# Guidelines for high temperature nanoindentation

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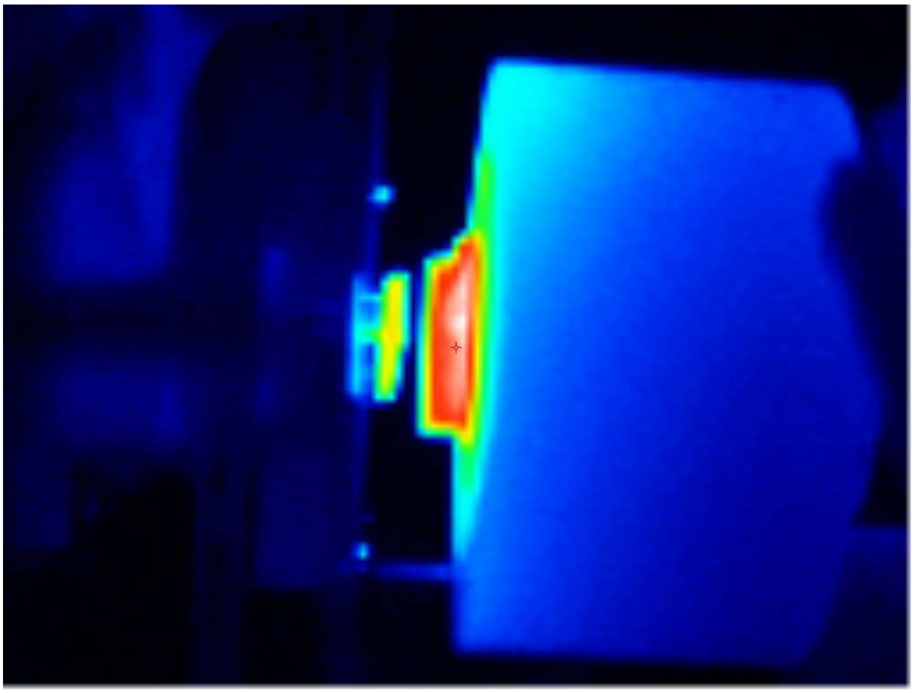

**Abstract:**

Nanoindentation is a powerful tool capable of accurately mapping the mechanical properties across the surface of advanced engineering materials and complex components [1]. High temperature nanoindentation is particularly useful as many applications for advanced engineer materials require mechanical performance at elevated temperatures where the mechanical properties of the product maybe significantly different to those at room temperature. However, despite the increasing importance of high temperature nanoindentation there are many aspects of the technique that if not addressed correctly can lead to erroneous results. These guidelines cover many of these issues, including; the effect temperature has on the calibration of load, displacement, indenter geometry and the frame compliance. Recommendations are also given for accurately determining the temperature at which experiments are conducted.

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## 1. Introduction

High temperature nanoindentation represents an extended capability of the well-established nanoindentation technique allowing specimens to be tested at elevated temperatures [1,2,3]. The basic principles of high temperature nanoindentation are the same as those conducted at room temperature as described in ISO 14577 [4-7]. Hardness, modulus and creep of a specimen can be obtained by applying a known force to an indenter of a known geometry. The indenter is pressed into the specimen at a controlled rate while continuously measuring the displacement and load applied to the indenter. The mechanical properties of the specimen are then obtained from the load-displacement curves [8]. Standard procedures for conducting these tests at room temperature have been produced [1] describing how to calibrate the instruments [5,6] and conduct indentation experiments in different materials including metals, ceramics and coatings [7]. There are, however, additional issues that must be considered at elevated temperatures including, the effect the temperature has on the load and displacement calibrations (sections 3 and 4, respectively), its effect on the frame compliance and indenter geometry (section 4) and how to determine the actual temperature of the experiments (section 5). However, provided that these issues are taken into account it has been found in the current project that uncertainties similar to those obtained at room temperature (modulus ±5%) can be achieved.

