

# Final Publishable JRP Summary for IND13 T3D Thermal design and time-dependent dimensional drift behaviour of sensors, materials and structures

#### Overview

This project developed approaches for measuring small changes in the accuracy of sensors, and in the dimensions and properties of materials and structures, used in precision engineering. The project developed new measurement equipment, facilities, modelling approaches, and standardised procedures, which will be used by a range of European industries to enhance their precision engineering capabilities to develop higher-performance, internationally competitive products.

### Need for the project

Precision engineering is central to maintaining and promoting the international competitiveness of European companies in a range of industries, including electronics, aerospace, semiconductors, and nano-materials. These industries rely on ultra-precision production techniques and sophisticated measurement instruments to develop ever smaller and higher-performance products.

However, such fine levels of precision are vulnerable to even small changes in the dimensions and properties of sensors, materials and production equipment, reducing the levels of accuracy and precision that can be achieved. Such changes occur through time, and can be caused by temperature fluctuations in the production environment, and mean that precision engineering equipment requires regular re-calibration, halting production, and that it must often be maintained in costly temperature-controlled laboratories.

The production efficiency and international competitiveness of a range of European industries will be enhanced though the development of more stable and temperature-resistant materials and measurement devices, supporting the development of increasingly stable precision engineering equipment.

### Scientific and technical objectives

The goal of this project was to develop methods and devices to measure small changes in the accuracy of precision engineering sensors, materials and structures, caused by temperature changes and over prolonged periods of time.

Objective 1 developed an instrument for measuring changes in the dimensions of materials, whilst objective 2 developed methods for measuring the effects of temperature on the long-term physical properties of materials. Objective 3 assessed changes in the long-term properties of a range of industrial materials and different joint types. Objective 4 developed self-correcting (self-calibrating) temperature measurement system for room temperatures. Objective 5 developed improved thermal models to better understand and predict the flow of heat through precision engineering equipment. Objective 6 set up a database of project results for end-users, whilst objective 7 produced a Good Practice Guide for the development of temperature-resistant precision engineering equipment.

- 1. Development of optical interferometric measurement equipment for the determination of dimensional drift with a measurement uncertainty of 10 pm to 100 pm (dependent on the timescale from minutes to weeks) over sample lengths of 0.05 m and durations up to one week.
- 2. Development of an improved indentation method (with traceable calibration at elevated temperatures and a measurement uncertainty analysis) for the measurement of hardness and indentation creep of samples in the nanometre range at elevated temperatures. Investigations of the thermal dependence of hardness and creep of materials with uncertainties about 1 nm in the range Ambient to >100 °C, including establishment of corresponding design rules of materials and joints. Investigations of the thermal dependence of hardness and creep of materials with uncertainties about 1 nm in the range ambient to >100 °C, including establishment of corresponding design rules of materials and joints.

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- 3. Measurement of time and temperature dependent behaviour of samples by optical interferometry with an uncertainty below 0.5 nm for measurement length up to 300 mm. The time scale of the measurements can be over a year and the temperature range allows measurements from 15 °C to 30 °C. The setup additionally allows measurement inhomogeneity of sample stability and dilatation.
- 4. Development of self-calibrating resistance temperature sensors by fixed points near 20 °C using alloys. To enable improved temperature measurement and control electronics in regard of sensor compatibility, control parameter determination and ease of use.
- 5. Development of improved thermal modelling
- 6. Setup of a database for measurement results of the JRP with information on stability, thermal dilatation and hardness of material samples, joint structures, sensors and actors
- 7. Good Practice Guide for developing temperature insensitive precision engineering measurement and tool machines. This includes the selection of appropriate materials and joining technologies as well as the placement of unavoidable heat sources in the machine and of the selection of temperature sensors in precision engineering.

#### Results

1. Development of optical interferometric measurement equipment for the determination of dimensional drift with a measurement uncertainty of 10 pm to 100 pm over sample lengths of 0.05 m and durations up to one week.

Optical interferometers can be used to create high-resolution images, suitable for measuring changes in the shape and dimensions of materials. An existing optical interferometer system needed to be adapted to make it sensitive and accurate enough to measure picometre-scale changes (one trillionth of a metre) over timescales of up to a week.

