IND03 HighPRES

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1 Executive Summary

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.

The Problem

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.

The Solution

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.
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 $5x10^{-4}$ (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.

2 Project context, rationale and objectives

2.1 Context

1.5 GPa pressure transducers such as those developed by HBM Ltd, WIKA Instruments Ltd or Dunze GmbH, are used in high pressure applications in the automotive, general engineering, petrochemical, pharmaceutical and food industries. In addition, several reports (e.g. Frost & Sullivan - European pressure sensors and transmitters markets - Oct 2005; ARC Advisory Group - Pressure transmitters - five year market analysis and forecast through 2013 - April 2009), forecast that high pressure sensing will grow from currently 1 % up to 5 % of the total pressure measurement market.

Prior to the start of this project IND03, there was an increasing number of high pressure calibrations and requests for pressure calibration beyond 1 GPa, however adequate standards for pressures higher than 1 GPa (with which modern pressure transducers could be adequately calibrated) were unavailable in the EU. New reference pressure standards were also needed to provide the metrological basis for high pressure technologies, as well as high pressure calibration services, which were needed for companies using new high-pressure technologies to fulfil their quality assurance and safety requirements.

In addition to missing primary pressure standards, no reliable, sufficiently investigated transfer pressure standards for pressures up to 1.5 GPa existed prior to the start of this project. Consequently in industry, measurement properties of pressure transducers calibrated at lower pressures must be extrapolated to higher pressure. However, this approach only allows a vague estimation of pressure outside of the transducers' calibrated range with negative consequences for industrial efficiency and safety. Therefore there was a strong need for metrological services in the pressure range beyond 1 GPa, and for guidelines and recommendations for calibrating high-pressure transducers for industry.

2.2 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:

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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.
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 $5x10^{-4}$ (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.

3 Scientific and technological results

3.1 Establishing finite-element based methods (FEM) for stress-strain analysis of elastic and elastic-plastic deformation in pressure-measuring piston-cylinder units and high-pressure components at pressures above 1 GPa

Determination of 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

Introduction

This work addressed 2 of the project's objectives:

Objective 1. To establish FEM for stress-strain analysis of elastic and elastic-plastic deformation in pressure-measuring piston-cylinder units and high-pressure components at pressures above 1 GPa

Objective 2. To 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

In order to achieve this, the work was divided into four subtasks:

3.1.1. FEM nonlinear analysis of plastic deformation and solid-to-solid contact processes
3.1.2. Analysis of possible design and materials of 1.6 GPa piston-cylinder assemblies (PCAs)
3.1.3. Analysis of elastic-plastic behaviour of tubes, connections, valves and sealing elements assemblies
3.1.4. FEM calculation of pressure distortion coefficient and piston fall rate

3.1.1 FEM nonlinear analysis of plastic deformation and solid-to-solid contact processes

In general high-pressure piston-cylinder assemblies (PCAs) are made of tungsten carbide and operate in the elastic regime. In contrast to this, the components of pressure balances such as tubing, valves and connectors are made of steel and can therefore undergo elastic and plastic deformations when subjected to high pressures. The elastic deformations in PCAs cause a reversible change of their effective area, whereas the elastic-plastic deformation in pressure balance components induces irreversible modifications and can cause their damage. The target pressure of 1.6 GPa is higher than the tensile strength of tungsten carbide and, also, than the elasticity limit of most high-strength steels. Consequently, special supporting arrangements were needed for the design of a 1.6 GPa pressure balance such as jacket pressure and sleeves attached by a shrinking, and this required application of elastic, non-linear plastic-elastic and contact FEM analysis.

FEM software codes were developed independently by project partners PTB and LNE, which described elastic-plastic deformations in cylindrical bodies made of materials with known Young modulus (E), Poisson coefficient (μ) and elasticity limits in the case of axis-symmetric inner loads as high as 1.6 GPa and under release conditions. Both PTB and LNE analysed the same model with its geometrical and material properties, as presented in Table 1. The resulting stress-strain and elastic-plastic deformation were compared between PTB and LNE to verify their calculations and typical results were the FEM calculations of the radial stress, see Figure 1, or of the radial displacement, see Figure 2. Figures 1 and 2 demonstrate the very good agreement between PTB and LNE's results for the FEM calculations of the deformations.

Component	Material	Length [mm]	OD [mm]	ID [mm]	E [GPa]	μ [-]	Yield stress S_y [MPa]	Ultimate stress S_{ut} [MPa]
Tube	Steel	50	19.05	1.8	200	0.3	1053	1216

Table 1. Geometry and elastic properties of the studied model

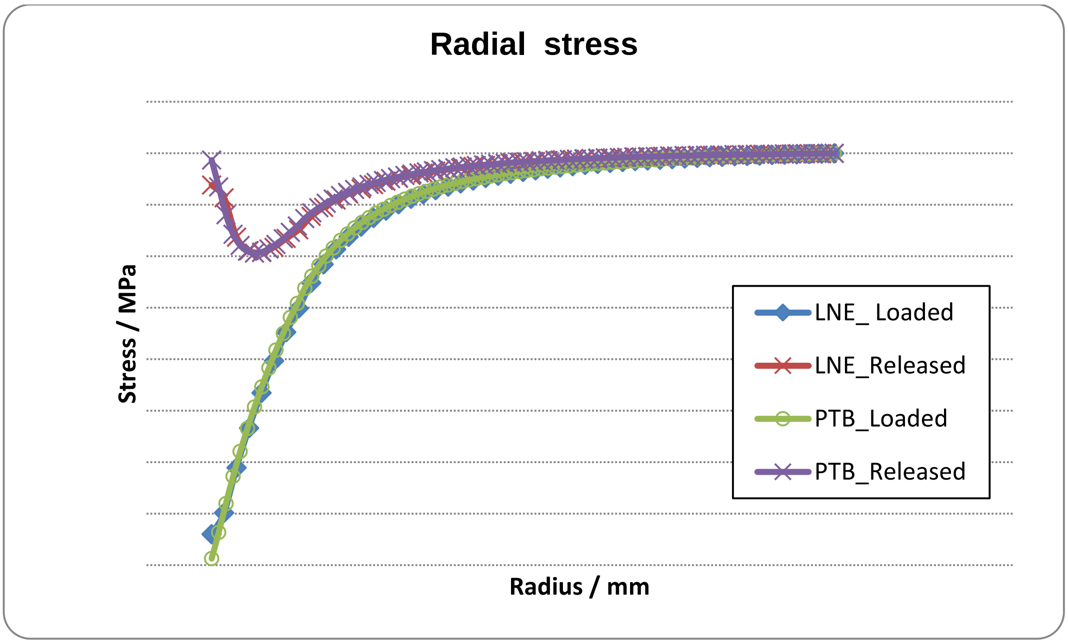


Figure 1. Radial stresses in the tube when loaded with 1.6 GPa and released calculated by PTB and LNE

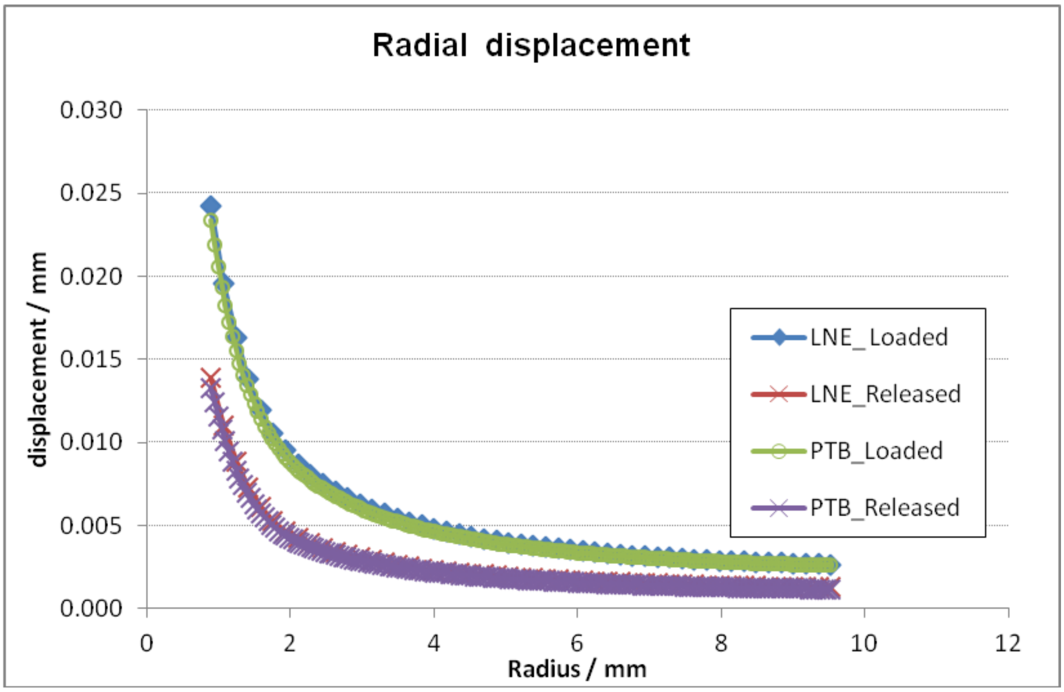


Figure 2. Radial displacements in the tube when loaded with 1.6 GPa and released calculated by PTB and LNE

To describe the contact and accompanying deformation processes which typically take place when tubing or a sealing lens is attached to a cylinder/connector/valve or when sleeves are set on a cylinder when performing thermal shrinking, contact process and plastic deformation analysis was carried out by PTB and

LNE. For such analysis a high pressure tube was assumed to be pressed to the cylinder with an inner pressure of 1.6 GPa acting on both tube and cylinder. Table 2 summarises the elastic properties of cylinder and tube.

Component	Material	E [GPa]	µ [-]	Yield stress S_y [MPa]	Ultimate stress S_{ut} [MPa]
Cylinder	Tungsten carbide	620	0.218	-	-
High pressure tube	Steel	200	0.3	1053	1216

Table 2. Elastic properties of the studied model

The contact pressure between the sealing components indicates whether this connection will withstand the pressure, and Figure 3 shows good agreement between the independent contact analyses of the PTB and LNE.

