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Local temperature surface measurement with intrinsic thermocouple

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ABSTRACT

This paper reports on a technique based on the well-known Seebeck effect to measure local surface temperature. The aim of this work is to increase the spatial resolution of temperature measurement performed using thermocouples. The method relies on an intrinsic thermocouple consisting of a conducting tip put in contact with a conducting sample. The tip is made of nickel or constantan wires fabricated using an electrochemical etching process. This paper shows that the electric behavior of an intrinsic junction is similar to the one of a standard soldered thermocouple. The main difference between these two techniques is the localization of the measure. For a standard soldered thermocouple, the temperature value is the result of an average of the real temperature of the weld volume while for an intrinsic thermocouple the temperature information is localized in the contact area. The first experimental results actually confirm that a better spatial resolution can be obtained in the case of the intrinsic method, compared to a standard soldered thermocouple used in the same experimental conditions.

1. Introduction

Temperature and heat flux measurements at submicrometer scales have a broad range of applications related for instance to semiconductor devices, chemical reactions and phase transformations. Existing techniques include thermoelectric probes such as thermocouples, semiconductor diodes and thermistors. Optical techniques such as infrared thermometry [1,13] and surface reflectometry [2] are also state-of-the-art tools. Scanning Thermal Microscope (SThM) is a quite recent metrology technique based on a thermocouple or a thermoresistive probe mounted on an Atomic Force Microscope (AFM) [3–9]. It provides thermal and topographical images of sample surfaces with high spatial resolution. The accuracy and the spatial resolution of those measurements are however affected by three main parameters: the coupling between the thermal and topographical signals, the heat transfer in air which spreads the thermal contact over a micrometer scale and the thermal conductivity of the studied materials or of the sub-surface materials. This last effect impacts on the measurement when the penetration depth of the thermal signal is comparable or greater than the contact radius.

In this paper, we describe a non standard technique based on an intrinsic thermocouple made of a metallic tip contacting a metallic heating surface. The local probing is based on the Seebeck effect. Local investigation is easy because the contact radius between the tip and the sample is estimated to be in the 0.5–1 μm range depending on the applied contact force and on the size tip radius [10]. The contact radius can be roughly assimilated to the electrical and thermal contact radii. The heat conduction through the air can be neglected. As the thermal measurement is based on the Seebeck junction effect, surface effects are predominant. The system will therefore not be sensitive to buried materials or impurities. The measurement method has been validated on a specific sample consisting of arrangements of thin layers on a glass substrate in order to demonstrate the intrinsic thermoelectric effect.

2. Theoretical basis

In 1821, Thomas Johan Seebeck discovered that it was possible to create an electrical voltage by soldering two different metals (see Fig. 1). The Seebeck effect is at the heart of thermocouples: if two metals having different Seebeck coefficients are joined together at one end, a voltage called ElectroMotive Force (EMF) can be detected using a voltmeter at the other end. The measured electromotive force is proportional to the thermal gradient between the hot and cold junction materials [11].

As mentioned earlier, the reported instrumentation method makes use of an intrinsic thermocouple made of a conductive tip (Metal A) and a metallic sample (Metal B). The hot junction lies at

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Nomenclature

Δ	difference between two values
T	temperature in Celcius degree
V	voltage in Volt
S_A	Seebeck coefficient of material A in V/°C
S_{A-B}	Seebeck coefficient of the couple $A-B$ in V/°C

Table 1
Seebeck coefficients of selected metals [11].

Metal	S at 273 K (μV/K)
NiCu	−37.3
Ni	−19.5
Au	+1.8
Cr	+18.5

the contact point between the tip and the sample, while the cold junction is at room temperature, giving the possibility to perform ultra-local temperature measurements. The resulting voltage is given by:

$$\Delta V = \int_{T_0}^{T} S\cdot \mathrm{d}T \tag{1}$$

which can be rewritten as $\Delta V = S_{A-B}\cdot\Delta T$

S_{A-B} is the Seebeck coefficient difference between the two metals.

The Seebeck coefficients of relevent materials are given in Table 1.

3. Experimental set-up

Fig. 2 shows the schematic of the experimental set-up. The conductive probe is made of a 3-cm-long, 500-μm-diameter metal wire. The end of the wire is then electrochemically processed to obtain the tip. To this aim, we use a 1 mol/L KCl solution and a platinum electrode. The applied DC voltage is between 3 and 5 V. The tip radius depends on the etching conditions, and is in the 1–20 μm range. The 3 cm length allows maintaining the other end of the wire at room temperature during the measurement. Fig. 3 shows an example of a tip obtained by this fabrication process.

A specific sample is then fabricated for both calibration and validation of the temperature measurements. It is made of two parts.

