General consideration – read before purchase and use

Thin-film measurements to 1000°C
Setup for ProboStat™

Complementary Material to the ProboStat™ Manual
Update: 06 December 2017

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NORECS

1. Introduction

Electrical characterisation of thin films is not straightforward, for many reasons. Each sample type may face different challenges and require a different approach. High temperatures and controlled atmospheres add to these challenges.

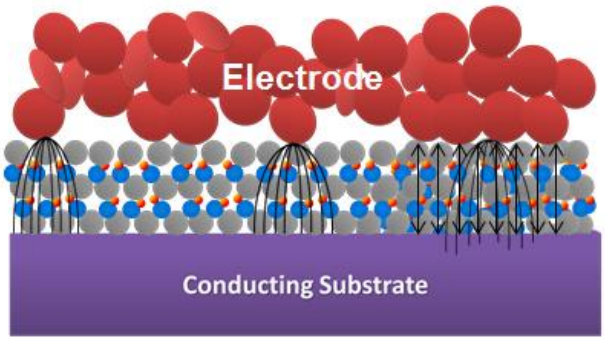
NORECS' high temperature thin film accessories aim to provide a clean, cost-effective, and versatile system that offers standard ways to measure both in- and through-plane for most samples, but that can also accommodate user tailored methods for special samples.

2. Key aspects and common challenges

Sample conductivity, substrate conductivity, and choice of geometry are closely linked. The user must know and understand these aspects, and choose the appropriate substrate, film production procedures, and electrode deposition. The user should also know and understand how to calculate measured electrical properties into specific ones using film and electrode geometries, and vice versa.

A *low conducting* (highly resistive) or *insulating* sample material must be measured *through-plane*, between a top electrode and a *conductive substrate* (typically a metal). Typical challenges in through-plane setups are:

1) The electrode may be made of a too coarse-grained material, or applied with an unsuitable method, so that it does not effectively cover the thin film with the area expected, as illustrated in the figure.



2) The top electrode may penetrate and short-circuit the thin film during electrode fabrication or fills a pinhole already existing on the film.

3) The contact to the top electrode may destroy the electrode and/or penetrate the sample film underneath, leading to loss of contact or short-circuit.

The NORECS 1000°C through-plane setup contains a Pt cup contact for the substrate and a softly spring-loaded Au top electrode contact for flexible and gentle contact to any electrode.

Suitable techniques and materials for fabricating the electrode depend on the sample, and comprise hand painting, physical vapor deposition, e-beam lithography, and sputtering, with varying materials and annealing if possible. Studying which method works best may require additional microscopy to check the appearance of the film, electrode, and contact, in combination with the electrical analysis.

A *highly conductive* sample material must be measured *in-plane* on an *insulating substrate* (e.g. typically alumina or silica). Typical challenge in in-plane setups is parasitic conduction in the substrate, in the substrate-film interface, or on the surface.

The NORECS 1000°C in-plane setup uses the same Pt cup as basis, but now contacts two electrodes on top using two soft spring-loaded gold contacts. These can be made using the same spring load or individual spring loads, as shown in the instructions below. The Pt plate is in this case not used as an electrode contact, however, one may contact it to a ground terminal so as to serve as a guard against (or reduce) parasitic conduction and capacitance in the substrate. A source of parasitic conduction is water adsorbed on the sample surface: Heating the sample above 100°C and/or flushing with dry air will reduce or eliminate adsorbed H_2O.

When measuring setups with high resistance, a stainless steel enclosing tube can be used to reduce noise. It may also help to ground the negative lead of the thermocouple(s) so that it does not act as an antenna inside the sample holder.

In case the user is only interested in in-plane measurements, the Pt cup can be replaced with alumina plate to reduce the system price.

If even the slightest movement causes puncturing of the thin film, another approach to mount the setup is recommended by one of our customer, see the figure.

Place the spring load triangle right on top of the sample support tube, and sample substrate on top of that. Attach 10 cm long, 0.25 mm diameter or similar thin intermediate electrode contact wires to the tips of the gold wire electrode contacts. Apply metal ink to improve contact. Place electrode contacts to the appropriate feedthroughs on the ProboStat™ base unit and align them up next to the sample support tube. Tie them tightly to the sample support tube with suitable metal wire, for example platinum wire. Once rigidly secured, bend the top wires so that they do not short circuit but that they end close to the electrodes on the sample. Attach them to the painted electrodes with a drop of suitable metal ink. Let dry and sinter if possible. When adding the enclosing tube, even if the enclosing tube touches the electrode contact alumina tubes the electrode contact wires do not move, and the sample layer remains intact.

The NORECS 1000°C through-plane setup and two in-plane setups are shown on the pages below. In all cases, remove protection silicone hose from Au electrode contacts before you start. *Especially the mounting of the one or two Au contacts may be considered difficult, and their position easily disturbed during mounting of the outer quartz tube etc. The mounting requires some practice, so test on a dummy sample is recommended.* We suggest that the capacitance or conductance of the sample is monitored during mounting and handling of the setup, to ensure that the electrodes and contacts are intact.