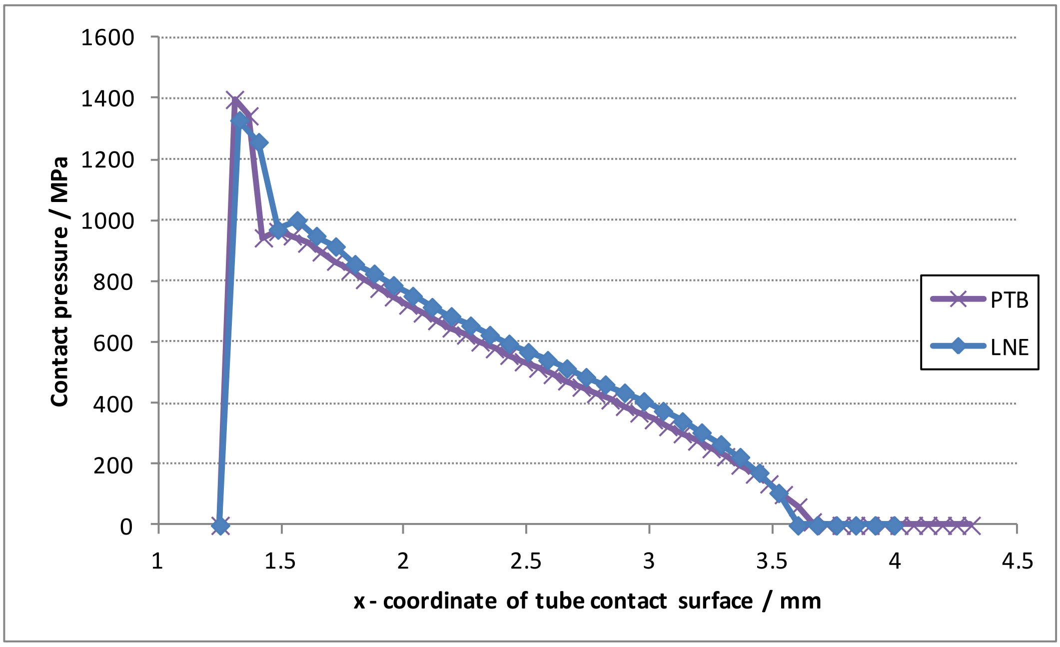


Figure 3. Contact normal stress between the tube and cylinder calculated by PTB and LNE

FEM software codes were established and validated by PTB and LNE, and this allowed for the first time stress-strain, elastic-plastic deformation, positions of boundaries between regions deformed elastically and elastic-plastically, and contact process analysis of the high pressure components of pressure balances. This sophisticated analysis and conclusions were verified by comparisons of the calculation results based on the independent software codes of PTB and LNE. Following this, PTB extended their verified software codes and methodology to three-dimensional analysis to investigate more complex structures of high-pressure connectors and valves (see section 3.1.3). With this new FEM methodology, it was possible to model and analyse novel 1.6 GPa pressure measuring multipliers, and to find out their optimal design.

The collaborative approach of this research enabled the project partners to progress beyond their individual state-of-the-art methods and to find solutions for overcoming material property limitations.

3.1.2 Analysis of possible design and materials of 1.6 GPa piston-cylinder assemblies (PCAs)

Basically, a PCA consists of a piston and a cylinder. The piston is only subject to compressing loads and therefore can be made of tungsten carbide. However, the cylinder is subject to tensile stresses by the measurement pressure and, therefore, it has to be supported from outside to withstand the measurement pressure, of as high as 1.6 GPa. In order to make the cylinder's deformation elastically reversible, the inner core of the cylinder should be made of tungsten carbide, and to support the tungsten carbide core from outside, one or two steel sleeves should be thermally shrunk on the core. Additionally, a jacket pressure should be applied to the side surface of the outer sleeve to compensate the tensile stresses in the tungsten carbide core. Several designs of PCAs were suggested and analysed in the project with the FEM software codes described in section 3.1.1.

In the analysis of the PCAs, three "outside" shapes of the inner sleeve were numerically tested. Typical elastic constants of tungsten carbide and steel are shown in Table 3 and a constant width's gap of between 0.2 µm and 0.5 µm were used in the FEM analysis calculations. Additionally, the calculation assumed large deflection and included contact and plastic capabilities, the latter is required for the tube connected to the high-pressure PCA. As a result, connection of the high pressure tube to the cylinder and deformation of the tube under pressure were studied. A tube tip angle of 59.5° and matching cylinder cone angle of 60° were found to best fulfil the requirements for the high pressure PCAs. With a pressure of 1.6 GPa applied, the tube was moved into the cylinder to provide contact along the whole length of the cylinder cone.

Component	Material	E [GPa]	µ [-]	Yield stress S_y [MPa]	Ultimate stress S_{ut} [MPa]
Cylinder	Tungsten carbide – 6% Co	620	0.218	-	≈ 700
Piston	Tungsten carbide – 10% Co	560	0.218		
Inner & Outer sleeves	Cr-Ni-Mo Steel	200	0.3	1200	1400
high pressure Tube	Austenitic Steel	200	0.3	1053	1216

Table 3. Material properties of the studied model

The decision on the optimal design for the high pressure PCA was made by analysing maximum stresses in the tungsten carbide core and the steel sleeves. Moreover, a low and constant pressure distortion coefficient as well as minimum nonlinearity of the pressure distribution in the piston-cylinder gap was used at the maximum pressure of 1.6 GPa applied on relevant surfaces. Simultaneous to this, a linear and constant pressure distribution in the piston-cylinder gap was applied, as well as a jacket pressure on the outer surface of the high pressure PCA sleeve. The correctness of this sophisticated analysis and the derived conclusions were verified by analysing the same problem based on the independent FEM software packages of PTB and LNE.

PTB developed 3 FEM models of 1.6 GPa PCAs and software codes to model the shrink process. PTB performed calculations for the 3 FEM models and different constant piston-cylinder gap widths. LNE analysed stresses, deformations and the linearity of the pressure distortion coefficients by combining the structural FEM analysis of the high pressure PCA with a hydrodynamic analysis for its piston-cylinder gap. PTB and LNE compared the results obtained with different FEM program codes and then PTB determined an optimal design and gap width for the PCA, as indicated by the FEM analysis, and produced technical drawings for the PCA.

Both the FEM analysis and analytical results demonstrated that the double shrink will compensate to a great extent for the stress produced by the internal pressure of 1.6 GPa. An example of the FEM models of 1.6 GPa piston-cylinder assembly is shown in Figure 4. The excellent agreement between the FEM analysis of PTB and LNE, is shown in Figure 5 and is indicated by the comparison of PTB and LNE's independent calculation of the radial displacement of the inner diameter of the high pressure cylinder of the 1.6 GPa PCA (which was internally loaded with the linear distributed pressure from 1.6 GPa at the inlet and zero pressure at the outlet side of the cylinder and externally loaded with a jacket pressure of 400 MPa).

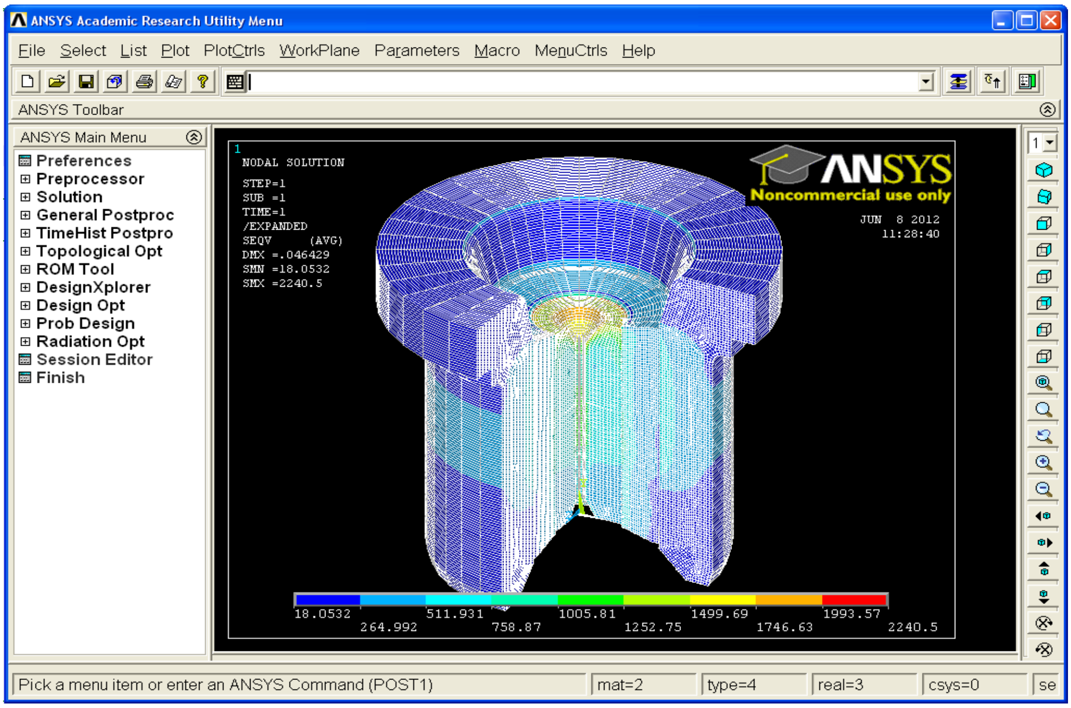


Figure 4. FEM models of the 1.6 GPa piston-cylinder assembly loaded with internal and jacket pressure

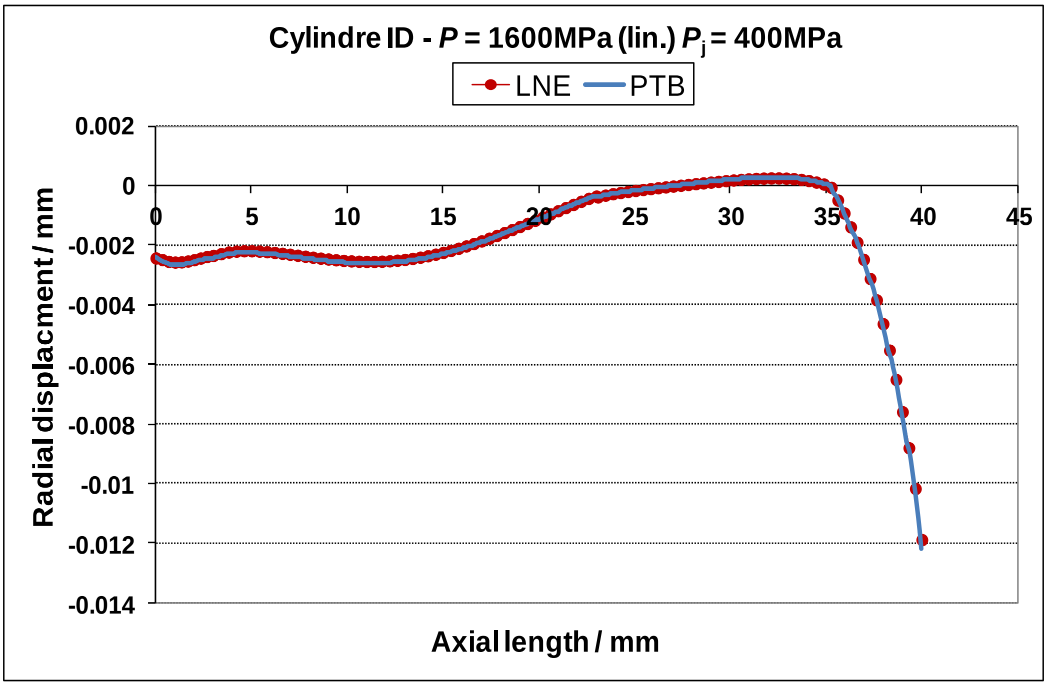


Figure 5. Radial displacement of the inner surface of the 1.6 GPa cylinder loaded with maximum pressure and jacket pressure of 400 MPa

3.1.3 Elastic-plastic analysis of tubes, connections, valves and sealing elements

The aim of this work was a feasibility analysis of the components to be used in the 1.6 GPa pressure balance. As no material for tubing and connections existed which would deform entirely elastically at pressures up to 1.6 GPa without a risk of a rupture, a compromise between possibly high tensile stress and the elongation limit needed to be found.