The measurement part of sample (Fig. 5) consists of five 150 nm thick, 250 μm width strips of gold. A 1 μm thick silica layer is then deposited by magnetron sputtering on the metal tracks to obtain an electrical isolation. These strips are heated by Joule effect, they are energized by a DC voltage between 1 and 20 V and their resistances are 60 Ohms in average. We obtain a temperature variation of 0–150 °C. Finally over the silica layer, a 200 nm thick strip of chromium or gold is deposited. This strip is the second hand of the thermocouple. One part of this strip is heated by Joule effect and a second part is maintained at room temperature by a Peltier cooler. The Peltier cooler allows to modify the temperature with a DC current and we can obtain with this system a range of temperature of 100 °C. This second part of this strip serves as a basis for the calibration part of the sample (Fig. 4). It is connected by a thin metal strip to another chromium coated area, this latter being maintained at room temperature for reference. The instantaneous temperature of the surface layer is controlled with a K-type microthermocouple.

4. Experimental results and discussion

4.1. Experimental measurement of Seebeck coefficients

The first step is to characterize the Seebeck coefficients of samples and tips using the calibration part of the sample. The metallic tip is put in contact with the layer and the contact temperature is changed by the Peltier cooler. The electrical potential is measured for different temperature values varying between 0 °C and 120 °C.

Results have been obtained with different thermoelectric couples. We have used two metallic wires: nickel and constantan. Different samples have been investigated such as chromium and gold. The Fig. 6 presents EMF variations obtained with the nickel and constantan tips on chromium and gold samples versus the temperature. As expected, the EMF voltage variations detected in all cases linearly depend on the temperature variations. All curves cross the temperature axis at room temperature (22 °C). The experimental average of the Seebeck coefficients for thermocouples Cr/NiCu, Au/NiCu, Cr/Ni and Au/Ni are respectively 38.5, 30, 19 and 11 μV/°C. In literature tables, for bulk metals, these coefficients are respectively equal to 55.8, 39.1, 38 and 20.3 μV/°C. These differences can be mainly accounted for by the fact that one part of our thermocouples is made of thin layers. Their behavior and properties are therefore very different from those of a bulk thermocouple. To check the validity of these results we successively measure the EMF between a thin Cr layer and two different bulk metals, namely Ni and NiCu. The EMF is proportional to the Seebeck coefficients and is equal to ($S_{Ni} - S_{Cr}$) on the one hand and to ($S_{Ni\text{-}Cu} - S_{Cr}$) on the other hand. The difference between these experimental Seebeck coefficients hence gives the Seebeck coefficient of the bulk couple NiCu/Ni. The obtained experimental value is around 19.5 μV/°C while it is 17.8 μV/°C in the literature. The quite good agreement between these two values confirms that the main cause of the error previously mentioned is linked to the thin Cr layer itself. There is still, however, a small difference between the bulk experimental coefficient and theoretical. This can be partly explained by the dependence of the temperature value on the

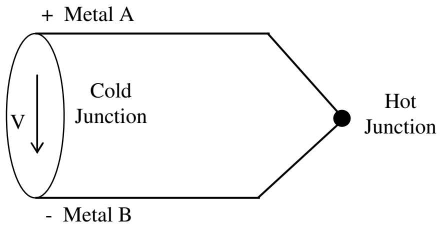

Fig. 1. Basic principle of the thermocouple.

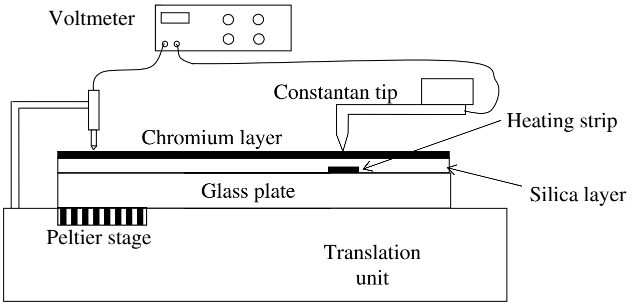

Fig. 2. Experimental set-up to measure temperature using an intrinsic thermocouple.

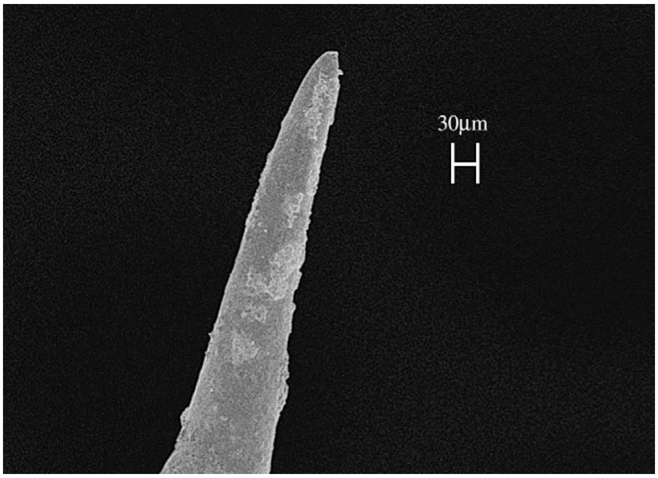

Fig. 3. Example of a microtip fabricated through an electrochemical process.

