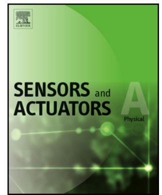
Sensors and Actuators A 214 (2014) 1–6

Contents lists available at ScienceDirect

Sensors and Actuators A: Physical

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



Standard-based direct calibration method for scanning thermal microscopy nanoprobes

Grzegorz Wielgoszewski*, Michał Babij, Roman F. Szeloch, Teodor Gotszalk

Wrocław University of Technology, Faculty of Microsystem Electronics and Photonics, Division of Metrology of Micro- and Nanostructures, ul. Z. Janiszewskiego 11/17, PL-50372 Wrocław, Poland

ARTICLE INFO

Article history:
Received 31 October 2013
Received in revised form 24 March 2014
Accepted 25 March 2014
Available online 18 April 2014

PACS:
68.37.Ps
07.79.Lh
81.70.Pg
06.20.fb

Keywords:
Scanning thermal microscope
Calibration
Temperature measurement

ABSTRACT

A direct calibration method, which is based on international temperature standards, developed for scanning thermal microscopy (SThM) nanoprobes is presented. The idea of calibration is intended mostly for use with thermoresistive SThM nanoprobes and is based on referencing the tip resistance to melting or freezing points of materials, which are contacted directly by the SThM tip. Particularly, in the presented experiment the gallium melting point is used, which is a defining fixed point of the International Temperature Scale of 1990 (ITS-90). Other points suitable for the SThM calibration are suggested, which makes the presented attempt the first step toward linking the quantitative SThM experiments with international temperature standards and therefore envisaging the traceability of nanoscale temperature measurements.

© 2014 Elsevier B.V. All rights reserved.

1. Introduction

Scanning thermal microscopy (SThM) is a variation of atomic force microscopy (AFM), which enables nanoscale investigation of thermal effects, including local mapping of temperature and thermal energy dissipation. The main advantage of SThM results from the fact that, while using the AFM set-up, it enables simultaneous recording of images of surface topography and the thermal properties. Provided a suitable nanoprobe is used, spatial resolution of less than 100 nm is achievable, combined with temperature measurement resolution of less than 0.1 K [1,2]. First ideas of using thermal effects for nanoscale imaging and investigation of thermal properties were reported over two decades ago [3,4] and were followed by AFM-based mapping of thermal properties few years later [5]. Since the beginning, the main challenge of the SThM technique was to develop a suitable and reliable high-resolution nanoprobe. Various kinds of SThM probes were developed, among which thermoresistive ones were the most common [1,6–15]. All SThM experiments reported to date showed a possibility of qualitative nanoscale investigation of thermal effects using various technical solutions. Nevertheless, not all of them provided well-founded quantitative data on thermal properties, e.g., a quantitative absolute temperature map. As the SThM method is recently becoming more common in industry research, it is important to develop reliable calibration methods for SThM nanoprobes. Similarly, access to such calibrated nanoprobes may lead to achieving breaking scientific conclusions, regarding e.g. nanoscale thermal transport in novel materials, such as graphene or "more-than-Moore/beyond-CMOS" microelectronic structures [16]. The above-mentioned thermoresistive probes enable SThM operation in two modes: passive and active. The passive SThM mode (P-SThM) means temperature measurement, based on tip resistance changes. The active SThM mode (A-SThM) is used for imaging of the thermal conductivity contrast. In this mode the heat generated by the current flowing through the probe is dissipated differently according to thermal resistance of the scanned material and the tip-surface thermal contact conditions. Moreover, an A-SThM experiment may be conducted in two modes: with constant current or constant tip temperature.

Abbreviations: SThM, scanning thermal microscopy; P-SThM, passive-mode scanning thermal microscopy; A-SThM, active-mode scanning thermal microscopy; AFM, atomic force microscopy; μTA, microthermal analysis; ITS-90, the International Temperature Scale of 1990; BPh, benzophenone (diphenylmethanone).

* Corresponding author. Tel.: +48 71 320 3202; fax: +48 71 328 35 04.
E-mail address: Grzegorz.Wielgoszewski@pwr.edu.pl (G. Wielgoszewski).

http://dx.doi.org/10.1016/j.sna.2014.03.035
0924-4247/© 2014 Elsevier B.V. All rights reserved.

Table 1
Defining fixed points of ITS-90, which may be considered in SThM calibration [18].

No. (ITS-90)	Defining fixed point	Temperature	
		T_{90} (K)	t_{90} (°C)
9	TP[a] of water	273.16	0.01
10	MP[a] of gallium	302.9146	29.7646
10a[b]	*MP of benzophenone*	*321.85*	*48.7*
11	FP[a] of indium	429.7485	156.5985
12	FP of tin	505.078	231.928
13	FP of zinc	692.677	419.527

[a] TP – triple point, MP – melting point, FP – freezing point.
[b] MP of benzophenone may be considered as one of secondary reference points suggested by Bedford et al., BPh being one of organic substances mentioned in Table 5 in [19]. This material has already been used be Nelson et al. as a specimen [33]. In the experiment described in this paper, the used melting point was verified using an SRS DigiMelt MPA161 device.

