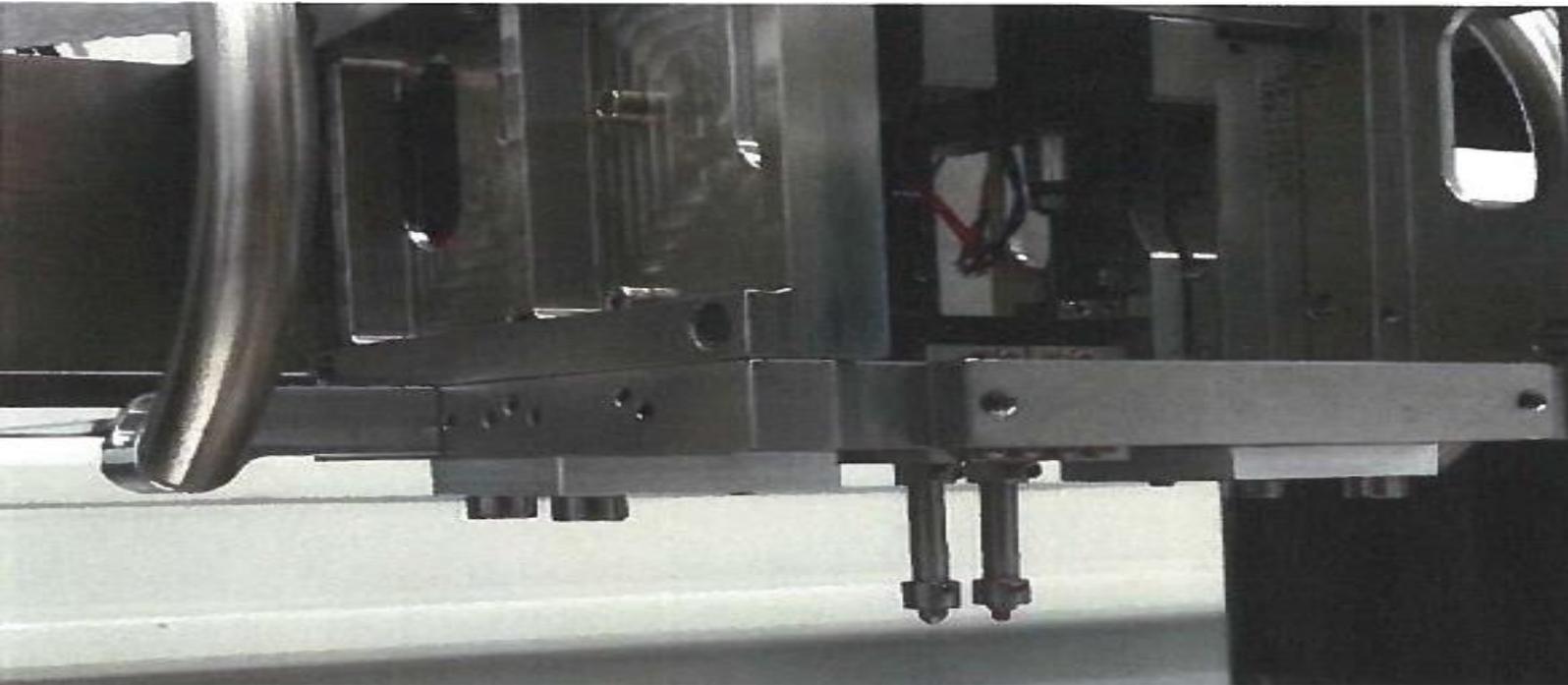


## JRC Technical Systems



# Installation, Commissioning and Acceptance Testing of a High Temperature Ultra Nanoindentation Tester

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2015

Report EUR 26988 EN

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**Abstract**

The Institute for Energy and Transport (IET) of the Joint Research Centre (JRC) has purchased a modular Nanoindentation Test System for testing the physical properties of metallic, ceramic and composite materials at small length scales. The instrument allows the measurements of nanoindentation hardness and Young's modulus, creep, fracture toughness and ductile-to-brittle transition temperature at nano length scales from cryogenic to high temperatures (-150 °C ~ 700 °C). Measurements can be applied to characterize the properties of hard coatings, single or multi-layer films, bulk soft materials, polymer films and multi-phase alloys.

# 1. Introduction

## 1.1. Background

The Institute for Energy and Transport (IET) decided to purchase a modular Nano-Indentation Test System to support its activities related to the testing of the physical and mechanical properties of metals, ceramics and composite materials. The equipment had to be capable of performing the classical nano-indentation experiments based on instrumented loading and unloading of the indenter tip onto a specimen in order to provide indentation hardness and indentation modulus versus depth at precisely predefined positions on the specimen surface. The system had to allow measuring the indentation hardness, elastic modulus, creep, fracture toughness and Ductile-to Brittle Transition Temperature at temperatures ranging from, at least, - 20°C up to 600°C. The equipment and measurements performed have to comply with ISO 14577 and ASTM E2546.