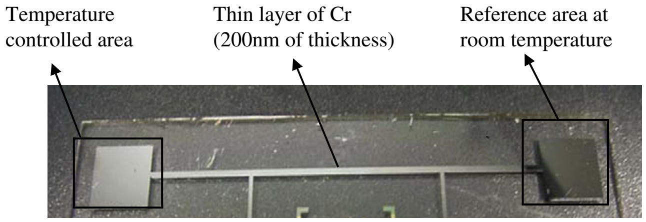

Fig. 4. Calibration part of the sample with thin layers of Cr.

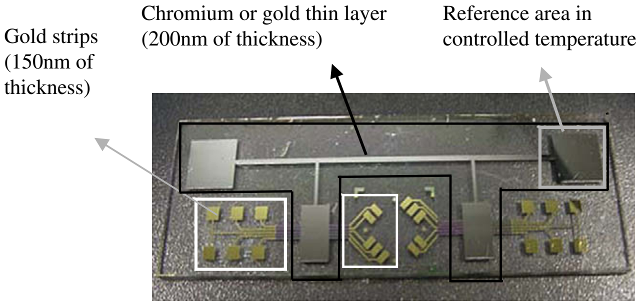

Fig. 5. Measurement part of the sample with a heating strip.

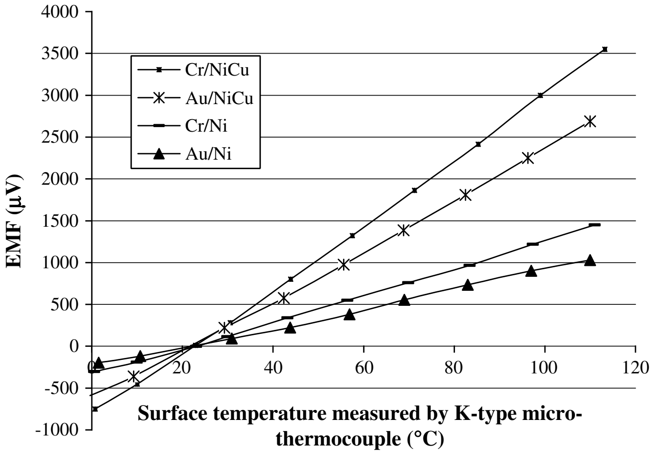

Fig. 6. EMF detected between different layers and nickel and constantan tips.

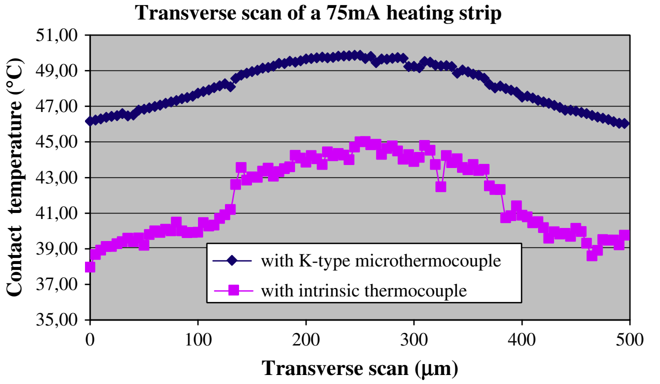

Fig. 7. Transverse scan of an electric strip heated by a 75 mA DC intensity. Comparison of the results obtained with a constantan tip and a K-type microthermocouple.

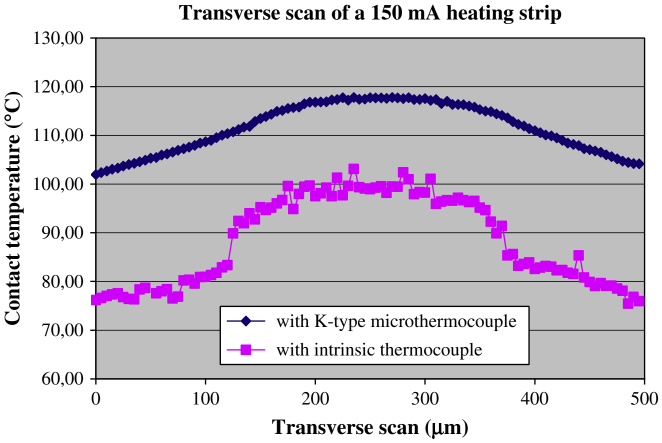

Fig. 8. Transverse scan of an electric strip heated by a 150 mA DC intensity. Comparison of the results obtained with a constantan tip and a K-type microthermocouple.

contact force. This latter has an influence on the EMF voltage value and cannot be controlled with this set-up at this stage. The development of an experimental set-up to control and quantify this influence is currently in progress. Moreover, the tips are etched using an electrochemical process and this operation could slightly modify the Seebeck coefficients.