PTB studied commercially available high pressure tubing to assess its suitability for 1.6 GPa pressure balances. Combinations of tubing or sealing lenses in contact with other components of the pressure balance was analysed in relation to the elastic-plastic properties of the contacting parts and the initial axial pre-stress in the connections. PTB and LNE then developed FEM models of sealing elements. LNE performed calculations and analysis of stresses, deformations and deformed shapes and PTB adjusted dimensional and mechanical properties of the sealing elements to remain leak-tight when loaded by a pressure of 1.6 GPa. The suitability of commercial high pressure connections such as tee, cross, elbow and valves was also studied by PTB using FEM. From the results of this, it was demonstrated that commercial high pressure connections could withstand high pressures of 1.6 GPa.

3.1.4 FEM calculation of pressure distortion coefficient and piston fall rate

The aim of this work was to calculate the pressure distortion coefficient (λ) of the pressure balance, which is its main metrological characteristic, and used to predict its piston fall rate using FEM.

PCA production phase (constant gap width): LNE and PTB developed different FEM models of low pressure PCAs. LNE and PTB performed FEM modelling of high and low pressure PCAs using nominal values of elastic constants of the PCAs' materials. In parallel, project partner CMI developed and applied fluid flow models to the piston-cylinder gap (which had a constant width) using pressure dependent densities and viscosities of pressure-transmitting liquids extracted from the literature available at the time. This was done to describe the pressure distribution along the piston-cylinder clearance at pressure differences from 1.6 GPa to zero. The structural problem of radial elastic distortions in the piston and cylinder was solved using the pressure distribution delivered by the fluid flow analysis taking into account other loads, e.g. jacket pressure, and the constraints in the pressure balance mounting post. Coupling of the piston-cylinder elastic distortion with the fluid flow problem calculations was performed by iterative calculations with algorithms leading to converging solutions at the maximum pressure of 1.6 GPa. PTB calculated pressure distortion coefficients and piston fall rates at different pressures for high and low pressure PCAs of constant width's gap (See Figures 6 to 9) in order to define the target mean gap width at the PCAs production phase.

PCAs metrological characterisation phase (real gap width): Fluid flow models were developed and applied that took into account piston-cylinder gaps of real geometry. FEM modelling of the high and low pressure PCAs were repeated using the elastic constants of their materials, dimensional properties of the pistons and cylinders and pressure dependent densities and viscosities of pressure-transmitting liquids. Fluid flow models were used to describe the pressure distribution along the piston-cylinder clearance of real geometry at pressure differences from 1.6 GPa to zero. The analysis was performed for both high and low pressure PCAs to determine their individual pressure distortion coefficients and, finally, to calculate the pressure dependence of multiplying the ratio of the pressure multiplier as a function of pressure. The maximum pressures of the high and low pressure PCAs were 1.6 GPa and 80 MPa, respectively, and pressure distortion coefficients and piston fall rate calculations at different pressures were determined for real geometry data of the pistons, cylinder bores and sleeves using FEM.

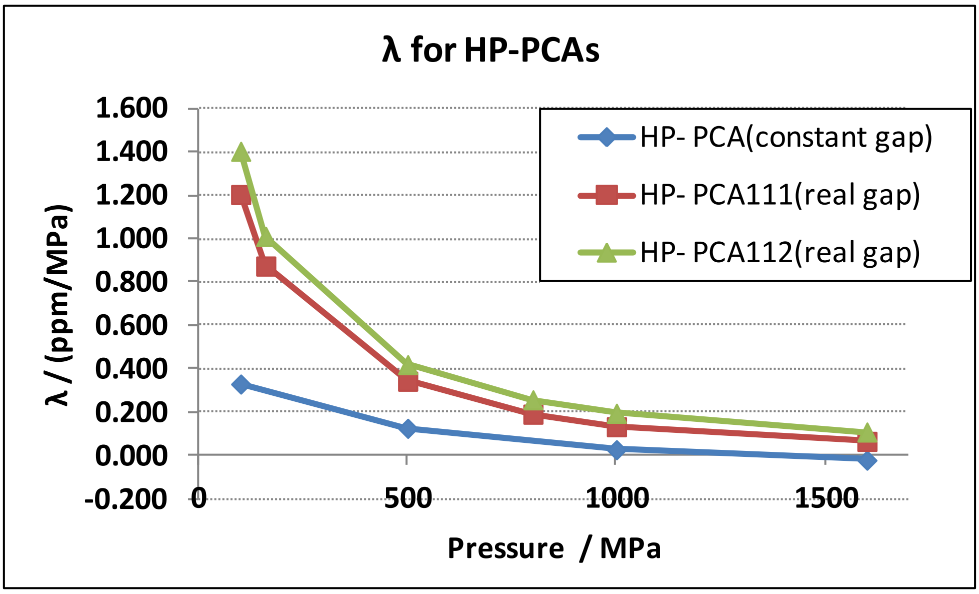


Figure 6. Pressure distortion coefficient of high pressure PCAs of constant and real gaps at different pressures

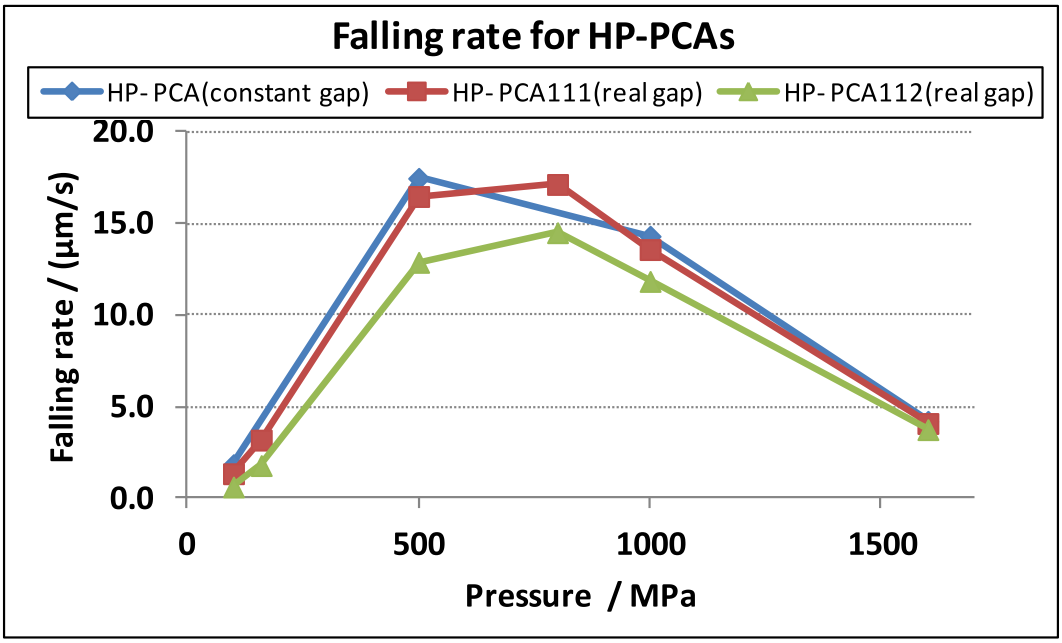


Figure 7. Piston fall rate of high pressure PCAs of constant and real gaps at different pressures

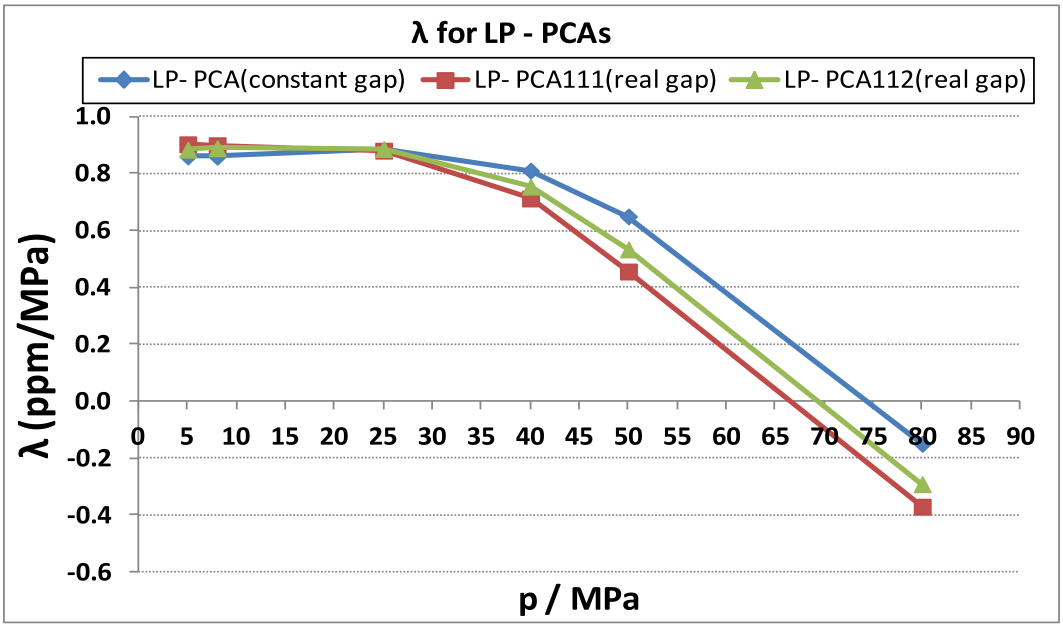


Figure 8. Pressure distortion coefficient of low pressure PCAs of constant and real gaps at different pressures

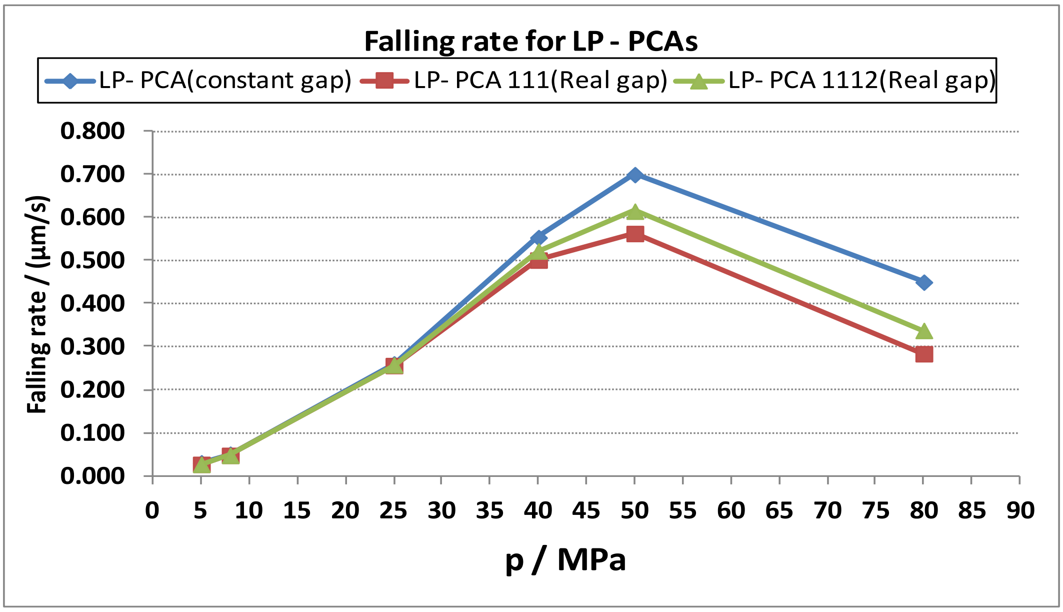


Figure 9. Piston fall rate of low pressure PCAs of constant and real gaps at different pressures

<u>Conclusions</u>

Pressure balances are the most accurate pressure measurement systems, and the most suitable for defining the 1.6 GPa primary standard. However, FEM software was 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.