In this scope, the IET launched a call for tenders requesting the following mandatory technical requirements:

- The equipment shall be modular constructed, capable to contain at least 3 different instruments attached to the frame. The load frame stiffness of the equipment shall be  $\geq 1 \times 10^7$  N/m. An anti-vibration table, air table or equivalent shall be used to minimize disturbances from the laboratory surrounding.
- The equipment shall be software controlled for test execution, data acquisition and data analysis and include the following loading techniques: force control, depth control, constant strain rate, step loading and depth profiling
- The equipment shall allow opting for a fully automated measurements program and testing parameters selectable by user, including at least: Initial contact load, maximum load, number of load and unload increments, holding time period, final unload, delayed starting of a test (up to 24h), time period to wait after each indentation, number of indentations and spacing, user file containing custom test schedule, user defined loading history including cyclic loading, matrix measurements by defining x, y coordinates or free settings via microscope.
- The equipment shall allow mounting and testing of multiple specimens with different heights on the sample holder. Maximum specimen dimensions (LxBxH): >50mm x 50mm x 50mm. Usable inspection area (x, y):  $\geq 50$ mm x 50mm
- The following standard indenter types shall be mountable on the equipment and selectable in the software: Sphero-conical indenter, Berkovich three-sided pyramid indenter, Vickers four-sided pyramid indenter, Knoop four-sided pyramid indenter, Cube-corner three-sided pyramid indenter.
- The equipment shall consist of either a vacuum chamber or a purge chamber to perform Nano-Indentation testing in oxygen reduced atmosphere.
- The equipment must allow the execution of tests at temperatures from - 20° up to 600°C in oxygen reduced atmosphere. An indenter-tip heating shall be provided for elevated temperature. Sample and indenter tip temperature within the temperature range (room temperature – maximum temperature) shall not differ by more than 3°C. Thermocouples or equivalent shall be installed to control and monitor temperatures of the sample, sample stage, indenter and other temperature sensitive parts. Additional merit points were awarded if the temperature for testing could reach the range -150 °C ~ 800 °C.
- The equipment shall provide thermal stability under normal laboratory conditions: thermal drift  $\leq 0.3$ nm/min at room temperature and  $\leq 20$  nm/min at 600°C.

- The maximum usable load shall not be lower than 50mN and shall not exceed 500mN. The continuously adjustable loading rate shall be at least 0 - 1000 mN/min. The load noise floor, under normal laboratory conditions, shall not exceed 0.5  $\mu$ N in the whole range of test temperatures. The load resolution shall be  $\leq$  0.01  $\mu$ N. The equipment shall offer a minimum contact load  $\leq$  1  $\mu$ N in the whole range of test temperatures. The minimum usable load shall not exceed 100  $\mu$ N in the whole range of test temperatures.
- The equipment shall provide a minimum usable displacement of 100  $\mu$ m to perform Nano-Indentation testing. The displacement noise floor, under normal laboratory conditions, shall be  $\leq$  0.2 nm. The displacement resolution shall be  $\leq$  0.01 nm.
- The equipment shall provide an X-Y positioning resolution of the specimen stage  $\leq$  0.5 $\mu$ m. The specimen stage shall allow accommodating more than one specimen at a time for room temperature testing. The load frame shall be fitted with a z-axis drive to allow for positioning of samples with different thicknesses or sample heights. The stage shall allow computer controlled movement to and from the optical microscope or AFM. The re-position accuracy between specimen stage and optical microscope/AFM shall be  $\leq$  1  $\mu$ m.
- The optical microscope shall provide at least 4 turret-mounted lenses, e.g. 5x, 10x, 20x and 40x objectives that can be positioned without the need for realignment of the specimen or the microscope. High temperature filters for viewing samples at elevated temperatures shall be included. A colour CCD camera shall be attached on the optical microscope allowing live imaging and image capture.
- The software shall allow the free control of indentation parameters, the use of automated measurement sequences, the automatic capture of pictures of each single indentation, the measurement of crack length after indentation, the real time monitoring of indentation curves, the recall of previous test cycles, and the means to generate a measurement report consisting of at least measurements data, indentation curves, pictures and comments. Positioning of indentations shall be done either by setting the coordinates or by using the optical microscope. Automated calibration for measuring physical distances on optical microscope (and optional AFM) as well as indenter calibration shall be provided. A correction function for initial penetration, thermal drift, instrument compliance and indenter area function shall be available. Real time display of indenter coordination position, load, displacement, temperature of sample and/or indenter, parameter of environmental chamber shall be selectable by user. Automated analysis routines shall be included to calculate indentation data for: Vickers Hardness, Knoop Hardness, Rockwell Hardness, Brinell Hardness, Stress/strain data, Creep, Relaxation, Plastic and elastic work, Fracture toughness, 3D mapping and profiling.
- Two pieces of each of the following standard indenters, together with a calibrated area function, shall be supplied: Berkovich and sphero-conical indenters, in diamond and in a material for high temperature tests compatible with carbon steel.
- The equipment shall be equipped with at least 2 different pieces standard reference blocks according to ISO 14577 and ASTM E2546. Each reference block shall be provided with certified values of dynamic Young's modulus and Poisson's ratio.