The details of these methods are described elsewhere [17]. In this paper, we present a direct calibration method to be used with SThM nanoprobes. While operating the microscope in constant-current active mode, we forced melting of gallium and benzophenone (BPh, also known as diphenylmethanone), after the SThM tip was brought into contact with the surface of these materials. Melting points were used as a reference for calibration of the probe resistance as a function of temperature. The use of gallium melting point linked this calibration method with the International Temperature Scale of 1990 (ITS-90), which is the main internationally recognized reference in temperature measurements [18]. In addition, the other point used as a reference, the melting point of benzophenone, is recommended as one of secondary reference points that complement the ITS-90 (see also Table 1) [19].

2. Calibration of a scanning thermal microscope

2.1. Temperature calibration

Generally, calibration means to establish a relation between the indication of a measurement device and the value of a physical quantity represented by an etalon [20]. In case of thermometers, the main calibration reference is the International Temperature Scale of 1990 (ITS-90) [18]. It consists of 17 defined fixed points within the range from 0.65 to 1358 K and defines the interpolation equations for the calibration curves. Particularly, realization of temperature measurement in the range from 13.8 to 1234.9 K is suggested with use of a calibrated platinum resistance thermometer [18]. For laboratory-based calibration of temperature, standard platinum resistance thermometers (SPRTs) are available [21], while for common use standardized Pt100 (Pt1000) platinum resistors are intended. Many of the thermoresistive SThM probes have the temperature-sensing element made of platinum – therefore they comply with the ITS-90 requirement. However, a calibration method that provides traceability is needed, as discussed in the following subsections.

2.2. Methods used in SThM

Calibration has not always been used in the SThM experiments since the technique was established. In the late 1990s, a method involving a macroscopic stage with a Peltier module was reported to be used for calibration of thermocouple SThM probes [22]. In the following years, various hot-plate stages were designed [2,9,23,24]. Other methods included Raman spectroscopy, transitions temperatures of polymers and mapping using a separate thermocouple [25–28]. So far, the most advanced stage that has been reported is a microfabricated Johnson noise reference, presented by Dobson et al. [29]. However, it needs additional calibration using an integrated thermocouple. The main disadvantage of the hot-plate stages is the fact that the heated area is significantly larger than the tip size. This, at least in ambient conditions, causes parasitic heat transfer, which results in probe resistance changes that are not connected with the temperature of the area just under the tip. What is more, the hot plate temperature has to be determined using another technique, which causes the calibration process indirect. The Raman spectroscopy has a similar drawback, as the sensing area of this method is larger than the tip size; additionally, it may happen that the change in the Raman signal is caused not only by temperature itself but also by temperature-induced changes in the atomic structure of the reference material. Aimed to provide calibration methods for microthermal analysis (μTA), an SThM-based method of polymer investigations [30–36], calibration procedure using known melting points and glass transition points of polymers was also reported [37]. Although a suitable set of reference materials was chosen and good results were obtained, all of the materials were polymers. Therefore, the traceability to international standards could not be easily achieved, as none of the used materials were listed within the ITS-90. As a result, such a method would need at least one more calibration step, e.g. the calibration of an auxiliary temperature sensor or measurement of the polymer-based reference points using a calibrated device.

2.3. Idea of ITS-90 direct calibration

The calibration techniques that are mentioned in Section 2.3 have one common disadvantage: no direct link with the ITS-90. Therefore, we propose the direct SThM calibration method that should provide such traceability of the temperature measurement. Taken into consideration the operating temperature range of most scanning thermal microscopes, especially those working in ambient conditions, 5 fixed points of the ITS-90 may be chosen as a reference for the calibration of SThM probes (see Table 1).

The proposed direct calibration procedure includes the following steps:

1. Placing a reasonable volume of the reference material (gallium, indium, etc.) as a sample in an SThM set-up.
2. Setting up the microscope to operate in active mode with enabled recording of the deflection signal.
3. Bringing the tip into contact with the surface of the reference material.
4. Heating up the tip gradually until a sudden significant change in the deflection signal is noticed. The moment, in which the tip is pushed into the substrate, possible to be noticed in the cantilever deflection signal, denotes that the substance achieved its melting point. The tip resistance and bridge output signal in that time point can be referenced to a certain melting temperature.

It has to be noticed that the suggested ITS-90 reference materials are metals, therefore have relatively high thermal conductivity. This means that the mentioned 'reasonable' volume of the specimen being melted should be low enough to enable being heated up by the SThM tip.

3. Experimental set-up

The described experiments were performed in ambient conditions using a home-made SThM module [23] integrated with a Veeco Nanoman VS microscope with a NanoScope V controller. A KNT-SThM thermal probe (Kelvin Nanotechnology Ltd. [38]; distributed by Bruker AXS as the VITA-DM-GLA probe) was used. The sensing element of the probe was a thin-film palladium wire placed at the end of a silicon nitride cantilever (Fig. 1). It is important that

Download English Version:

https://daneshyari.com/en/article/739326

Download Persian Version:

https://daneshyari.com/article/739326

Daneshyari.com