Objective 1 of the project 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.

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

Objective 2 of the project 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.

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

Introduction

Knowledge of elastic constants of materials used for fabrication of pressure balances is required to describe their behaviour (stresses and strains) under pressure. The literature available for steels was quite reliable when their composition and thermal processing were specified, however the elastic constants of tungsten carbide materials used for piston-cylinders were inaccurate. The reason for this, is due to the sintering from tungsten carbide powder and molten cobalt or nickel as a binder during a high-temperature production process. Therefore, depending on tungsten carbide-to-binder composition, the elastic properties of a sintered material can vary in a broad range from batch to batch even for a nominally identical material. This is also related to the inhomogeneity of the binder distribution and possible porosity and textures in the material, and only measurement of a specific material of a particular PCA can guarantee correctness of its elastic constants. Due to the very high Young modulus of tungsten carbides (about 3 times higher than that of steels) and to the small size of tungsten carbide samples, classical static load machines cannot be used for measurement. Therefore determining the tight connections between tubing and other parts of pressure balances (PCAs, lenses, connectors and valves), the hardness of their materials is important as well as their elastic properties.

This work addressed 1 of the project objectives:

Objective 3. To 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.

In order to achieve this the work was divided into three subtasks:

3.2.1. Strain gauge measurements of elastic and plastic deformations
3.2.2. Elastic constants of tungsten carbides and steels measured by the resonant ultrasound spectroscopy
3.2.3. Hardness measurements on high pressure components

3.2.1 Strain gauge measurements of elastic and plastic deformations

Cylindrical (Cy) and rectangular parallelepiped (RP) samples, of each of the 3 types of high strength steel used/supplied by Harwood (USA), Sandvik (Sweden) and Aubert & Duval (France), as used for cylinder sleeves, high pressure tubing and pressure connections were prepared by PTB and supplied to LNE for strain gauge measurements. The elastic constants of the 6 steel samples (3 Cy samples and 3 RP samples) were measured by LNE and PTB using strain gauge and resonant ultrasound spectroscopy, respectively and the results showed good consistency as shown in Figure 10.

PTB also prepared commercially available high pressure tubing, connectors and valves with strain gauges to carry out further measurements of strain and elastic-plastic deformations at pressures up to 1.6 GPa. To do this, PTB evaluated theoretical and experimental data of strains, and analysed the applicability of the commercial components in 1.6 GPa pressure balances. The evaluation of theoretical and experimental data of strains indicated that the commercial connections can be used up to 1.6 GPa without exceeding the safety limit of the relative plastic deformation to the whole diameter as shown in Figure 11 for HP-160 high pressure tubing. As can be seen in Figure 11, the experimental deformations, when the tube was loaded for the first time (red squares), were in a good agreement with the theoretical prediction. In the repeated load tests (the rest points), the experimental deformations were lower than those during the first load and also lower than the theoretical deformations, which was explained by the autofrettage effect which took place during the first load. The rest stresses produced by the first load considerably compensated the plastic deformation in the subsequent load trials.

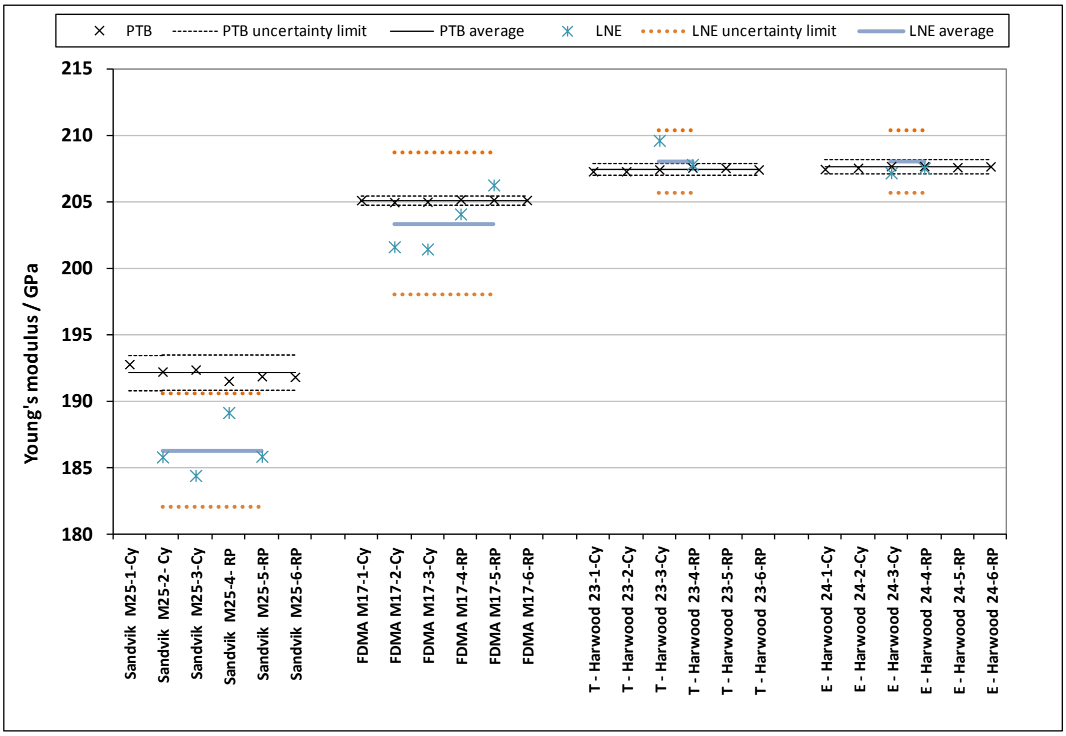


Figure 10. Young's modulus with expanded uncertainties of samples of each of the 3 types of high strength steel, used/supplied by Harwood (USA), Sandvik (Germany) and Aubert & Duval (France), measured by LNE and PTB

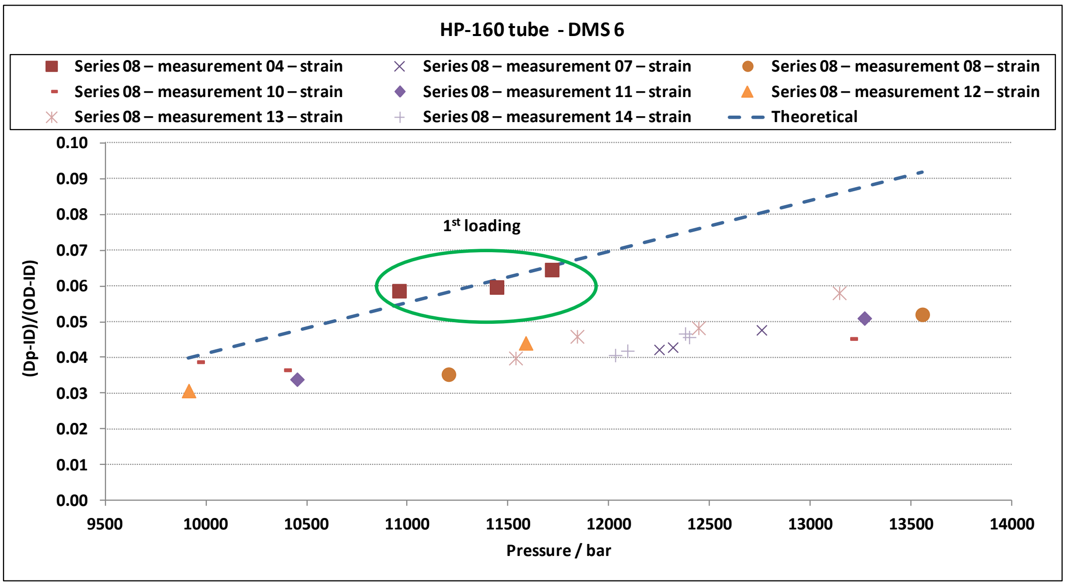


Figure 11. The relative plastic deformation to the whole diameter with pressure for HP-160 high pressure tubing

3.2.2 Elastic constants of tungsten carbides and steels measured by the resonant ultrasound spectroscopy

PTB measured the elastic constants of more than 30 tungsten carbide (WC) and steel samples used by different manufacturers of PCAs and 3 specific tungsten carbide batches used for the production of the 1.6 GPa (high pressure) and the 80 MPa (low pressure) PCAs of pressure multipliers using resonant ultrasound spectroscopy (RUS). The results showed that the density was inversely proportional to the cobalt (Co) concentration in a linear fashion, and the Young modulus (E), appeared to be directly proportional to the density. The results also demonstrated that the RUS technique is an efficient method for the accurate determination of the independent elastic stiffness constants of high hardness materials, such as WC and high pressure steel. In addition, the measurements demonstrated the suitability of RUS in determining E and Poisson coefficient (µ) with standard uncertainties as low as 0.03 % and 0.05 % for WC and 0.08 % and 0.28 % for steel, respectively. These measurements showed variations in E and µ of up to 4 % and 2 %, respectively, for different batches of nominally the same material. Even within one WC piece, material inhomogeneity was observed using RUS, with density measurements, with variations in E and µ of up to 0.8 % and 0.5 %, respectively. The measured values of E and µ using RUS contributed less than $5{\cdot}10^{-9}$ MPa^{-1} to the uncertainty of the pressure distortion coefficient, which corresponds to a relative standard uncertainty of only $8{\cdot}10^{-6}$ at pressure of 1.6 GPa, a value that meets the needs of the high pressure calibrations up to 1.6 GPa. Correlations between Co or nickel (Ni) concentration, E and density of the available WC materials at PTB were also studied. The results showed that Co or Ni concentration and E can be estimated from the material density (see Figures 12 and 13).