As options, tenderers were invited to provide quotations for a Scratch Tester and an Atomic Force Microscope (AFM).

## **1.2. Call for tender**

The contract notice of the open call for tenders was published in the O.J. 2013/S 150-260077 on 03/08/2013.

Four tenders from four companies were received and evaluated with respect to their compliance with the technical requirements. The evaluation committee decided to award the contract to ST Instruments offering equipment from CSM Instruments, as the company offered the most competitive quotation while it provided solid documented evidence of their technical capabilities to meet the specifications beyond the mandatory technical requirements..

## **1.3. Features of the instrument selected.**

The company awarded with the supply contract, ST Instruments, offered the delivery of a high temperature nanoindentation instrument from CSM Instruments, a company recently acquired by Anton Paar, GmbH. The CSM High Temperature Ultra Nanoindentation Tester allows the acquisition of accurate load-displacement data up to temperatures of  $\geq 700$  °C thanks to a transducer system that compensates thermal drift keeping it below 10 nm/min. The technology enables the measurement of hardness, Young's modulus, fatigue and creep data of materials at high temperatures at the nano scale.

At small length scales, thermal stability becomes the more important problem to extract accurate quantitative mechanical properties from load-displacement data. The CSM nanoindentation instrument solves the issue by using a unique referencing design that allows for continuous differential depth measurements, thus ensuring a very good thermal stability and a fast indentation cycle. The referencing system is based on the principle of using an axis of measurement and an axis of reference, each one having its own actuator and depth and force sensors, to carry out active referencing of the surface of the sample. The Ultra Nanoindentation head is made of Zerodur, a ceramic glass with an extremely low thermal expansion coefficient, and the thermal drift is minimized by preheating both the nanoindentation tip and the sample.

The instrument is also equipped with a software package that allows performing tests in a wide variety of testing modes, i.e. simple loading, multi-cycle loading, dynamic mechanical analysis, automated matrixes or pinpointed location testing.

The key features of the systems are the following:

- Active top referencing (Patented Design US 7,685,868,B2).
- Low thermal drift: the head is made of Zerodur glass and the electronic system has a drift rate of  $\approx 1$  ppm/°C.
- Load frame stiffness  $\gg 10^8$  N/m
- Two independent depth and load sensors
- High resolution and very low noise:
  - o Depth resolution: 0.0003 nm, noise floor: 0.03 nm
  - o Load resolution: 0.001  $\mu$ N, noise floor: 0.13  $\mu$ N
- Positional synchronization between the indentation head, the optical microscope head and the atomic force microscope head.
- Compliant to ISO 14577 and ASTM E2546.

## 2. Commissioning

### 2.1. Technical specifications

ST Instruments has provided the Ultra Nanoindentation Tester on a modular high vacuum platform including heating ( $\geq 700\text{ °C}$ ) and cooling ( $\leq -150\text{ °C}$ ) consisting of the following parts:

- Automated platform for secondary vacuum with secondary vacuum chamber, including adapted automated platform and frame (X, Y, Z), secondary vacuum chamber and control, and optical observation.
- Ultra Nanoindentation Tester Module for secondary vacuum and compatible with measurements at elevated temperatures ( $\geq 700\text{ °C}$ ), including a Ultra Nano Hardness head with laser tip heating.
- High temperature stage ( $\geq 700\text{ °C}$ ) for secondary vacuum, including heating, cooling and temperature controllers.
- Cryogenic sample stage for secondary vacuum, including liquid nitrogen pump, connectors and tubing, allowing cooling the sample down to  $-150\text{ °C}$ .
- Antivibration table for vacuum chamber.
- High load range (maximum load of 100mN) and high depth range (maximum penetration depth of 100  $\mu\text{m}$ ) sensors and actuators for Ultra Nanoindentation.
- Berkovich indenters of diamond and tungsten carbide.
- Spherical indenters of diamond and tungsten carbide.

A dedicated AFM (NaniteAFM, from Nanosurf) for the direct characterization of the nanoindentations is integrated to the Ultra Nanoindentation Tester. The embedded AFM has the following features:

- Synchronised positioning of AFM - optical microscope – nanoindentation head
- Optical top and side view of cantilever and sample.
- Large scan area up to 110 x 110 mm, with 22 mm in Z range.
- Contact modes of contact force, height and force modulation.
- Dynamic modes of non-contact force, phase contrast, force modulation and magnetic force.

