National Physical Laboratory

EMINDA

Electromagnetic Characterisation of Materials for Industrial Applications up to Microwave Frequencies

Good Practice in Specimen Preparation, Specimen Handling & Thickness Measurement

Andrew Gregory, Kevin Lees, Bob Clarke, NPL

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National Measurement System

This Presentation covers:

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Electromagnetic Characterisation of Materials for Industrial Applications up to Microwave Frequencies

- General Good Practice
- Specimen Handling and Preparation
- Specimen Size and Thickness Measurement

... giving particular emphasis to RF and Microwave dielectric measurements

Topics: Good Practice in Dielectric Measurements:

- Cleanliness
- Clear Identification of specimens
- Storage of specimens
- Manufacturing and preparation of specimens
- Designing specimens to minimise Uncertainty
- Health and Safety Guidelines – toxic materials
- Disposing of specimens safely

- Keep good records!

- Always measure the temperature – ideally to 0.2˚C

Cleanliness

- Do not touch low-loss specimens – you can contaminate them and increase their loss
  - Ceramic specimens can absorb salts from the skin and their loss may be permanently affected

- Toxic specimens may contaminate you!

- Ensure that measurement cells and systems are clean, both before and after use.
  - Specimens may contaminate other specimens

Moisture

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- Some specimens – especially liquids – are hygroscopic: they can absorb moisture from the atmosphere
- This may not matter for specimens that are already very lossy but absorption of moisture is to be avoided for medium and low loss materials
- Storing for some days in a jar with drying agents may be necessary if specimens are to be measured dry

Clear Identification

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- All specimens must be clearly identifiable to ensure that they do not get mixed up with others
  - It is not always a good idea to mark the specimens themselves as this may contaminate them
  - Marking them on supposedly 'unimportant' surfaces may limit the number of systems you can measure them in! For a different measurement system, you may have marked a key surface!
  - It may be helpful to record the batch of material from which each specimen has been cut

- Place specimens in clearly marked containers
  - <u>and only take one specimen out at a time!!</u>

Storage

- Store specimens in dark drawers or cupboards
  - some materials, especially polymers, will degrade when exposed to ultraviolet or visible light.

- Support systems adequately whilst in storage to ensure that they do not warp or fracture
  - polymer specimens are prone to warping over long periods of storage. Placing a weight on them can help to avoid this.

Manufacturing and Preparation - 1.

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It is very important to have properly prepared specimens

- The uncertainty of most dielectric measurements depends critically upon one or more specimen dimensions.
  - Surfaces need to be flat, circular or spherical (as appropriate) to high levels of tolerance
  - Specimen thicknesses often have to be know accurately, so opposite faces need to be parallel

- These requirements are often best achieved by machining specimens to the right size and shape by appropriate methods
  - Moulding or sintering specimens to size, without further machining, is often necessary but will generally lead to greater measurement uncertainties caused by specimen imperfections

Manufacturing and Preparation - 2.

It is very important to have properly prepared specimens

- Machining specimens:
  - Use low tool speeds for polymers
  - No cutting fluids if possible. If dry machining not possible use deionised water
  - Vacuum chucks offer way of holding specimens without contaminating or distorting them
  - The machines cut be thoroughly cleaned beforehand!
  - For coaxial-line/WG measurements minimise corner chipping (asymmetries can generate propagating higher-order modes)

- Remove all burrs and projections from specimens (they may prevent the specimen from lying flat on a surface
  - If possible use centre-less grinding: this avoids a ‘knob’ or projection at the centre of a turned specimen

Design specimens to match field geometries

- Be aware of the electromagnetic field geometries in your measurement cells

  - This is particularly important if you have imperfect specimens - e.g. warped specimens or specimens that are smaller than ideal, correct specimen placement can improve measurement accuracy.
  - The effect of chips on specimens can sometimes be reduced by orientating specimens in an optimal way
  - Where possible prepare specimens with 2-3 different thicknesses to improve confidence in measurements.