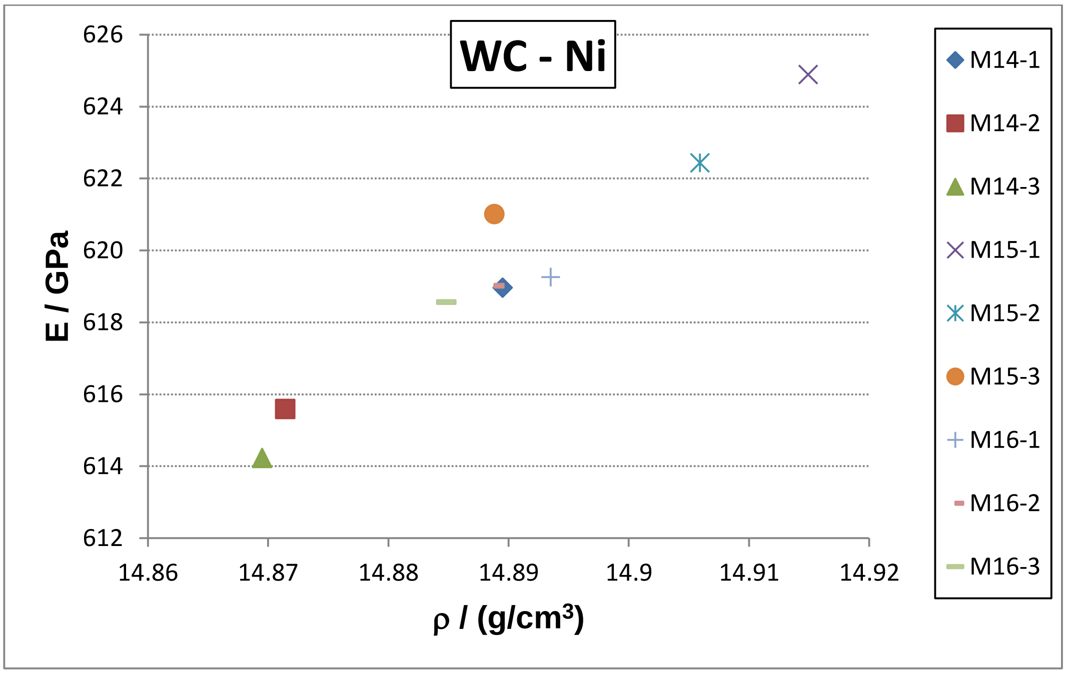


Figure 12. Correlation between the Young modulus and density of WC-Ni alloys

For the WC-Ni samples studied, a linear dependence of E on the material density was observed (see Figure 12), as well as for WC-Co alloys (see Figure 13), with the exception of sample M7-1, which was measured by two independent methods strain gauge and RUS, and showed an anomaly compared to the rest of the materials studied. This anomaly could not be explained as the material M7-1 stems from approximately 1987, and reliable information about its composition could not be obtained.

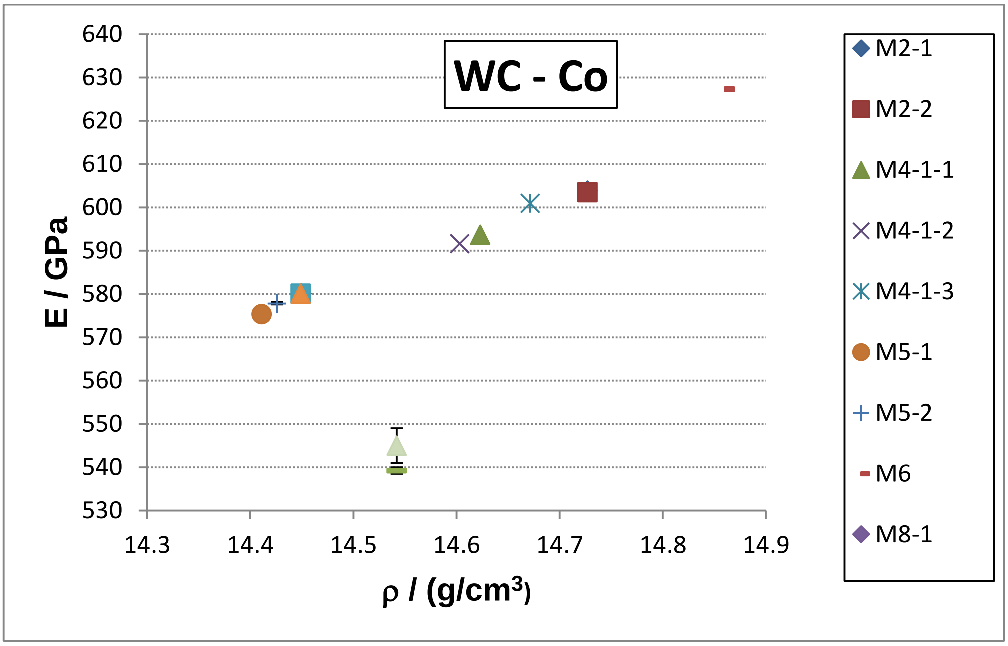


Figure 13. Correlation between the Young modulus and density of WC-Co alloys

3.2.3 Hardness measurements on high pressure components

Hardness measurements of more 15 samples from different high pressure tubing, connectors, valves and thermally treated sealing were performed at PTB (see Figure 14).

Effect of thermal treatment of the sealing lenses was investigated to determine the most appropriate processing to achieve optimal sealing performance. The results showed that, for several commercially available components the relation of their hardness is not optimal. However, optimal high pressure tight connections could be achieved with adjustment of the hardness of the sealing lenses using thermal treatment.

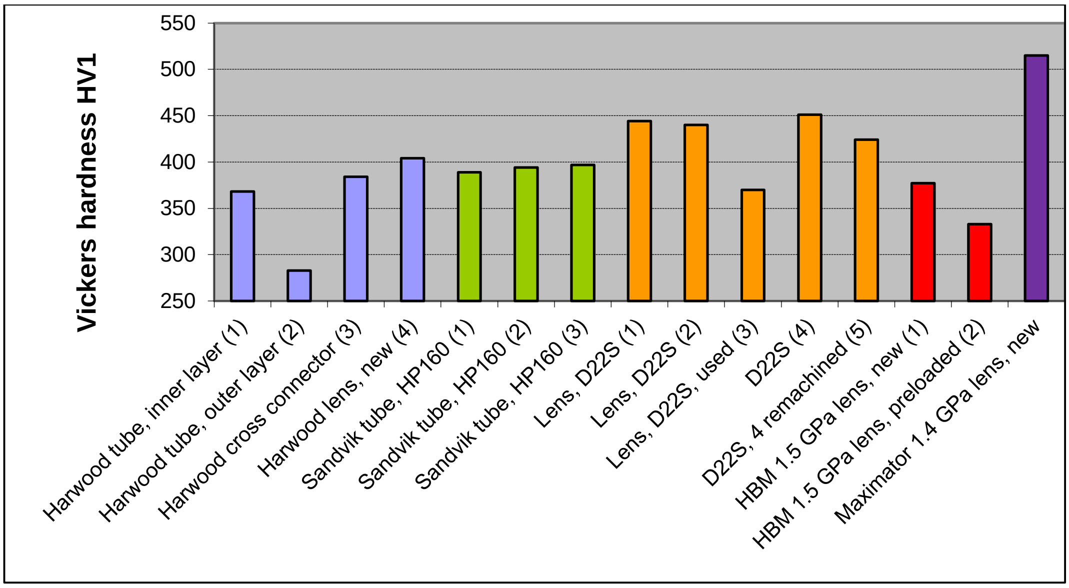


Figure 14. Vickers hardness of different high pressure components

Conclusions

The target pressure of 1.6 GPa is higher than the tensile strength of WC and most high-strength steels, the materials usually used to build pressure balances and transducers. Therefore, appropriate materials needed to 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 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 Objective 3 of the project 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.

3.3 Dimensional characterisation of piston-cylinder assemblies (PCAs)

Introduction

Dimensional properties of the cylinder bore and the piston are needed for fluid flow analysis for determining the pressure distribution in the piston-cylinder gap and, hence, the pressure distortion coefficient. Although the pressure distortion coefficient is usually associated with elastic distortions, previous studies showed that the initial shape of the gap under distortion-free conditions is important for the pressure distortion coefficient. However, measurement of the dimensional properties of high pressure PCAs is challenging because their cylinders have a thin bore and because of possible deflection of the thin piston when it is contacted by a measurement probe. In the case of low pressure piston-cylinders, when sleeves of a particular shape surround the cylinder, the outer dimensions of the cylinder and inner dimensions of the sleeve define boundary conditions on the cylinder outer generatrix surface (lateral surface) and thus have an effect on the pressure distortion coefficient of the assembly.

This work addressed 1 of the project objectives:

Objective 4. Dimensional characterisation of high-pressure piston-cylinder assemblies (PCAs).

METAS and PTB used their facilities to produce dimensional profiles of both low and high pressure PCAs. The nominal diameters of the high and low pressure pistons used were 2.5 mm and 11.3 mm, respectively and the typical piston-cylinder clearance width was between 0.2 µm and 0.6 µm, which made accurate dimensional measurements very challenging. The work was divided into two subtasks:

3.3.1. Dimensional measurements on low-pressure piston-cylinders and sleeves
3.3.2. Dimensional measurements on high-pressure pistons and cylinder bores to describe the piston-cylinder gap shape

3.3.1 Dimensional measurements on low-pressure piston-cylinders and sleeves

In a pressure-free state, there must be a thin tapered space between the outer surface of the cylinder and inner surface of the sleeve. However, when the cylinder is loaded from inside and the sleeve is subjected to the same pressure from the outside (re-entrant operation mode), the cylinder and sleeve become elastically deformed and come in contact with each other. The length of the contact line will depend on the initial geometry of the cylinder's and sleeves' matching surfaces as well as on pressure and was predicted in the project by FEM calculations in order to determine the pressure distortion coefficient of the PCA. In order to determine the initial cylinder-sleeve gap profile, METAS measured the diameters, roundness and straightness of 2 low pressure cylinders and 2 sleeves. Based on METAS's dimensional data, PTB then reconstructed the 3D cylinders and sleeves' geometry using a specially developed linking procedure.

Figure 15 shows the typical results of the dimensional reconstruction, i.e. low pressure cylinder's and sleeve's radii obtained by linking the measured straightness to the diameters.

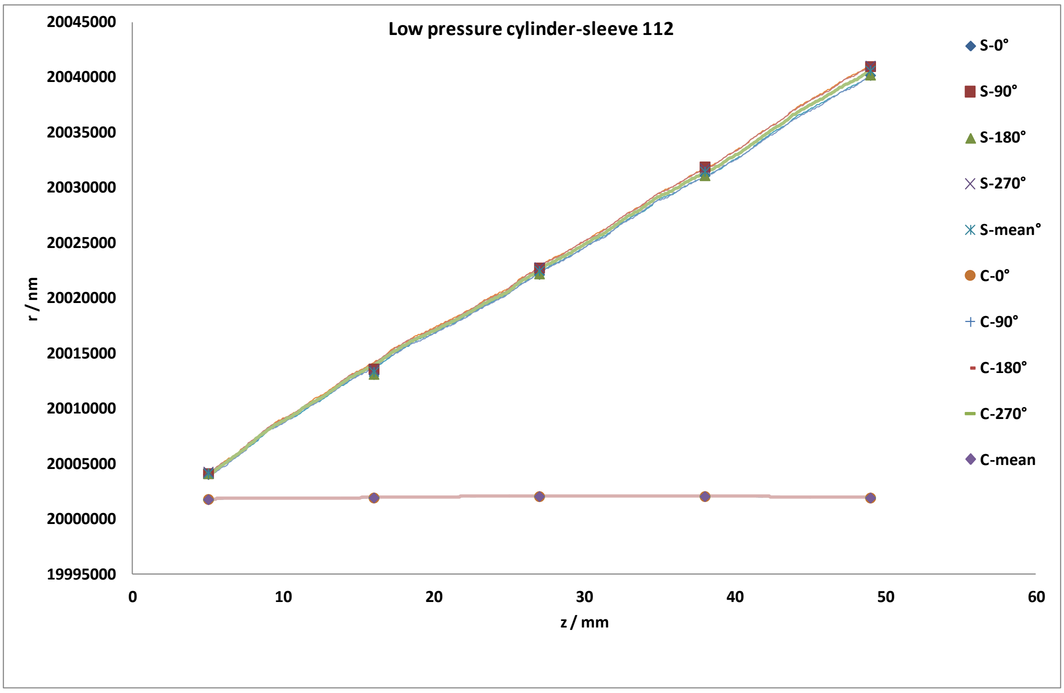


Figure 15. The outer and inner radii of low pressure cylinder and sleeve of PCA 112

3.3.2 Dimensional measurements on high-pressure pistons and cylinder bores to describe the piston-cylinder gap shape

The investigation of the small diameters of high pressure pistons and cylinders required special dimensional measurement instruments to be used. PTB determined dimensional properties of two high-pressure piston-cylinder assemblies, and numerical procedures developed at PTB were used to generate 3D data sets describing the geometry of the pistons and cylinder bores. Figure 16 shows the reconstructed gap profile of high pressure PCA 112 based on dimension measurements. Both the piston and, particularly, the cylinder bore are geometrically irregular and, the dimensional scale of the gap width, strongly deviates from a perfectly cylindrical body. The results also showed that, across the narrowest gap, the piston and cylinder almost touched each other.

