Rohde and Schwarz ZVB20) is used to make measurements of the complex transmission coefficient of the cavity over a swept frequency range. These form a circular arc (often referred to as a Q-circle) if plotted on a polar chart. By using a resonance model the resonant frequency f_r, loaded Q-factor Q_L, and the Q-circle diameter d can be obtained by a weighted fit [18]. To improve accuracy the leakage vector, which accounts for signal paths between the two ports of the VNA that bypass the resonant circuit, is also fitted. Prior to measurement of Q-factor a preliminary sweep is used to set the VNA frequency span to an optimum range: $f_r \pm f_r/Q_L$. The settings of the VNA used for the measurements presented in this paper were as follows: IFBW 1 kHz, averaging factor 1, source power 0 dBm, 51 points, sweep time 0.5 s. For these settings the standard deviation of repeated Q-factor measurements is approximately ± 1. As the coupling factors at each port were approximately equal, unloaded Q-factor is given by $Q_L/(1-d)$ [18]. The VNA is used uncalibrated, but to improve accuracy d is normalised to the magnitude of the transmission coefficient measured when the resonator is replaced by a direct connection.

The cavity is shaped so as to enable an optical beam deflection system to be constructed, though this is achieved at the cost of a reduction in Q-factor and the generation of undesirable higher-order modes. These cause a reduction in the Q-factor and an increase in the effect of higher-modes. At the lowest resonant frequency (1.21 GHz), corresponding to a cavity length of $\lambda/4$, the cavity resonance was observed to be very well-shaped – this is evidenced by the fact that if the if the VNA sweep range is set to

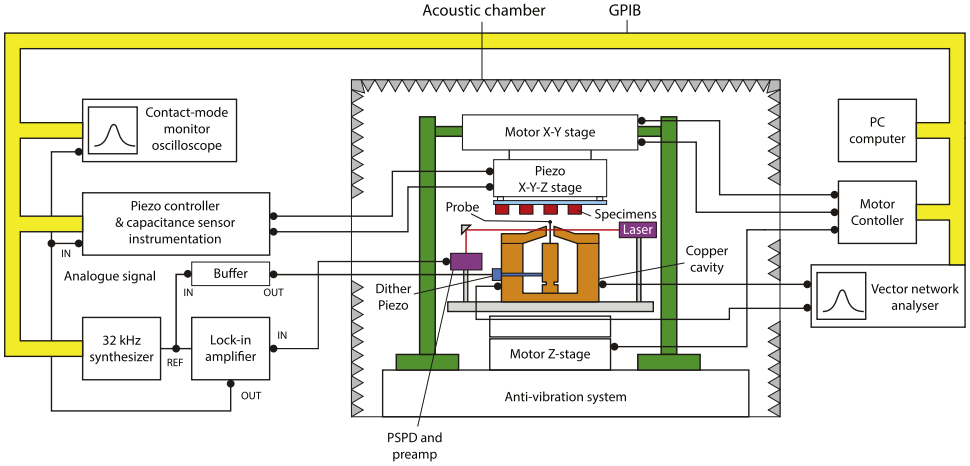

Fig. 2. Schematic of the NSMM.