Final Publishable JRP Summary for IND03 HighPRES
High pressure metrology for industrial applications

Overview

The use of new high-pressure manufacturing techniques by European industry was being constrained by an inability to accurately measure pressures from 1 GPa – 1.5 GPa. This project developed a capability to measure pressures up to 1.6 GPa, from which industrial users can calibrate their pressure measurement devices for accurate measurements up to 1.5 GPa. The techniques developed will allow European industry to use high-pressure techniques to manufacture durable, high-performance products, and to meet challenging sustainability requirements.

Need for the project

High-pressure manufacturing techniques are used widely in European industries to develop tough, high-performance products, and efficient manufacturing processes. The techniques, including autofrettage, hydroforming and isostatic pressing, are key to securing the future competitiveness and environmental sustainability of a wide range of European industries, from automobiles, to pharmaceuticals, petrochemicals, and material fabrication.

To achieve further required gains in product performance, European manufacturers need to be able to accurately measure and control pressures above 1 GPa. For instance, to develop cleaner and more efficient diesel vehicles, required by EU regulation Euro 6, greater diesel fuel efficiency is required. An increase in fuel injection pressure in vehicle engines would result in more efficient combustion and lower exhaust emissions. This requires more durable engine components that would need to be hardened during manufacture at pressures up to 1.5 GPa.

However, the pressure transducers used in industry are not sufficiently accurate at such high pressures, as the calibration capabilities of the European NMIs are limited to pressures up to 1 GPa. European industry therefore needs the NMI community to develop an infrastructure for the accurate measurement of pressures up to 1.5 GPa, including the definition of a primary measurement standard, and techniques to calibrate industrial pressure transducers against this standard.

Scientific and technical objectives

Eight objectives were defined to achieve the overall goal of developing a European NMI capability to accurately measure, and provide calibration services for pressures up to 1.5 GPa. Objectives 1 – 6 support the development of a pressure balance system accurate up to 1.6 GPa, to be used as the primary standard, and include the identification of suitable materials and the modelling of their properties. Objective 7 addresses the development of pressure transducers accurate up to 1.5 GPa as transfer standards, to allow industrial transducers (pressure measurement devices) to be calibrated to the primary standard, whilst objective 8 covers optimum calibration procedures:

1. Establish finite element methods (FEM) for stress-strain analysis of elastic and nonlinear elastic-plastic deformation, as well as of contact processes in pressure measuring piston-cylinder units, and high-pressure components at pressures above the current level of 1 GPa.
2. Determine the pressure-distortion coefficient of pressure balances up to 1.6 GPa, taking into account real shape and elastic properties of the piston-cylinder assembly, pressure dependent density and viscosity of the pressure-transmitting medium, and pressure distribution along the piston-cylinder gap.
3. Determine the mechanical properties, including elastic constants and hardness of high-strength steels and tungsten carbide materials to be used for components of high-pressure balances, pressure transducers and pressure-generation systems.
4. Dimensional characterisation of high-pressure piston-cylinder assemblies.

Report Status: PU Public

European Metrology Research Programme
Programme of EURAMET

The EMRP is jointly funded by the EMRP participating countries within EURAMET and the European Union

5. Recommend potential high-pressure transmitting liquids to be used in the pressure balances and industrial applications up to 1.6 GPa.
6. Create a primary pressure standard for pressures up to 1.6 GPa, with a relative expanded uncertainty as low as 5×10^{-4} (0.0005) GPa, its metrological characterisation, realisation of the pressure scale up to 1.6 GPa, providing pressure calibration service up to 1.5 GPa.
7. Provide transfer standards and calibration methods for the range 0.1 GPa to 1.5 GPa, and optimisation of modern 1.5 GPa pressure transducers.
8. Recommendations, norms and standards for high pressure components and applications.

Results

1. Establish finite element methods (FEM) for stress-strain analysis of elastic and nonlinear elastic-plastic deformation, as well as of contact processes in pressure-measuring piston-cylinder units, and high-pressure components at pressures above the current level of 1 GPa.

Pressure balances are the most accurate pressure measurement systems, and the most suitable for defining the 1.6 GPa primary standard. FEM software is needed that can model the deformation of pressure balance materials and components (such as the piston-cylinder assembly), as deformation will reduce the performance and accuracy of the measurement system.