## 2. Load calibration

Load calibration is necessary to establish the force that is applied to the indenter tip during the experiment. Load can be calibrated using a number of different techniques the choice of which will depend mainly on the type of instrument being used. The simplest method of calibration is to use a series of calibrated masses which can be compared to the load voltage from the instrument. Conducting the load calibration at different temperatures can however result in significant differences in the calibration coefficient (Figure 1). One method of reducing the effect of temperature on the instrumentation is to surround the specimen and indenter with a thermal shroud (Figure 1). Closing the shroud prevents heat from the hot-stage effecting the instrumentation reducing the effect elevated temperatures have on the load calibration. The results shown in figure 1 demonstrate the need to protect the instrumentation from as much heat as possible and to calibrate the load at the exact temperature and under the same conditions that the experiments are to be conducted.

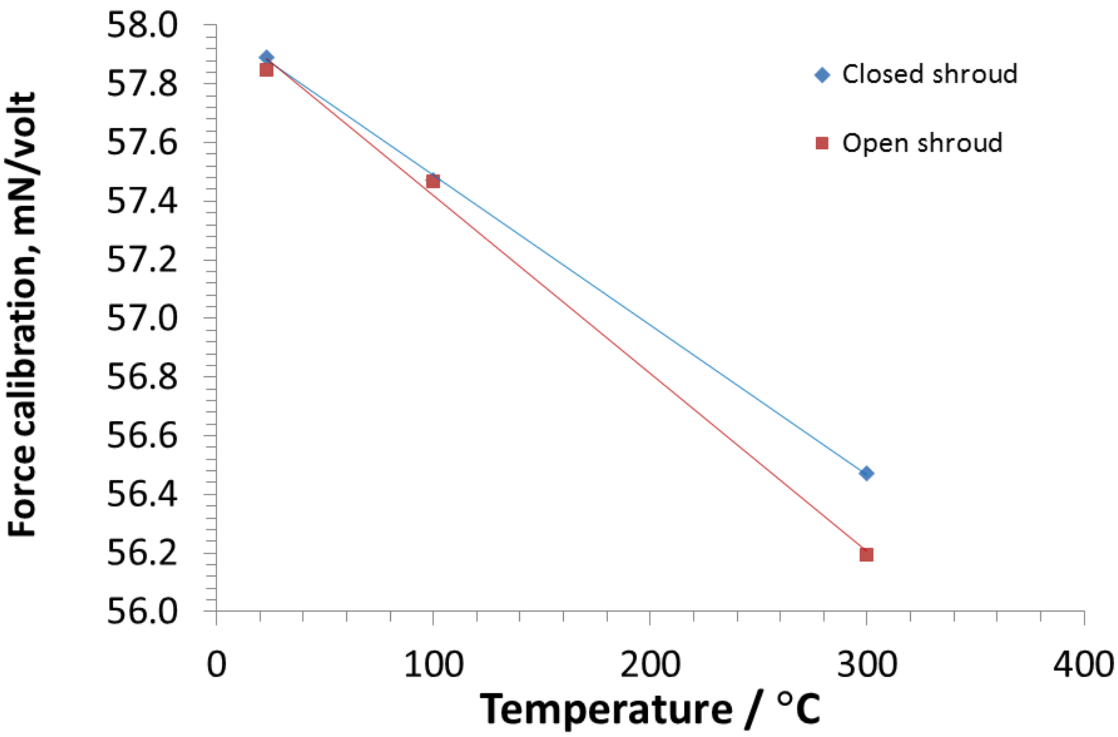

Figure 1 Load calibration as a function of temperature with two different experimental setups involving an open thermal shroud (red) and a closed shroud (blue).

## 3. Displacement calibration

The procedure used to calibrate the movement of the indenter tip during the experiments also depends significantly upon the type of instrument that is used. One of the most accurate methods of calibrating displacement is to measure the movement of the indenter tip using a differential “Jamin” optical interferometer. This allows the output voltage of the instrument's displacement capacitor plates to be comparing directly to the displacement measurements from the interferometer. Calibrating the displacement over a range of different temperatures from room temperature up to 500°C it can be seen (Figure 2) that there is a significant decrease in the calibration constant with temperature, demonstrating the need to calibrate the displacement at elevated temperatures.