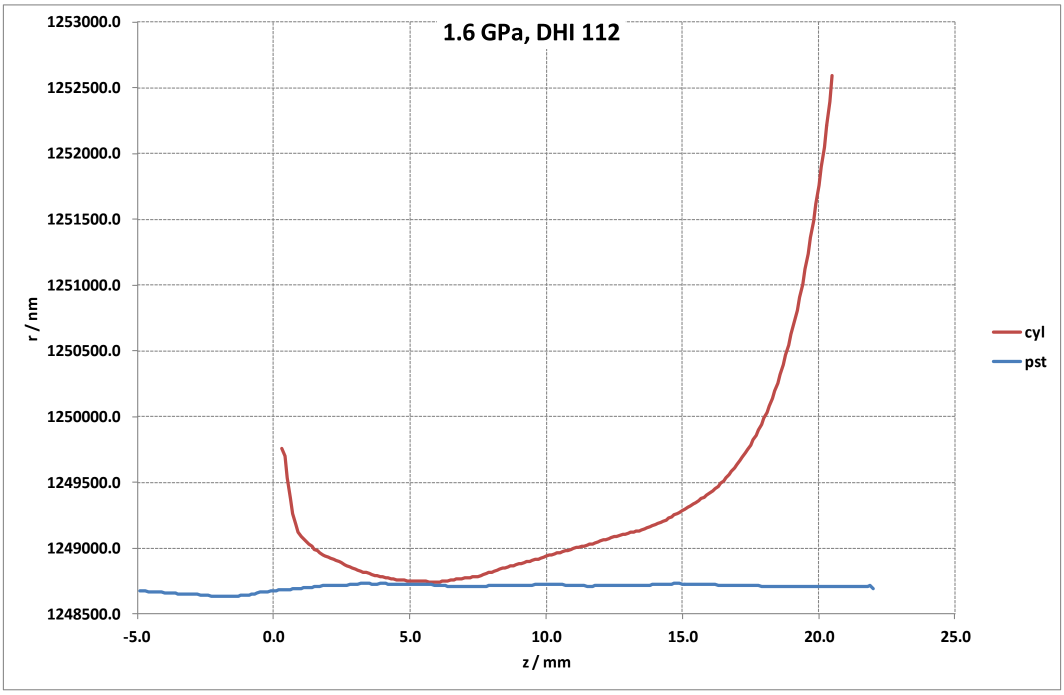


Figure 16. Radii of the piston and cylinder of high pressure PCA 112

Conclusions

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

Objective 4 of the project 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 also successfully quantified.

3.4 Investigation of potential high-pressure transmitting liquids to be used in pressure balances and industrial applications up to 1.6 GPa

Introduction

Knowledge of the pressure-dependent viscosity of a pressure-transmitting medium is needed to predict the performance of the pressure balance and to determine whether it is acceptable over the range of operation. Over the entire operating range of the pressure balance, the viscosity of the pressure-transmitting fluid should be sufficient to lubricate the piston rotating in the cylinder, but should not be too high as this can affect the sensitivity of the pressure balance. Moreover, density and viscosity vs. pressure have to be known with an uncertainty of better than 10 % in order to perform accurate fluid flow analysis for the piston-cylinder gap and to determine the pressure distortion coefficient of the pressure balance. Measurement of density and viscosity at pressures higher than 0.5 GPa is extremely challenging and prior to the start of this project no NMI worldwide could perform such measurements. However, such unique facilities were developed during the project at partner TUC.

This work addressed 1 of the project's objectives:

Objective 5. To recommend potential high-pressure transmitting liquids to be used in pressure balances and industrial applications up to 1.6 GPa

In order to achieve this, the work was divided into three subtasks:

3.4.1. Density and viscosity measurements at ambient pressure
3.4.2. Measurement of compressibility and pressure-dependent viscosity at pressures up to 1.4 GPa
3.4.3. Determination of liquids suitable for pressure balances and high-pressure industrial processes up to 1.6 GPa

3.4.1 Density and viscosity measurements at ambient pressure

Knowledge of the reference viscosity can be used to control the composition of the mixtures when using mixtures of lightly evaporating liquids as typically encountered in high pressure applications. Accurate values of density (within 0.01 %) and viscosity (within 1 %) at ambient pressure are needed to validate pressure-viscosity data measured using high-pressure viscometers. Repeated measurements before and after subjecting the liquids to a pressure of 1.6 GPa also allow assessment of the liquid's stability. Therefore, the density and viscosity of the samples of i) poly-ethyl-siloxane (PES-1), ii) di(2)-ethyl-hexyl-sebacate (DHS), Petroleum, and iii) 50 vol.% DHS + 50 vol.% Petroleum were measured by PTB at atmospheric pressure and at three temperatures 18, 20 and 22 °C. The expanded uncertainties (k = 2) of the measurements were 0.00002 g/cm^3 for density and 1 % for viscosity. For PES-1, the measurements were performed in its original state and with pressures up to 1.6 GPa. From the results, the effect of pressurisation up to 1.6 GPa on the oil viscosity and density could be quantified compared to atmospheric pressure and at temperatures of 18, 20 and 22 °C

3.4.2 Measurement of compressibility and pressure-dependent viscosity at pressures up to 1.4 GPa

Analysis of pressure-viscosity dependences should allow prediction of the liquids' solidification point and their viscosity up to 1.6 GPa. Project partner TUC developed a new and unique high-pressure viscometer and evaluated it as part of the project. Technical modifications were undertaken to solve the issues associated with tight sealing at high pressure. Measurements of viscosity and compressibility of DHS were performed by TUC at 20 °C in the range up to 1 GPa and at the temperatures 40, 70 and 120 °C up to 1.25 GPa. The viscosity and density of PES-1 and a mixture of 50 vol.% DHS + 50 vol.% Petroleum were measured at 20 °C up to 1.3 GPa and 1.35 GPa, respectively, and the results are shown in Figure 17.

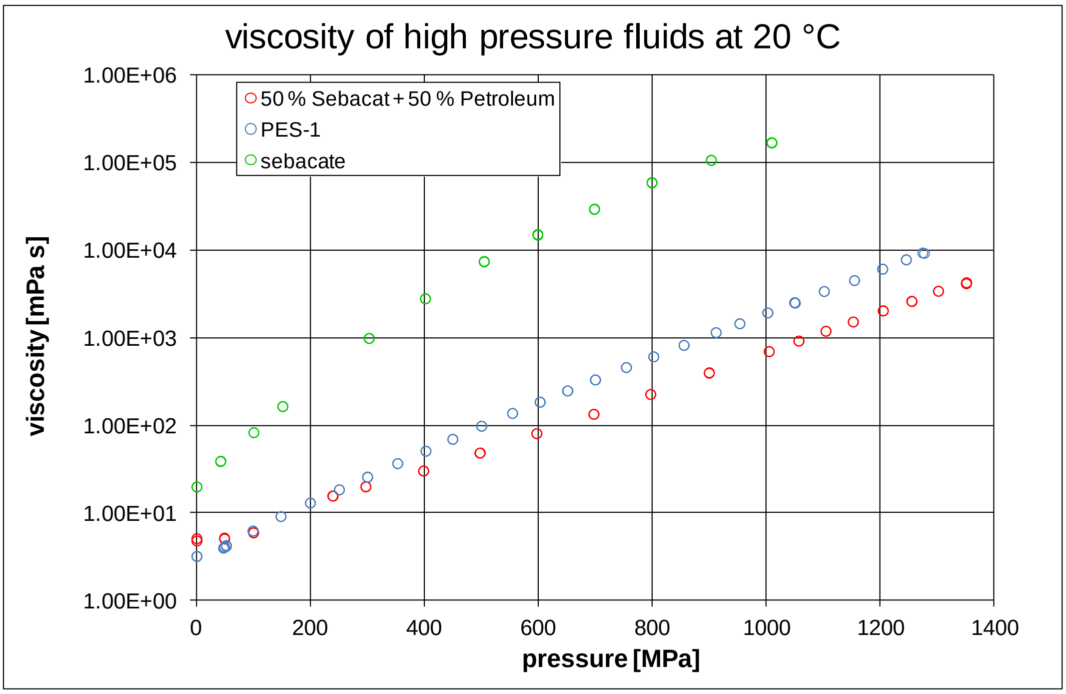


Figure 17. Viscosity of three high-pressure transmitting fluids vs. pressure at 20 ºC

3.4.3 Determination of liquids suitable for pressure balances and high-pressure industrial processes up to 1.6 GPa

Viscosity and density data and their dependence on pressure are required for the calculation of the pressure-distribution in the piston-cylinder gap and, hence, the pressure distortion coefficient of the PCAs. Potential liquids were selected whose viscosities were acceptable, i.e. lie between 0.01 Pa s and 400 Pa s in the pressure range 0 GPa – 1.6 GPa, and which are non-toxic, chemically unambiguous and stable. PTB analysed the results of the compressibility and viscosity measurements from section 3.4.2. The stability of the liquids after pressurising them up to 1.6 GPa and repeating density and viscosity measurements at atmospheric pressure was investigated by PTB. In particular, the mixture of DHS with petroleum (50 vol.% DHS + 50 vol.% Petroleum) showed a relatively low dependence of the viscosity on pressure with good lubricating properties at atmospheric pressure and, thus, was recommended as a pressure-transmitting medium in high pressure measurements and high-pressure industrial applications up to 1.6 GP.

Conclusions

The density and viscosity of potential liquids needed to be measured to determine their suitability for use in a 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 project's objective 5, a new high-pressure viscometer was developed to determine the properties of the potential pressure-transmitting liquids. The 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.

3.5 Development of a primary pressure standard for pressures up to 1.6 GPa, with a relative expanded uncertainty as low as $5x10^{-4}$ (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

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

Introduction

Providing industry with metrological service for the range of pressures up to 1.5 GPa requires appropriate primary and transfer pressure standards. The upper range for industrial applications and transfer standards was chosen by the project as 1.5 GPa due to the fact that it is the upper pressure limit of modern commercially available pressure transducers. The choice of 1.6 GPa as the highest pressure of the primary standard was dictated by the necessity to have an extended measurement capacity compared with the range of routine calibrations and by the need to preload 1.5 GPa pressure transducers with pressures higher than 1.5 GPa prior to calibration in order to achieve a better stability in the 1.5 GPa range.