The objective was met through developing FEM software to perform stress-strain, elastic-plastic deformation, and contact process analysis of the high-pressure components of pressure balances. The FEM software, and new fluid flow models, were used to devise an optimal design for a 1.6 GPa primary pressure standard, and to predict its performance properties. Commercial high-pressure components appropriate for the pressure balance and transducers were identified through modelling of their application ranges and parameter modifications.

2. Determine the pressure-distortion coefficient of pressure balances up to 1.6 GPa, taking into account real shape and elastic properties of the piston-cylinder assembly, pressure dependent density and viscosity of the pressure-transmitting medium, and pressure distribution along the piston-cylinder gap.

The pressure-distortion coefficient is the main source of measurement uncertainty in pressure balance systems. To develop a 1.6 GPa pressure balance, the pressure-distortion coefficient must be accurately calculated to limit measurement uncertainty.

The objective was met by using the FEM software to successfully determine the pressure-distortion coefficient for the piston-cylinder assemblies (PCA) of the new 1.6 GPa primary pressure standard, incorporating the elastic constants of the PCA materials, dimensional properties of pistons and cylinders, and pressure dependent densities and viscosities of pressure-transmitting liquids. Fluid flow models were developed and applied to the piston-cylinder gap to describe the pressure distribution along the piston-cylinder clearance at pressure differences from zero to 1.6 GPa. From these analyses, the key properties and working parameters of the 1.6 GPa primary pressure standard were defined with high accuracy, guaranteeing the uncertainty of the measured pressure will be below the target of 0.0005 GPa, up to pressure of 1.6 GPa.

3. Determine the mechanical properties, including elastic constants and hardness of high-strength steels and tungsten carbide materials to be used for components of high-pressure balances, pressure transducers and pressure-generation systems.

The target pressure of 1.6 GPa is higher than the tensile strength of tungsten carbide and most high-strength steels, the materials usually used to build pressure balances and transducers. Appropriate materials must be identified to develop accurate 1.6 GPa pressure balances and 1.5 GPa transducers.

Strain gauge measurements at pressures up to 1.6 GPa were performed on commercial high-pressure tubing, connectors and valves, to assess and their performance and suitability. Elastic constants of steels were accurately measured using resonant ultrasound spectroscopy (RUS) and strain gauge methods, with results from both demonstrating sufficient agreement. Elastic constants of tungsten carbide materials were measured by RUS. Correlations between the elastic constants, density and chemical composition were derived. The hardness of high-pressure components was determined, and the effect of thermal treatment of the sealing lenses investigated. With these results the objective was achieved – appropriate commercially available high-pressure components were selected for use in the 1.6 GPa pressure balance and 1.5 GPa transducers. The results also allow these components to be used more efficiently and accurately in high-pressure measurements and processes in NMIs, calibration laboratories and industry.

4. Dimensional characterisation of high-pressure piston-cylinder assemblies.

The dimensions of the piston-cylinder assemblies (a key component of pressure balances) also influence the pressure-distortion coefficient. Dimensions need to be measured to the nano-scale (a billionth of a metre), to produce a low-uncertainty 1.6 GPa pressure balance.

The objective was met through the use of state-of-the-art dimensional measurement techniques and numerical procedures to determine the 3D dimensional properties of piston-cylinder assemblies of the new 1.6 GPa primary pressure standard, to an uncertainty less than 50 nanometres. With this data, the contact behaviour of the cylinders and sleeves could be predicted, fluid flow in the piston-cylinder gap could be analysed, and the dependence of the effective area on pressure determined. The contribution of the dimensional irregularities of the pistons and cylinders to the uncertainty of the pressure realised with the 1.6 GPa primary pressure standard was successfully quantified.

5. Recommend potential high-pressure transmitting liquids to be used in the pressure balances and industrial applications up to 1.6 GPa.

The density and viscosity of potential liquids must be measured to determine their suitability for use in the 1.6 GPa pressure balance, and to model their effect on the pressure-distortion coefficient. Before this project, no NMI worldwide could perform such measurements.

To achieve the objective, a new high-pressure viscometer was developed to determine the properties of the potential pressure-transmitting liquids. Stability of the liquids was assessed up to 1.6 GPa, density and viscosity up to 1.35 GPa, for temperatures between 20 °C – 120 °C. The results were used to model fluid flow in the piston-cylinder gaps of the new 1.6 GPa pressure balance, and to recommend high-pressure transmitting liquids for industrial applications.