VSL, the Dutch NMI, developed an optical interferometer suitable for making picometre measurements of dimensional changes. Comparison measurements were performed against standard samples to calibrate measurements, and particular attention was paid to the temperature stability of the instrument. The baseline performance of the interferometer was determined using a double dead-path measurement, where both the sample and refractometer cell were removed and tested. The achievable uncertainties up to now depends on the stability of the weather conditions. The measurement uncertainty of the interferometer ranges from five picometres for one second intervals, and up to 28 picometres over an hour.

To achieve the objective by improving measurement uncertainties over longer time spans (from hours to weeks), a vacuum chamber for the interferometer was successfully designed and manufactured and will be in operation soon. This vacuum setup will also extend the measurement capabilities by allowing for bulk modulus measurements by controlling the pressure in the chamber. An included temperature control of the vacuum chamber also improves the capability for determining the thermal expansion coefficient (CTE).

2. Development of an improved indentation method (with traceable calibration at elevated temperatures and a measurement uncertainty analysis) for the measurement of hardness and indentation creep of samples in the nanometre range at elevated temperatures.

Indentation creep testing can be used to assess changes in the mechanical properties of material samples. To accurately measure mechanical changes caused by temperature, methods were needed to: (1) calibrate measurements made with existing indentation creep equipment at higher temperatures, and (2) identify and quantify the variables that contribute to measurement uncertainty (an uncertainty budget).

NPL, the UK's National Measurement Institute (NMI), developed a new method to calibrate nano-indentation equipment at elevated temperatures, including measurements of displacement, frame compliance and area function. An uncertainty budget was established using the procedures for determining uncertainty detailed in



the BIPM <u>Guide to the Expression of Uncertainty in Measurement</u> (GUM), incorporating both uncertainties introduced by the measurement device and the environment in which the device is used. The research revealed the two most important sources of uncertainty were caused by uncertainty over the size of the area of contact between the instrument indenter and the sample, and differences in sample stiffness. To address these, a method was devised to accurately estimate the area of contact from measurements of indenter depth, using an area function determined by scanning the indenter tip using atomic force microscopy.

The objective was achieved, as the calibration approach and uncertainty budget allowed measurements to be made from room temperatures up to 300°C, with an overall uncertainty of approximately 5%. Guidelines and a scientific paper describing the procedures for calibrating high-temperature nano-indentation instruments have been published by NPL.

3.Measurement of time and temperature dependent behaviour of samples by optical interferometry with an uncertainty below 0.5 nm for measurement length up to 300 mm. The time scale of the measurements can be over a year and the temperature range allows measurements from 15 °C to 30 °C.

Once the optical interferometer had been developed in objective 1, it was used to assess changes in the properties and dimensions of material samples caused by time and temperature.

30 samples were manufactured, including samples with soldered, glued, and screwed joints to test joint stability. The objective was achieved, as sub-nanometer measurements of stability and dimension changes were successfully performed. As the properties and behaviour of the standardised samples is now known, they can now be used as transfer standards for other laboratories to assess the accuracy of their precision engineering and measurement equipment.

Additionally, based on the experience gained during this objective, PTB, the German NMI, can now offer sample preparation as a service. The samples can be used as transfer standards to allow R&D labs and instrument/equipment manufacturers to calibrate their measurement devices, as the performance of each sample is known and has been documented. The samples can be produced to meet specific environmental requirements, with parameters such as pressure, humidity, and gas composition tailored to customer needs.

4. Development of self-calibrating resistance temperature sensors by fixed points near 20 °C using alloys. To enable improved temperature measurement and control electronics in regard of sensor compatibility, control parameter determination and ease of use including a verification of long time stability of thermocouples. Accurate and reliable temperature measurements are required in industrial settings, between 15°C and 30°C, to measure and manage thermal effects in high-precision production. A system was needed that could self-calibrate (self-correct), and that was stable over a time frame of years, without the need for maintenance or demounting. Although such accurate, self-calibrating industrial temperature sensors have recently been developed for high-temperatures, equivalent self-calibrating room-temperature sensors were not available.

A temperature measurement system was developed that incorporated multiple measurement sensors, with both platinum resistance thermometers and thermocouples. The performance of 32 thermocouples, made from different materials and from different manufacturers, was tested, including the effects of stress, motion and heat on their behavior. The temperature measurement system was verified to be accurate to the sub-millikelvin (mK) range (one thousandths of a degree Celsius).