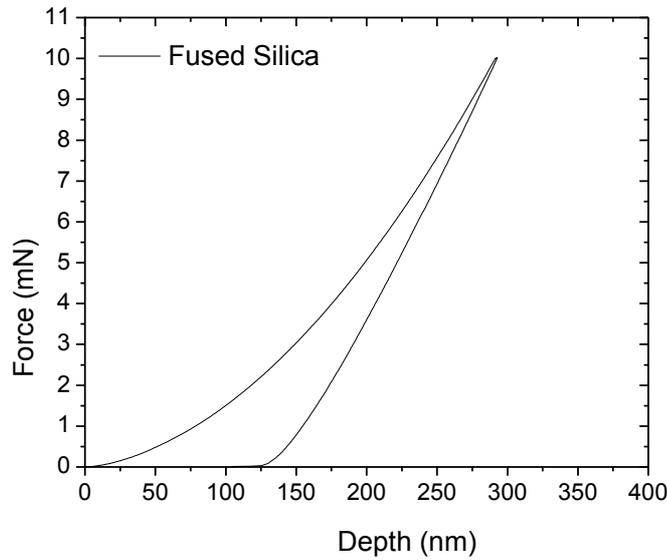
### 2.2. Delivery, installation and commissioning

The delivery, installation and commissioning has been carried out in two phases. The first phase, completed on 03.10.2014, encompassed the commissioning and acceptance testing of the Ultra Nanoindentation Tester only with regard to its room temperature measurement capabilities.

The second phase was delayed until 22.04.2014, due to a change in the design of the system by decision of the provider. In the final design the high temperature module included an infra-red source to heat the tip, while in the original design the heating of the tip was done by a laser. This second phase then addressed the commissioning and acceptance tests of the modules needed to perform tests at low ( $\leq -150\text{ °C}$ ) and high ( $\geq 700\text{ °C}$ ) temperature.

A first acceptance test of the load-displacement calibration was carried out by measuring the modulus of a Fused Silica reference sample at room temperature. The certified plane strain modulus of the reference silica is  $E^* = 75.4 \pm 0.3\text{ GPa}$  and the calculated indentation modulus is  $E_{IT} = 73.5 \pm 0.3\text{ GPa}$ . Figure 1 shows the load-displacement curve applying a maximum load

of 10 mN during 5 s, with loading and unloading rates of 20 mN/min. The measured moduli were within the errors of the certified values:  $E^* = 75.3 \text{ GPa}$ ;  $E_{IT} = 73.4 \text{ GPa}$



**Figure 1.** Load-displacement curve during Nanoindentation on a fused silica reference sample

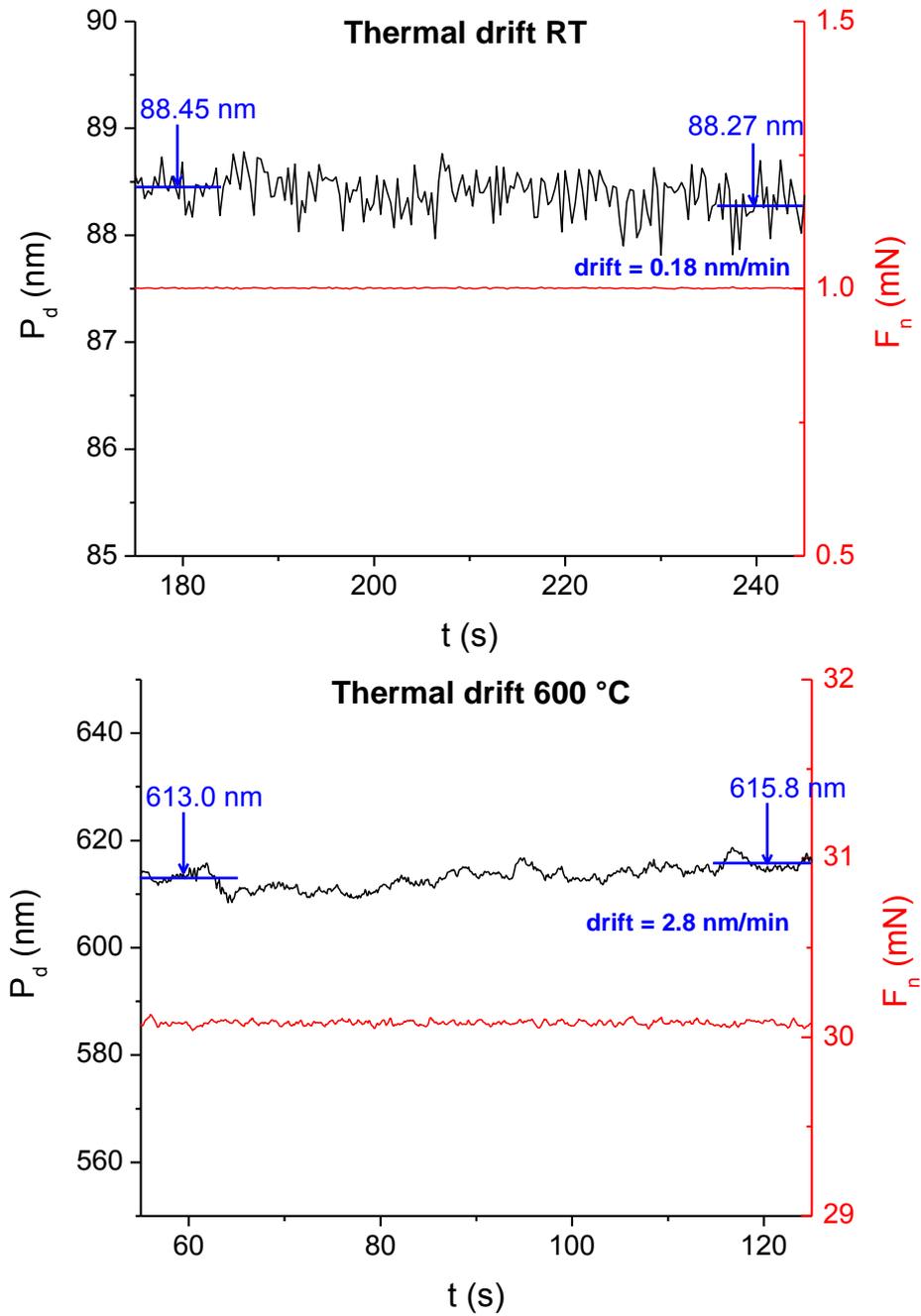
Further acceptance tests were carried out to validate the temperature range, stability and noise levels. Table 1 below lists the parameters tested:

Parameter	Requirement	Achieved during tests
High temperature indentation, including tip and reference heating	$T_{max}: \geq 700^{\circ}\text{C}$ $\Delta T$ of indenter to sample $\leq 3^{\circ}\text{C}$	$T = 700^{\circ}\text{C}$ $\Delta T = 2^{\circ}\text{C}$
Sub-zero indentation	$T_{min}: \leq -150^{\circ}\text{C}$	$T = -195^{\circ}\text{C}$
Secondary vacuum	$\leq 1 \times 10^{-5} \text{ mbar}$	$7 \times 10^{-6} \text{ mbar}$
Thermal stability	Room temperature (RT): $\leq 0.3 \text{ nm/min}$ Thermal drift at $600^{\circ}\text{C}$ : $\leq 20 \text{ nm/min}$	RT: $0.18 \text{ nm/min}$ $600^{\circ}\text{C}$ : $2.8 \text{ nm/min}$ (Figure 2)
Load noise floor	$\leq 0.5 \mu\text{N}$ in the whole temperature range	$0.19 \mu\text{N}$ at RT $0.27 \mu\text{N}$ at $600^{\circ}\text{C}$ (Figure 3)
Displacement noise floor	$\leq 0.2 \text{ nm}$	$0.14 \text{ nm}$ (Figure 4)