4.2. *Application: temperature measurement of a heating track*

For this application, the measurement part of the sample is investigated. A metal strip is heated by Joule effect with two different DC current 75 mA and 150 mA. A nickel tip is put in contact with the chromium layer near the heating strip. This contact forms the hot junction of the intrinsic thermocouple. On the reference area, the EMF is taken with a retractable electrical pin. The heating strip is scanned transversely to measure the temperature gradient. The measurement results are compared with those obtained after scanning the heating strip with a K-type microthermocouple (wire diameter: 12.5 µm) made by L. Thiery [12] in the same conditions. The temperature difference measured by the two microsensors is reported in Figs 7 and 8. The first comment which can be done by analysing these experimental results concerns the average temperature detected in the two cases. The measurements exhibit a difference of 20 °C on the same sample depending on the thermocouple actually used. The error

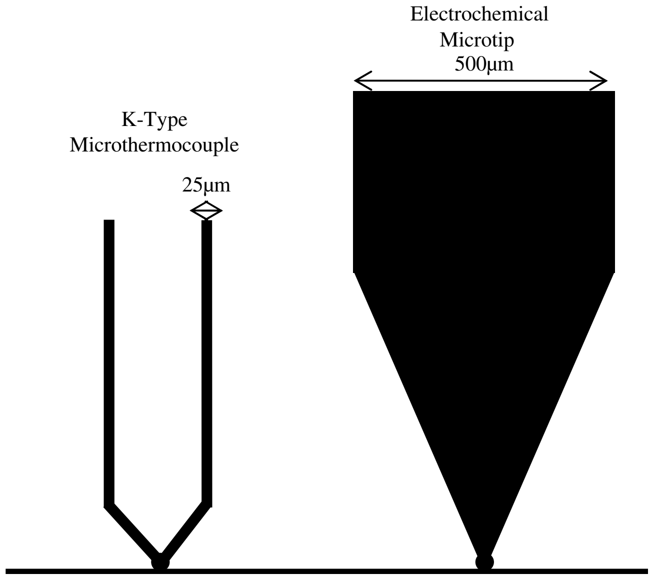

Fig. 9. Comparison of dimensions between a microtip and a K-type microthermocouple.

measurement is partly related to the heat transfer mechanism between the tip and the sample. Fig. 9 compares the dimensions and the volume of the two probes. In the case of the intrinsic thermocouple, the "cold" volume in contact with the "hot" sample is large compared to the case of the K-type thermocouple. As the heat transfer between the surface and the probe is mainly due to convection (the solid–solid contact contribution stands for only 20% of the overall thermal exchange), the temperature of the surface decreases when the "cold" volume increases. This, along with the contact force, can account for the temperature gap observed between the two sensors.

If the absolute temperature measurement itself still needs to be improved, the results presented in Figs 7 and 8 clearly demonstrate that the thermo-spatial resolution obtained with the intrinsic thermocouple is higher. The experimental data show that the strip boundaries are not clearly revealed by the scans obtained with the K-type thermocouple conversely to those demonstrated by our intrinsic thermocouple method. In this last case, the sensing area is only defined by the solid–solid contact zone while the sensing part of standard thermocouple is the entire weld volume. Therefore the averaging effect over the measurement is much more significant in the case of the K-type thermocouple and explains the better precision on the profile detection when using the intrinsic technique.

5. Conclusion

This communication reports on preliminary results concerning the use of an intrinsic thermocouple to perform ultra-local temperature measurements. The intrinsic thermocouple consists of an EFM conducting tip put in contact with a conducting sample and uses the well-known Seebeck effect. The experimental data demonstrate that is possible to measure the relative temperature variation of the sample in contact with a conductive tip by detecting the net EMF voltage. We have seen that this technique allows to achieve a better spatial resolution than some smallest K-type thermocouple in the same conditions. These preliminaries results are stimulating and further investigations are in progress concerning this method in order to completely analyze the behavior of this intrinsic thermocouple. This technique opens new prospects for providing a complete characterization tool for use in applied materials research, quantitative thermal microscopy and micro-thermal analysis, to explore heat transfer phenomenon at small lengths and timescales.

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