6. Create a primary pressure standard for pressures up to 1.6 GPa, with a relative expanded uncertainty as low as 5×10^{-4} (0.0005) GPa, its metrological characterisation, realisation of the pressure scale up to 1.6 GPa, providing pressure calibration service up to 1.5 GPa.

The primary pressure standard is the core measurement reference upon which the calibration service is founded. Results from the previous objectives were combined to develop a 1.6 GPa pressure balance device to act as the primary standard.

A 1.6 GPa pressure generation and control system was designed, manufactured and tested, based on the knowledge gained from objectives 1 to 5. The performance characteristics of the 1.6 GPa pressure balance were determined theoretically, and validated experimentally by measurement against 100 MPa and 1 GPa primary pressure standards and, above 1 GPa, against 1.5 GPa pressure transducers. The objective was achieved through the successful development of a 1.6 GPa pressure balance system to be used to define the primary standard, with an uncertainty estimated to be as low as 0.0001 GPa. Based on this primary standard, a calibration service has been established in the German NMI PTB, for high-pressure measurement instruments, including 1.5 GPa pressure transducers accurate to 0.0005 GPa.

7. Provide transfer standards and calibration methods for the range 0.1 GPa to 1.5 GPa, and optimisation of modern 1.5 GPa pressure transducers.

Pressure transducers are sufficiently accurate to be used as transfer standards, to transfer the primary standard measurement to devices in use in industry or commercial calibration laboratories.

Eight, 0.5 GPa, 1 GPa and 1.5 GPa strain gauge and thin layer piezo-resistive pressure transducers were analysed to determine their performance, including their calibration curves, hysteresis, sensitivity, repeatability, mounting effects, and short and long-term stability. Transfer standards based on the high-pressure transducers were designed, assembled and studied. The objective was achieved when one

1.5 GPa transducer was successfully tested as a transfer standard against the 0.5 GPa and 1 GPa national pressure standards of five NMIs. The results of the experimental characterisation also allowed the manufacturers of the transducers tested to further optimise their transducers for high-pressure applications.

8. Recommendations, norms and standards for high pressure components and applications.

To ensure pressure transducers are effectively calibrated by industrial end-users, the project incorporated procedures developed for calibrating high-pressure transducers in a revised EURAMET guide cg-17 '*Guidelines on the Calibration of Electromechanical Manometers*', due to be approved at the EURAMET TC-M meeting in May 2016.

Actual and potential impact

Dissemination of results

To promote the uptake of the high-pressure calibration service, and other project achievements, results have been shared with scientific and industrial end-users through the publication of 12 papers in international journals (listed in the next section), and presentations at international conferences and committees, including the 50th European High Pressure Research Group (EHPRG) meeting in 2012. During the project, two workshops were run on high-pressure metrology for industrial applications, and a training course on high-pressure measurement and calibration, targeted at research organisations and industrial users. Suitable results have also been made available on the project website http://emrp-highpres.cmi.cz/. The EURAMET guide cg-17 '*Guidelines on the Calibration of Electromechanical Manometers*', has been revised to include the procedures developed for accredited laboratories to provide the new calibration service (to be approved at the EURAMET TC-M meeting in May 2016).

A comparison of NMI high-pressure standards was organised within EURAMET, and resulted in the extension of the Calibration and Measurement Capabilities (CMCs) statements of the participating NMIs, an important step towards harmonising global high-pressure standards. Additionally, the results will be used in the accreditation of calibration laboratories and the on-site calibration of reference high-pressure standards.

Early impact on industry

The high-pressure measurement service is now available for industrial users to calibrate their pressure measurement devices up to 1.5 GPa, enabling more accurate control of high-pressure manufacturing processes. A world-leading manufacturer of high-pressure equipment has used the calibration facility to develop autofrettage machines that operate over high-pressure ranges, which have subsequently been used by manufacturers to develop more durable engine components for the European automotive industry. More durable diesel engines will be able to operate at higher fuel injection pressures, and use fuel more efficiently, reducing diesel engine emissions in compliance with EU requirements.