3. Assembly

3.1. Through-plane setup

The following explains how through-plane measurements are set up using parts from the NORECS thin films measurements accessories package.

Mount the 16 mmØ sample support tube on the base unit. Place the Pt cup on top of the tube.

Hook the end of the lower Au electrode contact through one of the holes in the Pt cup, and connect its female contact to one of the feedthrough connectors.

Place the top of the spring load assembly over the middle of the Pt cup and connect the two long springs. The end of the spring hook should point towards the sample support tube. Hook the springs to the pedestal rim.

Connect the female contact of an Au electrode contact to one of the feedthrough connectors. Insert the upper, flat end so that it is held in place by the alumina spring load tube.

Lift the alumina spring load tube and the Au hook, and place the sample. Carefully lower the parts again so that the gold wire gently touches the selected sample electrode on top of the film.

Lower the enclosing tube gently so that the setup is not touched. Secure the enclosing tube with O-ring and outer flange.

3.2. In-plane setup, alternative A: One common spring load

Mount the 16 mmØ sample support tube. Place the Pt cup on top of the tube.

Spring load: Place the top part of the spring load assembly on the middle part of the Pt cup and connect the two long springs. The end of the spring hook should face towards the sample support tube.

Au electrode contact: Connect the lower end to an appropriate feedthrough connector. Hook the upper, flat end so that it is held in place by the alumina spring load tube.

Lift the alumina spring load tube and the Au hook, and carefully place the sample. Carefully lower the parts again so that the gold wire gently touches an area of no interest on the sample.

Mount the second upper electrode contact the same way as the first one. Now, lift up slightly the alumina spring load tube and move the Au hooks to two selected electrodes.

Lower the enclosing tube gently so that the setup is not touched. Secure the enclosing tube with O-ring and outer flange.

3.3. In-plane setup, alternative B: Two individual spring loads

Mount the 16 mmØ sample support tube. Place the Pt cup on top of the tube.

Spring load: Place the top part of the spring load assembly on the middle part of the Pt cup and connect the two long springs. The end of the spring hook should face towards the sample support tube.

Au electrode contact: Connect the lower end to one of the feedthrough connectors. Hook the upper flat end so that it is held in place by the alumina spring load tube.

Lift the alumina spring load tube and the Au hook, and place the sample. Carefully lower the parts again so that the gold wire gently touches an area of no interest on the sample.

Mount the second set of alumina spring load assembly and upper electrode contact the same way as the first one. Now, lift up slightly the alumina spring load tubes and move the Au ”hooks” to two selected electrodes.

Lower the enclosing tube gently so that the setup is not touched. Secure the enclosing tube with O-ring and outer flange.

4. Materials and maximum temperature

The used materials are alumina AL23, Au, Pt, Pt10%Rh, Inconel. This normally allows an operating temperature in the hot part of the cell of 1000°C, of course depending on sample and exposure time.

5. Instructions for fabrication of selected parts

All measures given here are for standard cells (enclosing tube length – 60 cm). Use the appropriate measure for your cell if it is shorter than the standard cell.

5.1. Lower electrode plate

5.1.1. Materials

Pt cup with inner diameter Ø=16 mm
Diamond saw

5.1.2. Procedure

Drill holes for electrode connections.
Cut 1 mm segments from opposite cup sides.

5.2. Electrode contact assembly for lower electrode plate

5.2.1. Materials

Alumina tube, 2 mm Ø, 50 cm length
Au wire, 0.5 mmØ, 54.5 cm length
Female feedthrough connector
Soldering tin

5.2.2. Procedure

Cut 54.5 cm of Au wire.
Thread the wire end in the alumina tube.
Solder feedthrough connector to one end at the soldering iron temperature 250°C or lower.

5.3. Upper electrode contact assembly

5.3.1. Materials

Alumina tube, 2 mm Ø, 50 cm length
2 alumina tubes, 2 mm Ø, 0.8 cm length
Au wire, 0.5 mm Ø, 58 cm length
Female feedthrough connector
Soldering tin

5.3.2. Procedure

Cut Au wire to 58 cm.
Thread the Au wire in the long tube.
Solder feedthrough connector to one end at the soldering iron temperature 250°C or lower.

From the open wire end thread two 0.8 cm alumina tubes.

5.4. Spring load system

5.4.1. Materials

4 Inconel springs, spring length 15 cm, with one long straight end
2 alumina tubes, 2 mm Ø, 2.4 cm length
8 alumina tubes, 2 mm Ø, 0.8 cm length
Pt10Rh wire, 0.5 mm Ø, 21 cm length

5.4.2. Procedure

Cut springs to the total length 49 cm.
Make 5 mm hook on straight end of the springs.
Cut two 10.5 mm lengths of Pt10Rh wire.
Make a loop at one end of the wire.
Thread wire series in two 0.8 cm tubes, 2.4 mm tube, two 0.8 cm tubes.
Male a loop at the second end.
Place 2.4 mm tube in the center and bend wire to 90° from both side of the tube.
Thread wire in the alumina tube.
Solder feedthrough connector to one end at the soldering iron temperature 250°C or lower.
From the open wire end thread two 0.8 cm alumina tubes.