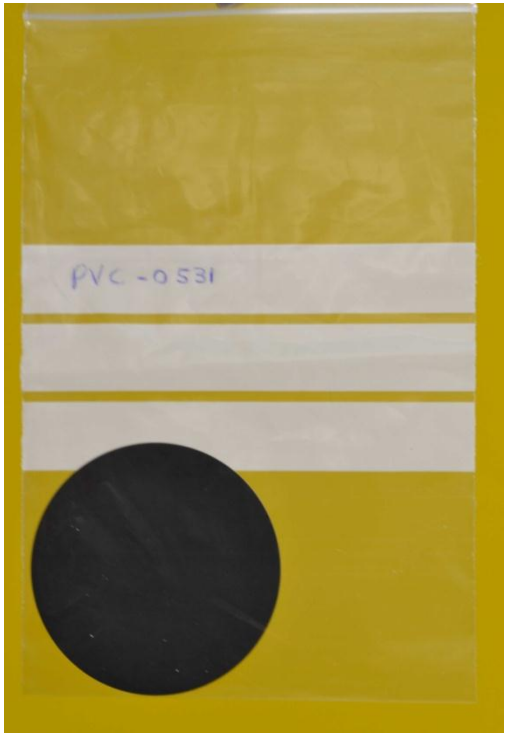
- In general, place the most imperfect part of the specimen in a part of the measurement cell where the electric (or magnetic) field is the lowest allowable.

Health and Safety

- Follow Health and Safety Guidelines for the materials you are dealing with – especially if they are toxic materials
- Dispose of specimens in accordance with Health and Safety guidelines
- This is especially important for liquids – don't just pour them down the sink!
- If in doubt, consult a safety expert
- National and International chemical practices should always be recognised and followed

Low Loss specimens

- Be especially aware of contamination, especially by moisture
- Do not touch low loss specimens
- Store them in clean environments



Liquids

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- Avoid contamination in either direction – from or to the liquid
  - Don't underestimate the ease with which solvents can absorb contaminants
  - Re-stopper bottle immediately after use

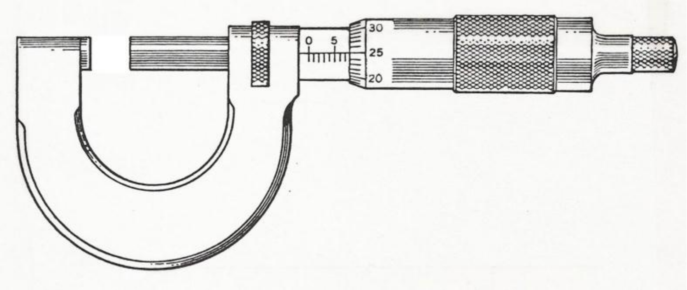
- Beware of evaporation, it cools liquids – measure the temperature of the **liquid**, *<u>not</u>* the cell it is in.

- Don't reuse liquids after measurement, at least don't put them back into the bottle containing unused liquid.

- Beware of the **Electrode Polarisation Effect** at low frequencies in conducting liquids – dielectric measurements on them can give the wrong results.