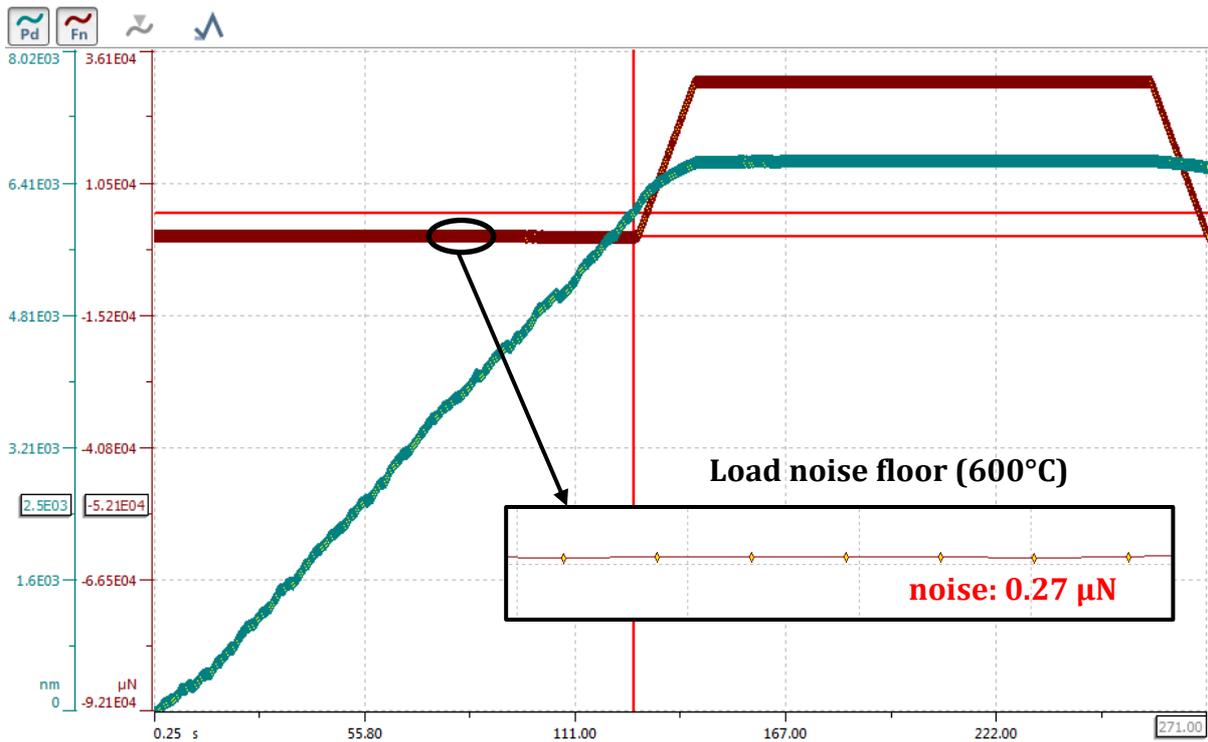
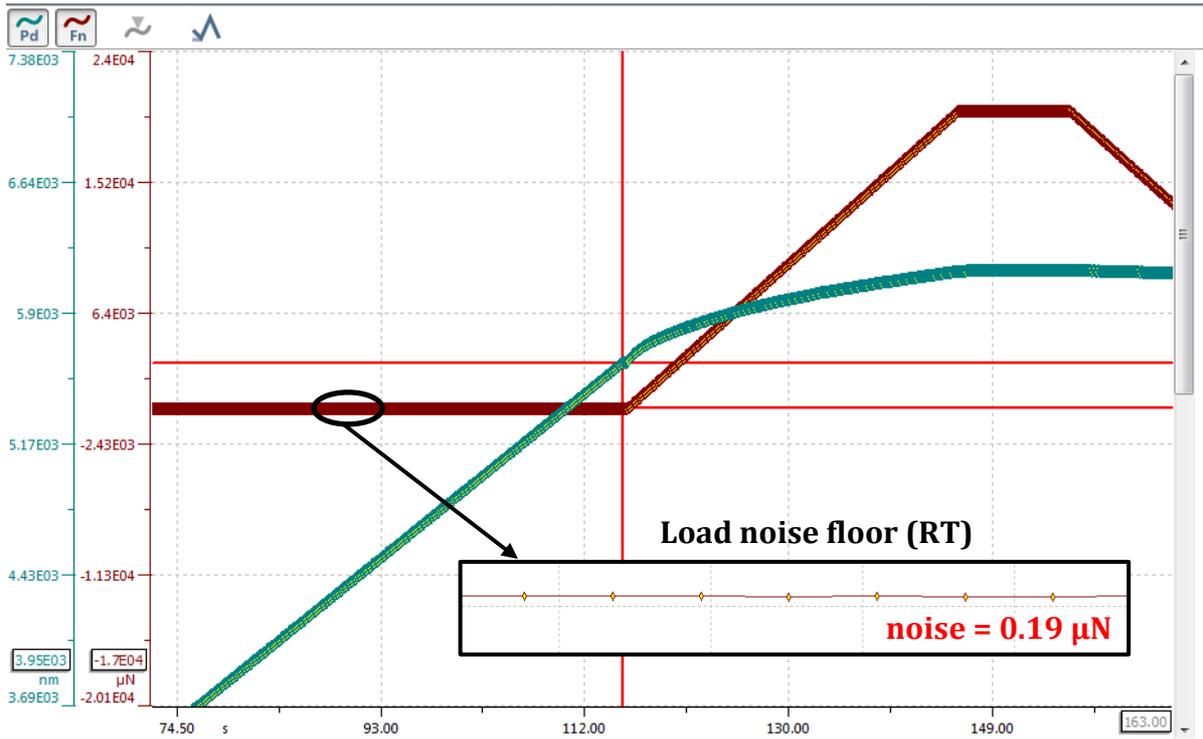
The temperature range was measured on a circumferential 316L bulk sample. Temperatures from  $-150^{\circ}\text{C}$  up to  $700^{\circ}\text{C}$  were achieved.  $700^{\circ}\text{C}$  were observed at an output power of the heater of 82%. The temperature difference between indenter and sample was around  $2^{\circ}\text{C}$ , i.e. below the value required in the technical specifications ( $\leq 3^{\circ}\text{C}$ ).

The thermal stability at room and high temperatures was checked doing a load controlled indentation test on fused silica and calculating the thermal drift from the displacement signal. Variations lower than  $0.3 \text{ nm/min}$  and  $20 \text{ nm/min}$  were measured at RT and  $600^{\circ}\text{C}$ , respectively, as required in the technical specifications (Fig. 2).