The important building block of the project's high-pressure primary standard was a pressure balance. Pressure balances are known as the most accurate high pressure standards with the smallest possible uncertainties of few ppm. Worldwide, pressure balances that can measure pressures above 1 GPa exist in China, India and Russia. However, in Europe, only pressure balances that were operable up to 1 GPa were available prior to the start of this project. In terms of accurate high pressure transfer standards, commercial 500 MPa pressure balances and, above 500 MPa, 1 GPa pressure multipliers can be used. However, the latter are very expensive, difficult to operate and maintain and due to this were rarely used in industry.

This work addressed 2 of the project objectives:

Objective 6. To create a primary pressure standard for pressures up to 1.6 GPa, with a relative expanded uncertainty as low as $5x10^{-4}$ (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.

Objective 7. To 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.

In order to achieve this the work was divided into five subtasks:

3.5.1. Development of 1.6 GPa pressure multipliers as primary pressure standards, including design, technical drawings, manufacture of parts and assembly of the multipliers

3.5.2. Development, design, manufacture and testing of high pressure generation and control systems and their components (intensifiers, tubing, connections, valves, sealing elements) for pressures up to 1.6 GPa

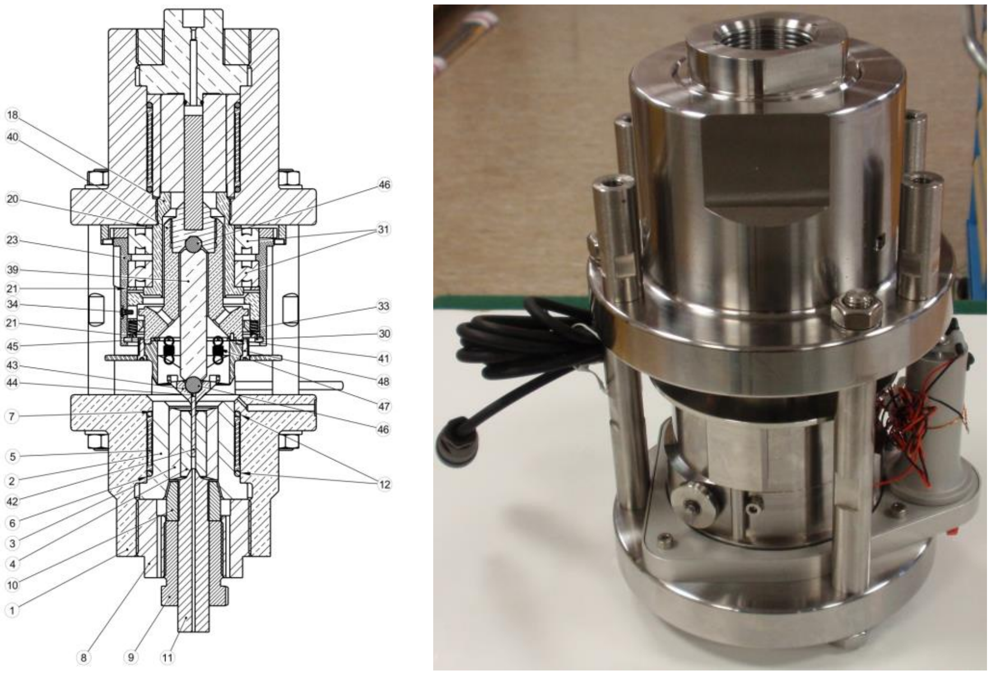
3.5.3. Tests and metrological characterisation of the multipliers. Experimental determination of zero pressure effective areas, multiplying ratios and pressure distortion coefficients against 100 MPa and 1 GPa primary pressure standards. Verification of the theoretical pressure distortion coefficients by experiments with variable jacket pressures

3.5.4. Characterisation and optimisation of modern 1.5 GPa pressure transducers. Investigation of their drift, hysteresis, sensitivity, repeatability, long-term stability and load cycling effects. Specification of calibration methods

3.5.5. Development and testing of 1.5 GPa transfer standards realised on the basis of high-precision pressure transducers. Verification of the transfer standards, application in comparison measurements in the range from 0.1 to 1.5 GPa

3.5.1 Development of 1.6 GPa pressure multipliers as primary pressure standards, including design, technical drawings, manufacture of parts and assembly of the multipliers

PTB created technical drawings of two pressure multipliers with an operational range of 1.6 GPa. Each multiplier included a low pressure and a high pressure PCA with a nominal ratio of their effective areas of 20:1. In the multiplier, the PCAs were axially aligned and their pistons were mechanically coupled to transmit the force from the low pressure to the high pressure piston. With a pressure of 80 MPa applied to the low pressure PCA, a pressure of 1.6 GPa was able to be measured by the high pressure PCA. Based on the FEM analysis results obtained in section 3.1.4, the high pressure PCA design with the best predicted properties was chosen. The high pressure cylinder consisted of a WC core with two steel sleeves shrunk on it. Additionally, a jacket pressure was applied to the outer surface of the high pressure cylinder to support it. The design of 1.6 GPa pressure multipliers to be used as primary pressure standards was completed and PTB produced parts and ordered low and high pressure PCAs for the two multipliers. Two 1.6 GPa multipliers were assembled at PTB as shown in Figure 18.

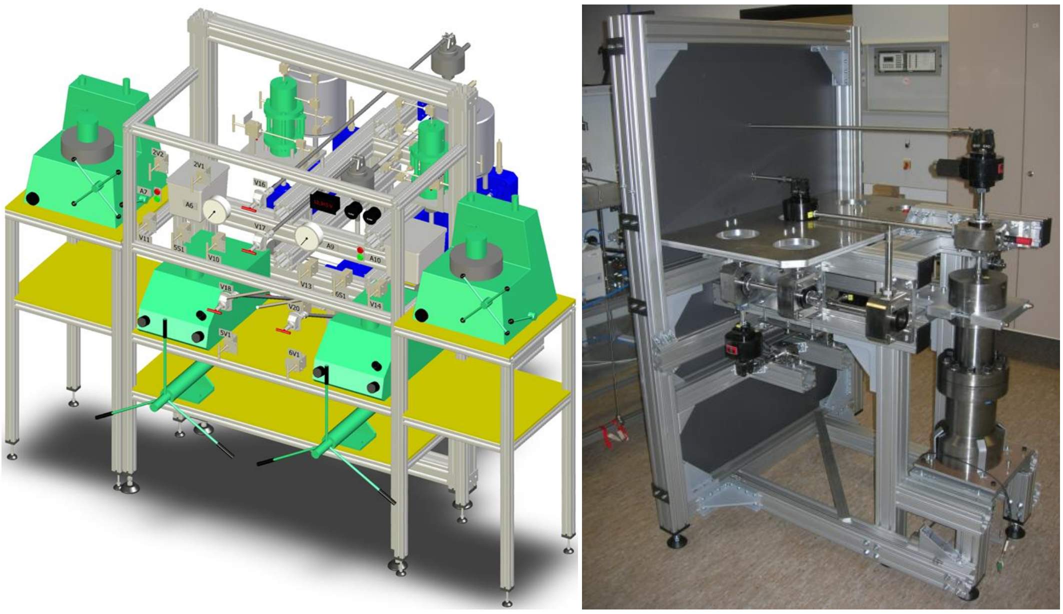


a **b**

Figure 18. Design of the 1.6 GPa pressure-measuring multiplier (a) and its appearance (b)

<u>3.5.2 Development, design, manufacture and testing of high pressure generation and control systems and their components (intensifiers, tubing, connections, valves, sealing elements) for pressures up to 1.6 GPa</u>

PTB designed and built a 1.6 GPa pressure generation and control system by PTB, which included a low pressure generation and regulation system for 100 MPa, built using commercial components including a hydraulic pump, system of valves, tubing and manometers (Low pressure supply). The low pressure generated by this system was supplied to the low pressure side of a 2 GPa 1:35 pressure intensifier which was available at PTB. In terms of high pressure, a pressure of 1.6 GPa or more was generated by the intensifier and transmitted through a system of tubing, connectors and valves to the high pressure cylinders of the pressure measuring multipliers and/or pressure transducers. In addition, the pressure generation system included a500 MPa pump to pre-pressurise liquid under high pressure and a 100 MPa pump to supply pressure to the low pressure PCAs of the pressure multipliers. The development, design, manufacture and testing of high pressure generation, the control systems and their components for pressures up to 1.6 GPa was successfully carried out at PTB. A remote observation system was installed for the identification of untight connections and PTB designed special connectors and tested the performance of commercial high pressure components at pressures up to 1.6 GPa. The tests showed that these components could withstand pressure of 1.6 GPa. The pressure generation system and its assembly process are shown in Figure 19. The system was tested and demonstrated its capability to generate pressures up to 1.6 GPa and to keep them nearly constant over many hours.



a **b**

Figure 19. Design of the 1.6 GPa pressure generation system (a, front view) and its production (b, back view)

3.5.3 Tests and metrological characterisation of the multipliers. Experimental determination of zero pressure effective areas, multiplying ratios and pressure distortion coefficients against 100 MPa and 1 GPa primary pressure standards. Verification of the theoretical pressure distortion coefficients by experiments with variable jacket pressures

Metrological characteristics of the 1.6 GPa pressure multipliers was carried out by PTB. The multiplying ratio of each multiplier was determined against existing PTB primary 100 MPa and 1 GPa pressure balances and measurement of the jacket pressure distortion coefficients of the high pressure PCAs of the multipliers were determined at variable jacket pressures up to 1 GPa. The piston fall rate was also found to be dependent upon pressure. Theoretical data obtained from the FEM calculations in section 3.1.4 was compared with experimental results obtained up to 1 GPa and form this the optimal pressure distortion coefficients were derived . The uncertainties of the two 1.6 GPa multipliers were estimated up to 1.6 GPa and Figure 20 shows the relative differences between the reference pressures of the PTB primary 1 GPa pressure balance and of the 1.6 GPa multiplier. The latter was based on three models in which the tare pressure (P_0) and the pressure distortion coefficients of first (L_1) and second (L_2) order were determined via different methods. The best agreement with the reference pressure was achieved when L_1 was determined experimentally and L_2 was calculated using FEM. The dashed lines in Figure 20 show the relative uncertainties of the multiplier pressure corresponding to the three models over the full operation range of 1.6 GPa. These uncertainties are at least 6 times lower than the project's target uncertainty of $5 \cdot 10^{-4}$.

The operation and performance of the 1.6 GPa pressure multipliers were tested against high-pressure transducers in the range up to 1.3 GPa. Final experimental verification of the parameters and performance of the two pressure multipliers over their full operation range of 1.6 GPa was performed by a direct comparison.