Measurement of Specimen Size and Thickness

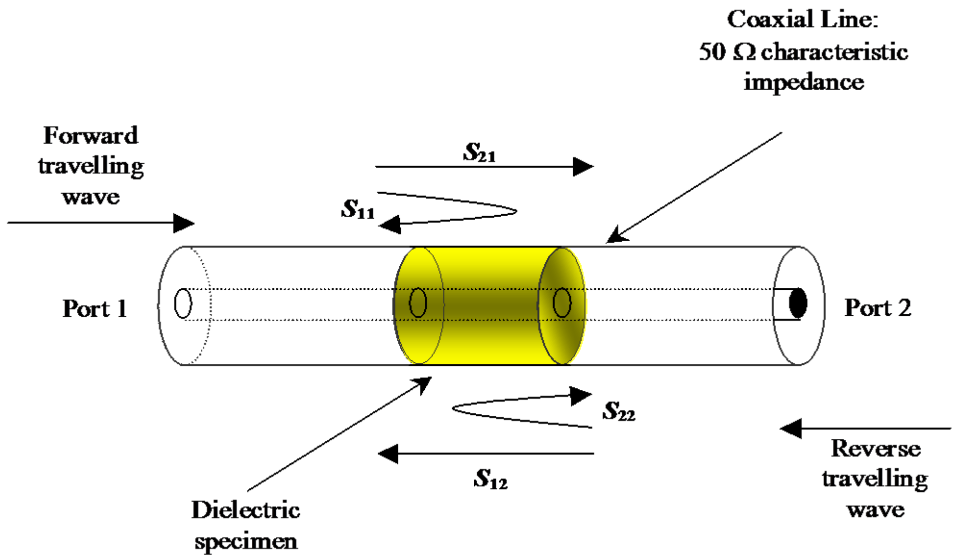
1. Use of Micrometers

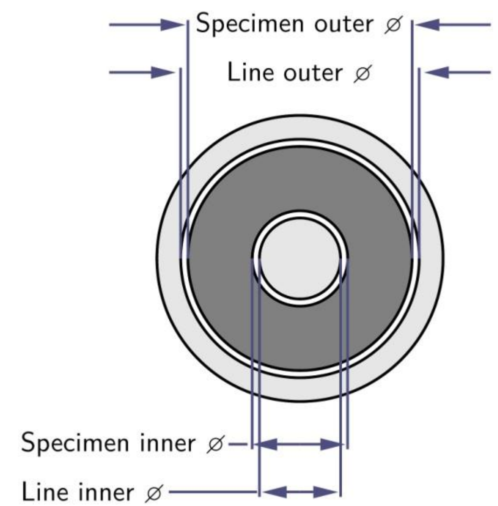


- Use calibrated traceable micrometers
- Measure at a number of places across the face of the specimen … the more uneven it is the more points should be measured.
- Bench-mounted micrometers are to be preferred to hand-held because they present less risk of dropping or contaminating specimens
- Clean the anvils beforehand by drawing a lint free cloth sideways across them
- Ratchet mechanisms can be used for hard specimens
- Soft specimens require minimal force, do not use ratchet mechanisms - feel contact with fingers.
- Rough specimens - bear in mind that micrometers measure the top of projections – the mean thickness will be systematically thinner. Use smaller anvil heads.

Measurement of Specimen Size and Thickness

2. Diameters – Air-gauging





- In some techniques, e.g. measurement of coaxial specimens in a coaxial transmission line, air gaps can cause large measurement errors. Corrections can be applied if the inner and outer diameters of the specimen (and line) are measured.

- Air-gauging can measure these diameters to about 1 micron.

Measurement of Specimen Size and Thickness

3a. Thin films

Potential Techniques with comparison of costs (in £UK)

This is a very complex topic!

Name	Resolution	Equipment Cost	Upkeep Cost	Time (not including setup)
Bench Micrometer	1 µm	£500	£200 per year recalibration	Seconds
Stylus Profilometre	1 nm	£25,000		A Few Minutes
AFM	Sub-nm	£50,000+		Tens of minutes
Confocal microscopy	10 nm	£15,000+	£3000 per annum	Less than a minute
Ellipsometry	Sub nm	£10,000s+	£200 per 1k hrs use.	Tens of minutes
White light interferometry	~1 µm	£5,000+	£3000 per annum	Tens of minutes
SEM	Sub nm	£100,000+	£15k service contract	Half an hour

Measurement of Specimen Size and Thickness

3b. Thin films – some thoughts:

- Specimens of 10 microns and above in thickness: a bench micrometer may give sufficient accuracy.
- If the film is available before metallisation, its thickness is below 1 µm and it is transparent at some optical wave length then ellipsometry may be recommended due to its low cost and accuracy.
- If ellipsometry is not possible and if metallisation thickness must be measured the preferred option could be confocal microscopy, provided the thin films have thicknesses greater than 50 nm.
- If the equipment and expertise is already available , AFM techniques would be the preferred measurement for films below 500 nm.

Conclusions

The most important things to remember:

- Cleanliness is important
- Be aware of specimen contamination
- Always measure the specimen temperature
- Specimen dimensions are critical parameters in EM materials measurements:
  - Machine specimens to optimise thicknesses and the necessary tolerances
  - Use the best methods to measure thickness.
- Take care not to mix specimens up
- Health and safety are important: <u>know</u> the risks associated with the materials you are measuring.