Producers of high-pressure equipment have shown interest in the project's results and new capabilities, including the technical developments in high-pressure generation and transmission, the new high-pressure working liquids, and the high-pressure transducers, in addition to the practical calibration methods demonstrated on the project's training course. A review of industrial high-pressure measurement needs has also been collated and communicated to the manufacturers of high-pressure transducers, to indicate opportunities for them to optimise their products to satisfy unmet needs. Results of the investigation into strain gauge and thin layer piezo-resistive-based pressure transducers are also available for manufacturers to optimise the accuracy and reliability of their high-pressure transducers.

Potential future impact on industry

The calibration service offered by PTB, developed in collaboration with TUC (Germany), CMI (the Czech Republic), METAS (Switzerland), LNE (France), and SMU (Slovakia), is available for industrial laboratories to gain accreditation for pressure calibration above 1 GPa, to provide high-pressure calibration for industrial end-users in-line with ISO 9001 and other international standards. This service will ultimately allow European industry to measure and control higher pressures more accurately, facilitating the development of higher performance products and more efficient manufacturing processes. High-pressure techniques will promote industrial competitiveness and will contribute to the achievement of required reductions in environmental impact.

List of publications

1. Sabuga W., Haines R., Development of 1.6 GPa pressure-measuring multipliers, *Acta IMEKO*, 2014, Vol. 3, No 2
2. Salama A.D., Sabuga W., Ulbig P, Measurement of the elastic constants of pressure balance materials using resonance ultrasound spectroscopy, *Measurement*, 2012, Vol. 45, 2472–2475
3. Sabuga W., Haines R., Development of 1.6 GPa pressure-measuring multipliers, *Proc. of XX IMEKO World Congress, "Metrology for Green Growth"*, Busan, Rep. of Korea, Sep 9-14, 2012, www.imeko.org/publications/wc-2012/IMEKO-WC-2012-TC16-O1.pdf
4. Pražák D., Sabuga W., Outline of the European joint research project – high pressure metrology for industrial applications, *Program and Book of Abstracts of 50th EHPRG Meeting*, Thessaloniki, Greece, Aristotle University of Thessaloniki, Sep 16-21, 2012
5. Sabuga W., Pražák D., Rabault T., Recent progress in high pressure metrology in Europe, *Europ. Phys. J. - Web of Conferences*, 2014, Vol. 77, http://dx.doi.org/10.1051/epjconf/20147700006
6. Sabuga W., Gluschko A., Konczak T., Development of 1.6 GPa pressure standards, in "Proc. of HighPRES workshop held at PTB Braunschweig, Germany, September 17, 2014", *PTB Bericht*, accepted
7. Salama A.D., Rabault T., FEM analysis of the high-pressure multipliers and components, in "Proc. of HighPRES workshop held at PTB Braunschweig, Germany, September 17, 2014", *PTB Bericht*, accepted
8. Rabault T., Salama A.D., Elastic constant measurements by strain gauges and RUS methods, in "Proc. of HighPRES workshop held at PTB Braunschweig, Germany, September 17, 2014", *PTB Bericht*, accepted
9. Könemann J., Gluschko A., Konczak T., Sabuga W., High-pressure transducers as transfer pressure standards evaluated by a 1 GPa pressure comparison, in "Proc. of HighPRES workshop held at PTB Braunschweig, Germany, September 17, 2014", *PTB Bericht*, accepted
10. Brouwer L., Pressure dependence of viscosity and compressibility of fluids for high pressure applications, in "Proc. of HighPRES workshop held at PTB Braunschweig, Germany, September 17, 2014", *PTB Bericht*, accepted
11. Pražák D., Sabuga W., A common European project "High pressure metrology for industrial applications", *Metrologie*, 2014, Vol. 23, No. 2, 28-29 (in Czech)
12. Sabuga W., Pražák D., Chytil M., Fíra R., Common European research project "High-pressure metrology for industrial applications", *Metrológia a skúšobníctvo*, 2014, 25-27 (in Slovak)

JRP start date and duration:	October 2011, 36 months
JRP-Coordinator: Dr Wladimir Sabuga, PTB, Tel: +49 531 5923230 E-mail: wladimir.sabuga@ptb.de JRP website address: http://emrp-highpres.cmi.cz	
JRP-Partners: JRP-Partner 1: PTB, Germany JRP-Partner 2: CMI, Czech Republic JRP-Partner 3: METAS, Switzerland	 JRP-Partner 4: LNE, France JRP-Partner 5: SMU, Slovakia JRP-Partner 6: TUC, Germany

The EMRP is jointly funded by the EMRP participating countries within EURAMET and the European Union