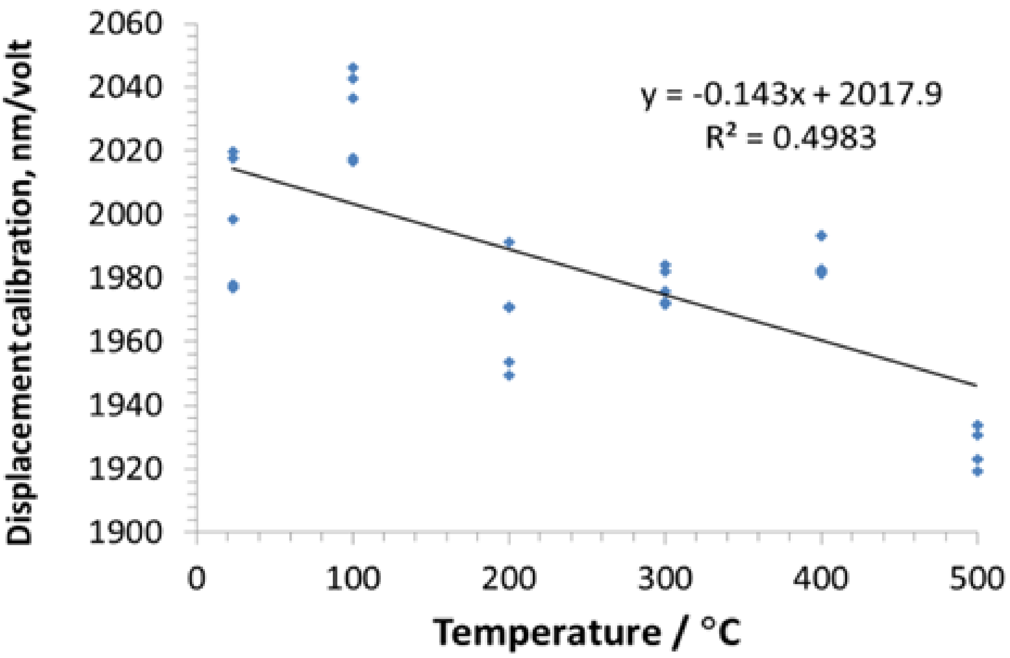

Figure 2 Influence of temperature on the displacement calibration of a nanoindentation

## 4. Frame compliance and area function calibrations

“Frame compliance” that is the stiffness of the instrument and “area function” the contact area of the indenter tip as a function of depth are most commonly calibrated using reference materials. ISO 14577 [5-6] recommends freshly polished tungsten for the frame compliance and fused silica for the area function. In principle this technique can be easily used at elevated temperatures, for unlike alternative techniques such as scanning the indenter tip using an AFM the reference materials can be easily indented at elevated temperatures. The one drawback of using reference materials is that certain materials such as tungsten cannot be used at elevated temperatures as their surfaces can easily oxidise at elevated temperatures. Alternative thermally stable reference materials have therefore been developed for conducting the two reference material method at elevated temperatures. The effects of temperature on the area function and frame compliance of the NPL instrument are shown in figures 3 and 4. As can be seen, for our particular instrument, the frame compliance and area function are not particularly temperature sensitive; however this is unlikely to be the case for most instruments. It is therefore essential to check the calibration of both the area function and frame compliance at the temperature the experiments are to be performed.