A room-temperature calibration device was developed using materials that melt at known fixed points – water at 0.01  $^{\circ}$ C and gallium at 29.76  $^{\circ}$ C. A range of different gallium-alloys were investigated to find the optimal compound. To validate the performance of the calibration device, were investigated over a period of 18 months, and compared with a 4th cell which was newly manufactured. All four cells produced results that agreed to within  $\pm$  4 mK.

The objective was achieved, as the room-temperature, self-calibrating measurement system was successfully developed. The system was then further enhanced to act as an active temperature control system for precision engineering equipment, capable of reducing heat flow through equipment to maintain its stability. Control software was developed for the thermocouples, and different types of control algorithms, including PID and Model Prediction Control, have been implemented. Using a preamplifier developed at PTB, an uncertainty level of 0.2 mK<sub>pp</sub> can be reached at measurement times of below two seconds, approximately a thousand-fold improvement compared to a nanovolt meter.



### 5. Development of improved thermal modelling.

Thermal modelling is required to predict and understand the flow of heat through precision engineering equipment. The project sought to develop improved thermal models for the more effective management of heat, and for the development of increasingly heat resistant equipment.

The project developed reduced thermal models that incorporated only the variables that were found to significantly affect the flow of heat. The model was verified through: 1) a comparison to results from finite element analysis, and 2) experimentally, using a simplified demonstrator setup. The successful modelling approach and results have been reported in a best practice guide. The models were then used to develop control algorithms to increase temperature stability of a form measurement machine and for the optimized placement of cooling elements for a line scale comparator.

As a case study, a novel cooling system was developed for a camera in an ultraviolet microscope, based on a passive cooling method developed by the Ilmenau University of Technology in Germany. The cooling system improved the microscope's stability, and the accuracy of the length measurements the microscope is used to make. A prototype of the cooling device was produced, and verified through modelling. By implementing the improved temperature measurement electronics at the PTB length comparator, combined with the new cooling element for the microscope camera, the measurement uncertainty was successfully reduced. This reduction helps meet customer demands for the calibration of length scales with the highest worldwide accuracy.

# 6. Setup of a database for measurement results of the JRP with information on stability, thermal dilatation and hardness of material samples, joint structures, sensors and actors.

The project has generated and collected data on material and joint performance under various environmental fluctuations, and under time variation from weeks to months. Measurements have covered dimensional, surface (hardness and creep) and thermal variations. These results have been published to support the design of optimised precision tools, industrial equipment, and standards for reference materials. All measurement results from material samples or joints are available in a <u>publically available database</u>, which can be found under <a href="http://projects.npl.co.uk/T3D/publications.html">http://projects.npl.co.uk/T3D/publications.html</a>. The project partners will continue to update with non-confidential measurement results.

# 7. Good Practice Guide for developing temperature insensitive precision engineering measurement and tool machines.

Good Practice Guides based on the project's results have been published on the project website, in the <u>EURAMET Publications Repository</u>, and have been partly published in journals. These guides will help improve the control of production processes, contributing to more efficient, safe and reliable precision engineering.

## **Actual and potential impact**

### **Dissemination of results**

To promote the uptake of the project's results, outputs were shared broadly with scientific and industrial endusers. 20 papers have, or are in the process, of being published in international journals, 34 presentations have been made at national and international conferences, and three presentations at exhibitions. Two workshops on the project were held during the 2014 European Society for Precision Engineering and Nanotechnology conference in Dubrovnik, and the 2014 IWK conference in Ilmenau. Results have been shared with stakeholder companies, and standards committees governing hardness and nano-measurement science, through 11 in-house workshops. Measurement data from the testing of material and joint samples has been made available through a public database, and experience and results have been described in good practice guides and 26 publications available on the project website and the <u>EURAMET Publications</u> Repository.



### Early impact

The project's results are allowing precision instrument manufacturers to further improve the performance and stability of their products.

For instance, the <u>Fraunhofer IOF</u> research institute, an unfunded research partner, needed methods to measure the time and temperature dependent properties of their products. Fraunhofer IOF have developed and refined specialized inorganic joining techniques, including silicate bonding, Au/Sn laser-based thin-film soldering, and Solderjet Bumping, to assemble optical components for ultra-high-precision instruments. Fraunhofer IOF produced sample materials for the project, which were assessed for time scales of over one year, and for temperatures between 10 °C and 40 °C. The results of this testing have provided Fraunhofer IOF with a detailed assessment of these joining techniques, and have allowed them to guarantee the stability of their joining techniques to their customers.