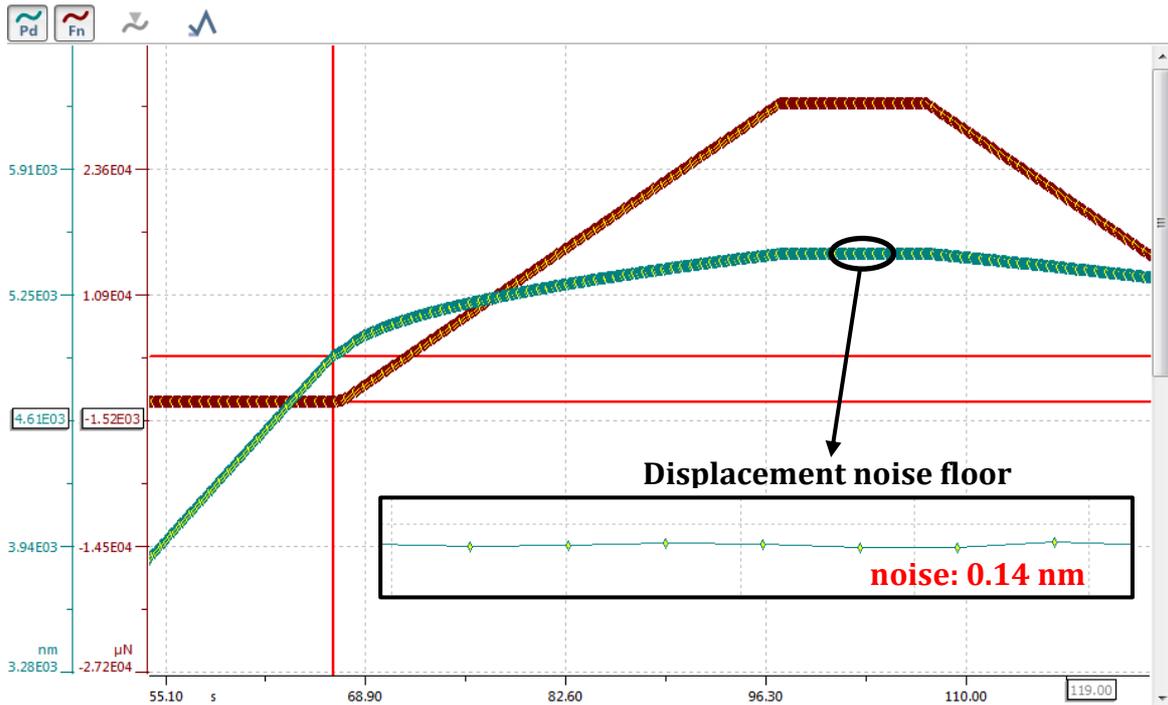
The load noise floor is calculated from the load curve during the indenter approach and the displacement noise floor from the displacement curve during the pause. Figures 3 and 4 highlight the data regions used for the estimation of the noise.



**Figure 2.** Evaluation of the thermal drift.



**Figure 3.** Evaluation of the compliance with the load noise floor value at room temperature and at 600 °C.



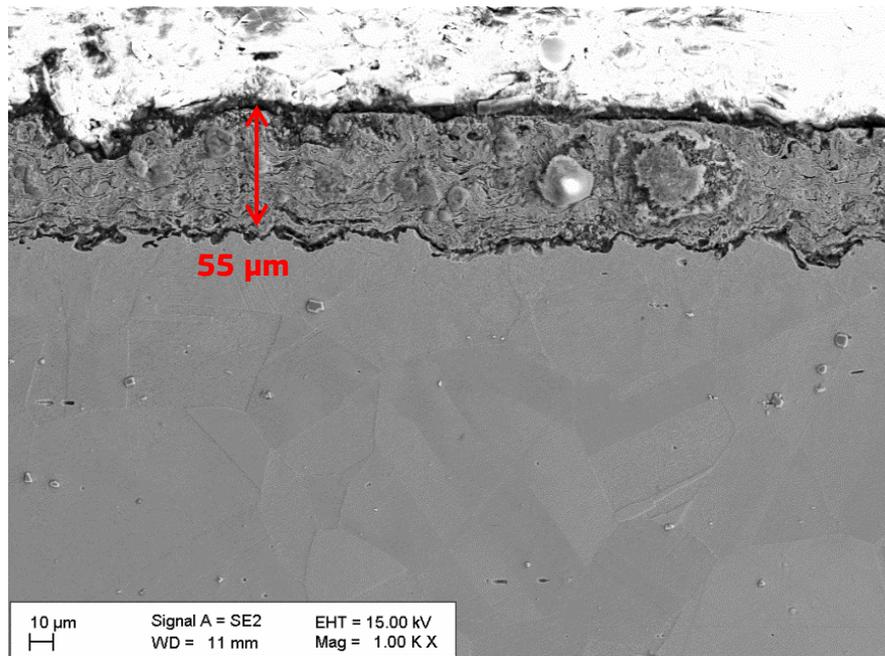
**Figure 4.** Evaluation of the compliance with the displacement noise floor value. Data acquired at room temperature.