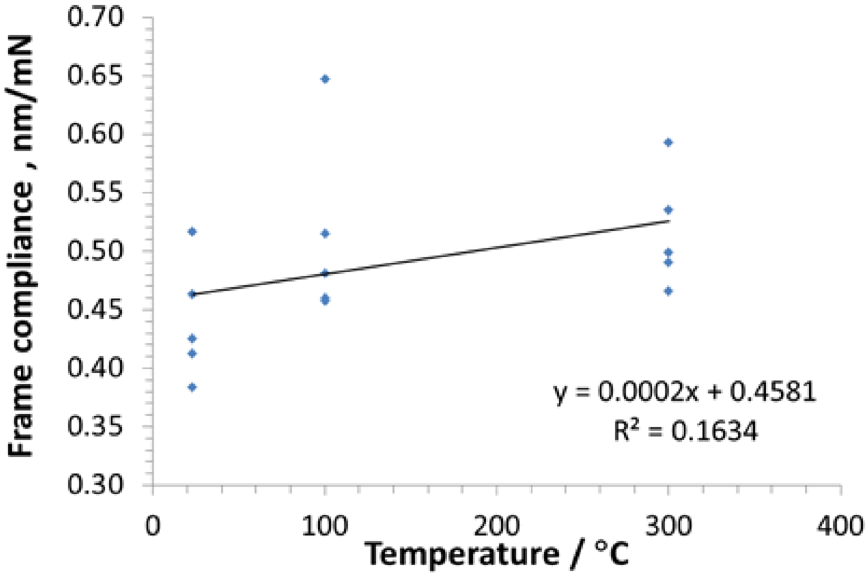

Figure 3 Frame compliance as a function of temperature from room temperature to 300°C.

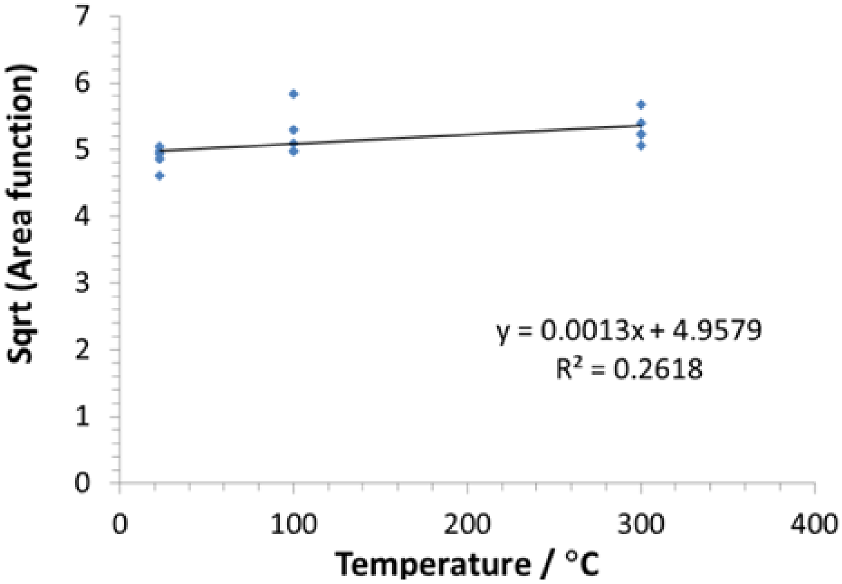

Figure 4 Area function as a function of temperature from room temperature to 300°C.

## 5. Temperature

When examining a sample at elevated temperatures it is important to note that due to thermal gradients there is inevitably going to be a difference between the nominal temperature (i.e. the temperature set up on the temperature controllers) and the real temperature at the surface of the specimen beneath the indenter. This is due to the location of the thermocouples controlling the heaters in relation to the indenter and the sample. This will depend essential on the design of the instrument but also upon the thermal properties of the specimen and the indenter assembly. To compare the actual temperature of the specimen beneath the indenter tip to the nominal temperature, indentations should be conducted into a thermocouple attached to the specimen surface. This enables the actual temperature of the indenter tip to be measured directly from the thermocouple on the specimen surface. As can be seen (Figure 5) the actual temperature of the specimen surface can be significantly lower than the nominal temperature set on the instrument. The difference between the nominal temperature and the actual measured temperature is less significant in highly conducting specimens such as copper and steel and most significant in ceramics (JGC118 and JGG007) and polymeric materials which act as thermal insulators.

The temperature of the sample surface can be measured during an actual experiment by attaching a thermocouple to the edge of the sample allowing the material to be indented at the centre of the specimen. This can be highly accurate for thin, highly conductive materials such as copper, steel or aluminium with uncertainties in temperature of less than 1%. However, care must be taken using this technique with thermal insulators such as polymers and ceramics as temperature can vary significantly across the surface of the specimen. The use of cements/adhesives to attach the thermocouple to the surface may also significantly affect the measured temperature.