A prototype room-temperature measurement system was developed for objective 4, and is now available to precision engineering tool manufacturers. The system can be used to assess the effects of temperature on their products, and to help develop more thermally-stable instruments. For example, SIOS Meßtechnik GmbH, a manufacturer of precision laser-interferometric measuring instruments, used the prototype system to monitor temperature changes at their facility and to understand their instruments' thermal stability. Using this data, they have improved an ultra-precise dimensional measurement instrument by eliminating small uncertainties related to temperature fluctuations. The product will allow their customers to measure small components such as microelectronic, micromechanical or optical objects, to sub-nanometre precision, without the need for highly controlled environments.

During the development of the prototype temperature measurement system, the project team commissioned instrument manufacturer Magnicon GmbH to develop a high-sensitivity SQUID amplifier suitable for room-temperature measurements. SQUID amplifiers are used to amplify very small changes in voltage to levels that can be measured with a high degree of accuracy, therefore were judged to be suitable amplifiers for the prototype system, which measured temperature changes in terms of voltage changes (via thermocouples). However, before the project, there were no commercially available SQUIDs suited to room-temperature measurement. Through contributing to the project, Magnicon developed a room-temperature SQUID amplifier, a completely new application for their technology, and have since launched new room-temperature products.

The project also commissioned MPro GmbH, a developer of custom electronics, to contribute to the development of the room-temperature measurement system. Since project completion, MPro have begun to develop a commercial version of the system to be launched in 2017, expanding MPro's product line and making the project's prototype more widely available to other National Measurement Institutes, research facilities and manufacturers.

<u>MicroMaterials</u>, a leading manufacturer of nanomechanical test instruments, has also adopted procedures developed in the project as alternative techniques for monitoring and controlling specimen and indenter temperatures for their instruments.

Early impact will also be achieved through the development of new and improved calibration services for the measurement of thermal dilatation and dimensional stability for material samples, and for joints, sensors and actuators. Additionally, NPL is now able to perform indentation measurements for a greater temperature range, LNE can now provide industrial customers with more accurate thermally optimised cylindricity comparator measurements, and PTB can offer sample preparation (for secondary calibration), particularly for smaller companies.

### Potential future impact

The knowledge, techniques and instruments developed in this project are available to European industry, and will allow end-users to accurately measure and manage thermal and time-dependent drift in their precision engineering processes, and to develop more time and temperature-stable precision engineering equipment. These developments will reduce time, energy consumption and costs in manufacturing, and will ultimately support the development of higher-performance, internationally-competitive products.