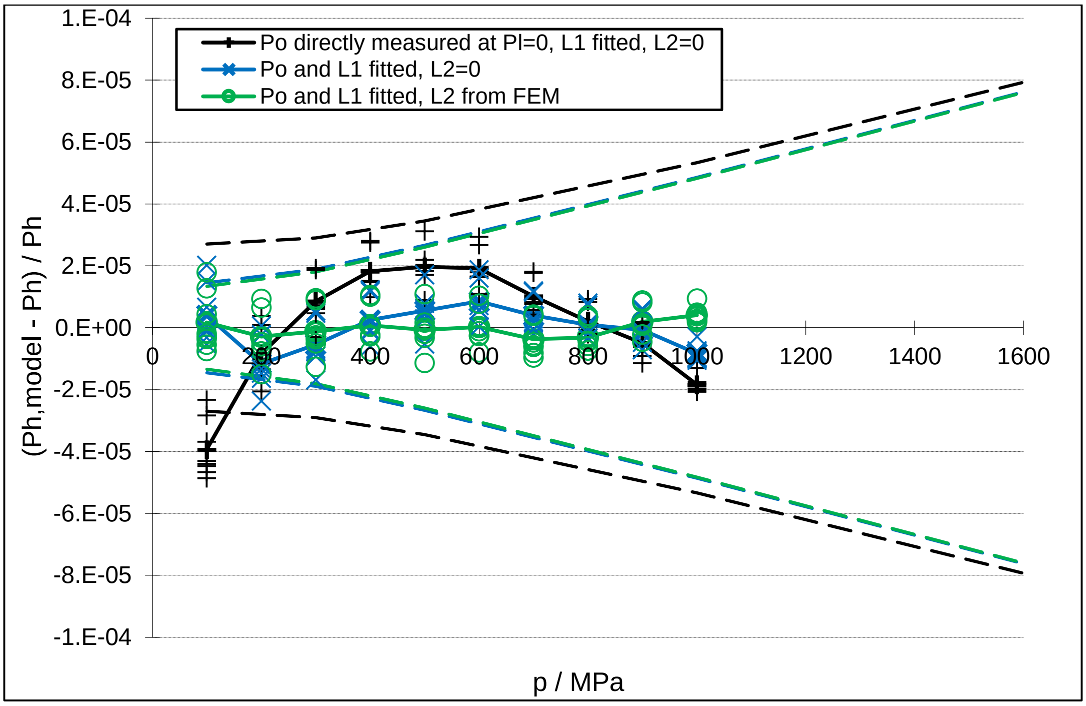


Figure 20. Relative deviations of multiplier's pressures ($P_{h,model}$) from the reference ones and uncertainties of $P_{h,model}$

3.5.4 Characterisation and optimisation of modern 1.5 GPa pressure transducers. Investigation of their drift, hysteresis, sensitivity, repeatability, long-term stability and load cycling effects. Specification of calibration methods

Foil strain gauge and silicon technology-based piezo-resistive pressure transducers, two for 0.5 GPa, two for 1 GPa and four for 1.5 GPa range, were obtained from two of the project's collaborators and the calibration curve, hysteresis sensitivity and repeatability were determined for each of the pressure transducers.

Mounting effects, their short and long-term stability and the effect of load cycling were also studied for the pressure transducers and for each type of pressure transducers the preload procedures included overload by 5 % of the maximum pressure. Measurements of high pressure transducers were performed against the existing national pressure standards of project partners PTB, SMU, CMI, LNE and METAS, with SMU specifically analysing high pressure transducers. PTB preloaded the transducers to maximum excessive pressures in order to achieve optimal stability and SMU and CMI characterised two 0.5 GPa, two 1 GPa and four 1.5 GPa pressure transducers over the 500 MPa pressure range. LNE, METAS and PTB characterised the same pressure transducers in the pressure ranges up to 1 GPa. Finally, stability test measurements were done at PTB and evaluation of all measurement results and subsequent optimisation of calibration procedures was performed by PTB.

3.5.5 Development and testing of 1.5 GPa transfer standards realised on the basis of high-precision pressure transducers. Verification of the transfer standards, application in comparison measurements in the range from 0.1 to 1.5 GPa

PTB drafted a technical protocol for comparison measurements for testing the high pressure transfer standards (based on pressure transducers). SMU then finalised the technical protocol for the comparison in accordance with the requirements of existing national pressure standards and PTB assembled and tested its transfer standard (see Figure 21), and created software for its operation and data acquisition.

A high pressure transfer standard based on pressure transducers was then created and characterised up to 1 GPa and a procedure for the calibration of 0.5, 1 and 1.5 GPa pressure transducers was also developed.

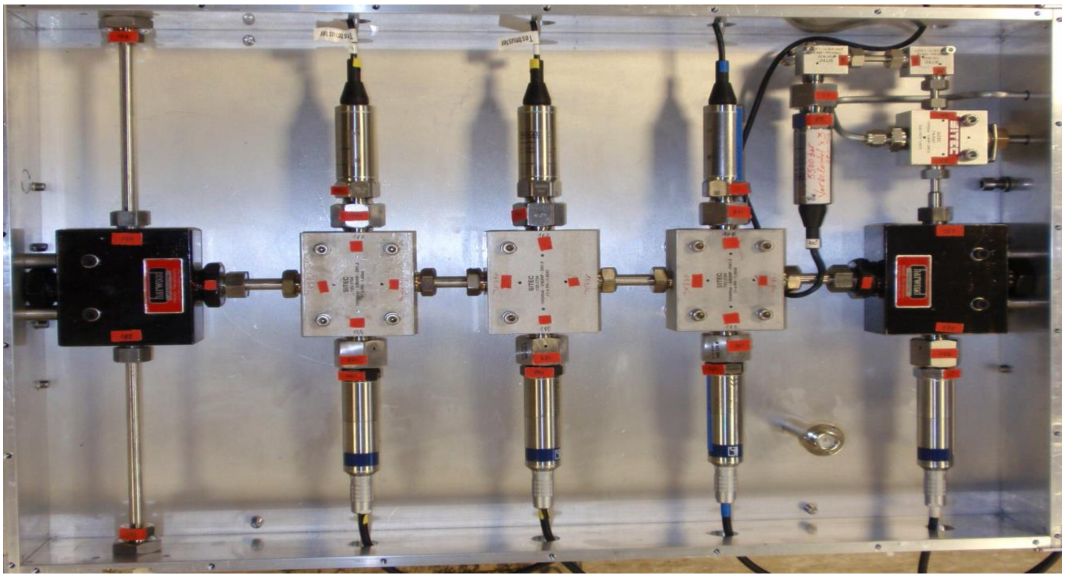


Figure 21. High pressure transfer standard based on 0.5, 1, and 1.5 GPa pressure transducers

The results of the comparison of the high pressure transfer standard were analysed and demonstrated, that state-of-the-art 0.5, 1 and 1.5 GPa pressure transducers can be successfully used as transfer pressure standards. Nevertheless it was also found that the pressure transducers behave as individuals, even those produced by the same technology and having the same pressure range. Therefore, pressure transducers of two types were monitored over 1.5 years and each was measured in more than 150 measurement cycles, from this the best pressure transducer type was identified in terms of the most stable and suitable as a high pressure transfer standard.

Conclusions

The primary pressure standard is the core measurement reference upon which the calibration service is founded. A 1.6 GPa pressure generation and control system was designed, manufactured and tested, based on the knowledge gained from the project's 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 project's objective 6 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 at PTB, for high-pressure measurement instruments, including 1.5 GPa pressure transducers accurate to 0.0005 GPa.

Pressure transducers are sufficiently accurate and could 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 then designed, assembled and studied. The project's objective 7 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.

4 Actual and potential impact

4.1 Dissemination activities

4.2.1 Publications and Conferences

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 section 6), and presentations at international conferences and committees. This included 22 oral and 2 poster presentations, given at such conferences as the XX IMEKO World Congress (Busan, Korea, 9-14 Sep 2012), the 16th International Congress of Metrology (Paris, France, 7-10 Oct 2013) the 50th European High Pressure Research Group Meeting (EHPRG, Thessaloniki, Greece, 16-21 Sep 2012) and the XIII European Vacuum Conference (Aveiro, Portugal, 8-12 Sep 2014).

4.2.2 Workshops & Training Courses

During the project, two workshops were held on high-pressure metrology for industrial applications, and a training course on high-pressure measurement and calibration. The first workshop was entitled "High Pressure Metrology for Industry", and was held in Brno, Czech Republic in June 2012 http://emrp-highpres.cmi.cz/Download/en/WS-Brno-flyer_120229.pdf. The workshop was attended by approximately 30 participants, including representatives of high pressure metrology and instrument manufacturers.

The second workshop, entitled “High-Pressure Metrology for Industrial Applications” took place in Braunschweig, Germany in September 2014 http://www.highpres-workshop.ptb.de. Within the scope of this workshop, high pressure measurements and the calibration of high pressure transducers were presented. Moreover, advanced topics on high pressure technologies were addressed such as; primary and secondary high-pressure standards; the traceability of high-pressure measurements; high-pressure measurement methods and instrumentation; the properties of high-pressure fluids and materials; high-pressure generation and testing equipment; innovative industrial high-pressure technologies; and the challenges and demands in industrial high-pressure applications. Approximately 40 representatives of industry, including both manufacturers and users of high-pressure transducers, academic and national metrology institutes from seven European countries and participated in the workshop. In both workshops, the delegates were highly interested in the project’s outputs, especially, the role of the NMI partners in the development of a new primary standard up to 1.6 GPa and the associated transfer standards.

A one-day training course on ‘High-Pressure Measurement and Calibration’ was also held by the project, in Braunschweig, Germany, on 18 September 2014 http://www.highpres-training.ptb.de. The objective of the training course was to promote the application of high-pressure technologies across Europe by providing theoretical and practical training on the calibration and application of high-pressure measuring instruments. The training course was aimed at professionals specialised in pressure calibrations and measurements in the range from 100 MPa to 1 GPa and above, and approximately 30 such specialists participated in the training course.

4.2.2 Guidelines and Standards

One of the European standards relevant for high-pressure calibrations is EURAMET calibration guide cg-17 "Guidelines on the Calibration of Electromechanical Manometers". The project was presented to the EURAMET TC-M meetings in February 2012, April 2013, April 2014 and April 2015. Each time to an audience of 35-50 experts, who welcomed the results of the project and as a consequence a revision of Calibration guide cg-17 "Guidelines on the Calibration of Electromechanical Manometers" was agreed.

The revised EURAMET guide cg-17 ‘*Guidelines on the Calibration of Electromechanical Manometers*’, now includes the project’s 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 also 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.

Further to this, the project had an impact on the activities of the DIN German Institute for Standardisation, NA 152 - Standards Committee Technical Fundamentals (NATG), Division 4 - pressure, flow, temperature, in A suggestion was submitted to the DIN NATG to include an additional section into the standard series EN 837, so that it takes into account special process connections, bursting collaterals and safety implementations for high pressures above 160 MPa (the respective draft, prEN 837-4:2014 entitled "Pressure gauges for highest pressure", is still in progress).

4.2 Effective cooperation between project partners

This project was a good example of the implementation of the EMRP program and effective cooperation between project partners. The project included six NMIs and one academic partner from six European countries and was one of the first collaborative projects in which partners were involved who have a wide range of expertise in different areas of high pressure physics and metrology, e.g. high pressure and dimensional metrology, material test capabilities and high pressure rheology. The project's objectives could not have been achieved without effective cooperation and examples of this are elastic constants measured by the strain gauge method at LNE, the dimensional measurements on low pressure cylinders and sleeves carried out by METAS, joint FEM analyses at PTB and LNE, and the joint publications and presentations produced by the partners.

The cooperation was also beneficial for the project partners in terms of knowledge transfer and the enhancement of their capabilities. This was particularly important for those less experienced NMI partners and produced important advancements and calibration services for European high-pressure metrology.

4.3 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.

4.4 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.

5 Website address and contact details

Public website http://emrp-highpres.cmi.cz

Contact person Dr Wladimir Sabuga, PTB, email: wladimir.sabuga@ptb.de

6 List of publications

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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)