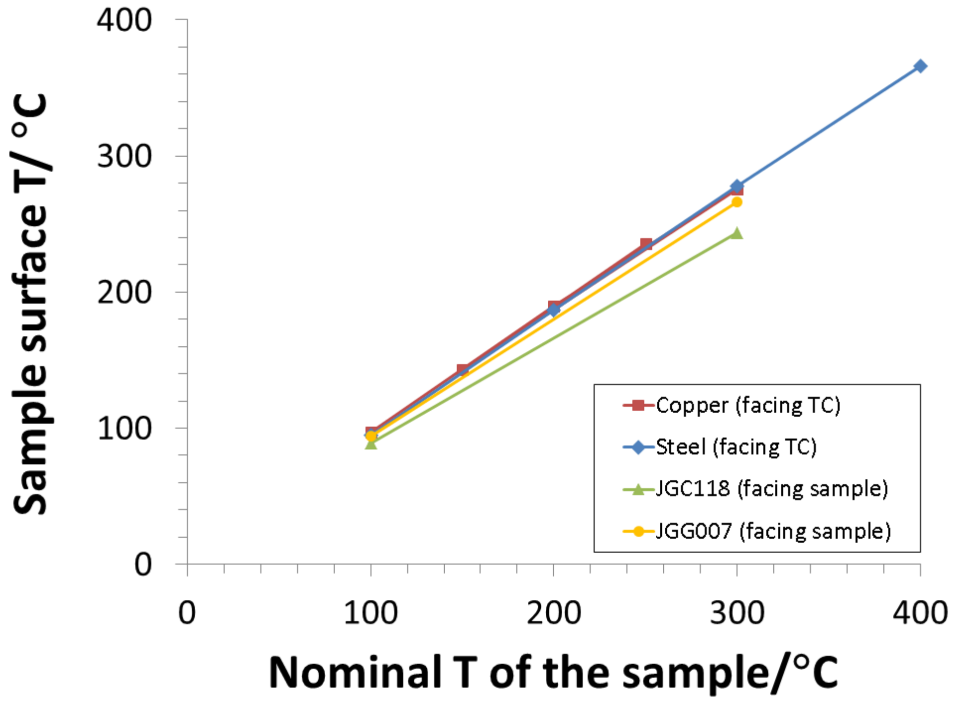

Figure 5 Sample surface temperature as a function of the nominal temperature set in the temperature controller of the sample.

In addition, to accurately measuring the temperature it is also necessary to achieve a stable temperature over the timescale of the experiment and to minimise any mismatch between the temperature of the sample and the indenter which could lead to the formation of thermal temperature gradients. For instruments were the nominal temperature of the indenter tip and the specimen stage can be set independently it is advisable to match the temperature of the tip to that of the specimen to avoid a thermal gradients forming when the two are brought into contact. This can be achieved by indenting into a thermocouple attached to the surface of the specimen and continuously monitoring the temperature. If the indenter and specimen surface are at the same temperature there should be no variation in the temperature as the indenter approaches and enters the thermocouple attached to the specimen surface.

## 6. Conclusions

The basic technique used in high temperature nanoindentation is the same as that outlined in ISO 14577 for tests conducted at room temperature [4-7]. However, additional issues have to be considered when conducting nanoindentation at elevated temperatures including, the effect temperature has on:

- Load calibration
- Displacement calibrations
- Frame compliance
- Area function

Furthermore, care must be taken to calibrate the temperature of the sample and the indenter tip to ensure that: the actual temperature beneath the indenter is known within a known uncertainty; that there is a minimal mismatch between the temperature of the specimen and that of the indenter; and that the specimen is thermally stable over the timescale of the experiment.

References

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5 ISO 14577: 2002 Metallic materials - Instrumented indentation test for hardness and materials parameters Part 2: Verification and calibration of testing machines

6 ISO 14577: 2002 Metallic materials - Instrumented indentation test for hardness and materials parameters Part 3: Calibration of reference blocks

7 ISO 14577: 2002 Metallic materials - Instrumented indentation test for hardness and materials parameters Part 4: Test method for metallic and non-metallic coatings

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