### List of publications

- [1] The EMRP project "Thermal design and dimensional drift", J. Flügge, E. Beckert, N. Jennet, T. Maxwell, D. Petit, S. Rudtsch, J. Salgado, M. Schalles, R. Schödel, D. Voigt, M. Voigt, Proceedings 12. EUSPEN International Conference, June 2012, Vol. I, p. 315 (2012)
- [2] Metrology to reduce thermal effects and drift in precision engineering, J. Flügge, PTB Mitteilungen, Vol. 122 (2012), Heft 4, p. 71
- [3] Untersuchung binärer eutektischer Legierungen für die Kalibrierung von Berührungsthermometern im Raumtemperaturbereich, D. Heyer, B. Polomski, S. Rudtsch, Proceedings Temperature 2013, p. 123-127 (2013)
- [4] Dimensional stability validation and sensor calibration with sub-nanometer accuracy, D. Voigt, R.H. Bergmans, Proceedings International Conference on Space Optics 2012, paper ID 123 (2012)
- [5] Investigation on the thermal behaviour of an ultra-high precision system used in dimensional metrology, K. Bouderbala, H. Nouira, M. Girault, E. Videcoq, J. Salgado, D. Petit Proceedings 14. EUSPEN International Conference, June 2014, Vol. I, p. 305-308 (2014)
- [6] Parametric investigation of Linear Quadratic Gaussian and Model Predictive Control approaches for thermal regulation of a high precision geometric measurement machine, E. Videcoq, M. Girault, K. Bouderbala, H. Nouira, J. Salgado, D. Petit, Applied Thermal Engineering, Vol. 78, p. 720-730 (2014)
- [7] Interferometric characterization of dimensional and thermal stability of materials and joints, H. Lorenz, R. Schödel, Proceedings 14. EUSPEN International Conference, June 2014, Vol. I, p. 301.304 (2014)
- [8] Interferometric measurement of dimensional and thermal stability of joints, H. Lorenz, R. Schödel, Proceedings SPIE, Instrumentation, Metrology, and Standards for Nanomanufacturing, Optics and Semiconductors VIII, Vol. 9173, 9173OB (2014)
- [9] Picometer resolution heterodyne interferometry for short to medium term dimensional stability measurements, A. van de Nes, D. Voigt, Proceedings 58th IWK, Ilmenau Scientific Colloquium, paper ID 25282 (2014)
- [10] Thermometry Fixed-Points Based on Binary Eutectic Alloys, S. Rudtsch, Proceedings 58th IWK, Ilmenau Scientific Colloquium, paper ID 25281 (2014)
- [11] Reduction of thermal effects on precise dimensional measurements, M. Schalles, J. Flügge, R. Köning, Proceedings 58th IWK, Ilmenau Scientific Colloquium, submitted, paper ID 25271 (2014)
- [12] Absolute interferometric measurement of the dimensional and thermal stability of joining techniques, H. Lorenz, R. Schödel, Proceedings 58th IWK, Ilmenau Scientific Colloquium, submitted, paper ID 25271
- [13] Phase topography-based characterization of thermal effects on materials and joining techniques, H. Lorenz, E. Beckert, R. Schödel, Applied Optics, Vol. 54, Issue 8, pp. 2046-2056 (2015)
- [14] A dedicated calibration standard for nanoscale areal surface texture measurements, J. R. Koops, M. van Veghel, A. van de Nes, Microelectronic Engineering, Vol. 141, pp 250-255 (2015)
- [15] Stability of thermocouples near room temperature for measurement and control applications in precision engineering, J. Flügge, M. Voigt , Precision Engineering, submitted
- [16] A virtual lateral standard for AFM calibration, R. Koops, M. van Veghel, A van de Nes, Microelectronic Engineering, Vol. 153, pp 29.36 (2016)
- [17] PCM-shielding of precision measuring equipment by means of latent heat, M. Schalles, T. Fröhlich, J. Flügge, Precision Engineering, , Vol. S0141-6359 (15) 00056-2 (2015)
- [18] Thermal investigation on ultra-high precision apparatus for dimensional metrology at the nanometre level of accuracy, K. Bouderbala, H. Nouira, M. Girault, E. Videcoq, J. Salgado, International Journal of Precision Engineering and Manufacturing, Vol. PP, Issue 99 (2015)
- [19] Good Practice Guide: Traceable calibration of instrumented indentation instruments as a function of temperature, A.S. Maxwell, L. Mera Alvarez, http://projects.npl.co.uk/T3D/publications.html



- [20] Good Practice Guide: Measurement of relative length changes in sample up to 50mm with picometre uncertainty over durations up to one week, A. van der Ness, http://projects.npl.co.uk/T3D/publications.html.
- [21] Good Practice Guide: Optimising the thermal stability of joints including atleast two examples of exemplarily selected samples, H. Lorenz, http://projects.npl.co.uk/T3D/publications.html.
- [22] Good Practice Guide: Thermal modeling of precision engineering equipment, M. Schalles, http://projects.npl.co.uk/T3D/publications.html.
- [23] Good Practice Guide: Locating and control cooling devices on thermal sensitive instruments, M. Schalles, http://projects.npl.co.uk/T3D/publications.html.
- [24] Good Practice Guide: Developing instruments with minimal thermal sensitivity, M. Schalles, http://projects.npl.co.uk/T3D/publications.html.

# The following publications are in preparation:

- [25] Pictometer resolution dimensional stability measurements using precision interferometry, A. van de Nes, D. Voigt, H. Lorenz, R. Schödel
- [26] Challenges to achieve isothermal contact between the indenter and the sample in thermal equilibrium, H. Xiaodong, N. Jennet, T. Maxwell, Philosophical Magazine

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