### 2.3. Sample tests

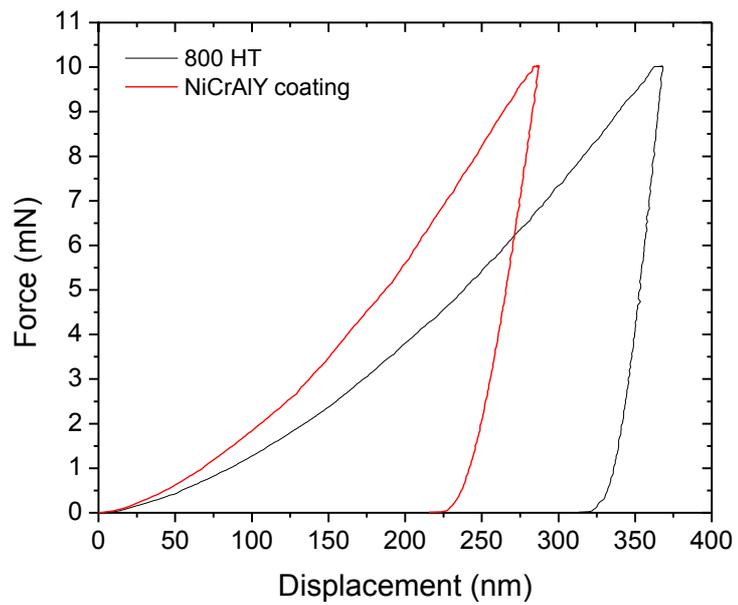
A basic measurement of a metallic sample was carried out during the commissioning of the equipment. Alloy 800 HT, a solid solution strengthened iron-nickel-chromium alloy, was used for the tests. The specimen had been coated with a layer of NiCrAlY by plasma spraying with the aim of improving the surface mechanical and corrosion properties. The sample was mounted in bakelite and polished. Figure 5 shows a SEM cross section of the sample and Figure 6 the load-displacement curves of indentations performed on the base material (alloy 800 HT) and on the coating (plasma spray NiCrAlY) applying a maximum load of 10mN during 10s, with loading and unloading rates of 20mN/min. Table 2 summarises the measured mechanical properties. The indentation hardness,  $H_{IT}$ , is expressed by the ratio between the applied load and the contact area and is a measure of the resistance to permanent deformation.  $E_{IT}$  and  $E^*$  are the indentation modulus and the plane strain modulus, respectively. The plane strain modulus is unaffected by elastic anisotropy.  $E_{IT}$  is calculated by multiplying  $E^*$  by a factor of one minus the square of Poisson's ratio. The results show a lower modulus of the coating and a much higher hardness as compared to the base material, in accordance to expectations.

**Table 2.** Measured hardness and moduli of a 800 HT stainless steel sample and its NiCrAlY coating.

	800 HT	NiCrAlY coating
<b><math>H_{IT}</math> (MPa)</b>	3118.9	5551
<b><math>E_{IT}</math> (GPa)</b>	185.13	168.27
<b><math>E^*</math> (GPa)</b>	203.44	184.91



**Figure 5.** SEM image of the NiCrAlY-coated 800 HT alloy sample.



**Figure 6.** Load-displacement curves during nanoindentation on a 800 HT stainless steel sample coated with NiCrAlY

The following additional sample tests of different materials were also carried out to verify the capabilities of the equipment at low and high temperatures:

**Test 1:** Matrix measurement on oxide-dispersion strengthened steel MA956 bulk sample at room temperature

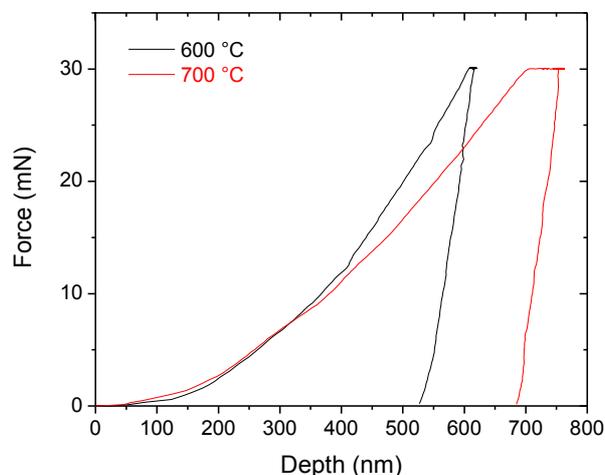
- Number of indents: 144 (12 x 12)
- Indenter material: Diamond
- Test parameters:
  - x / y spacing: 15 $\mu$ m
  - Load controlled
  - Loading rate: 20mN/min
- Max load: 10mN
- Pause: 10s
- Unloading rate: 20mN/min

A delayed start (at 7  $\mu$ m) of the measurement was programmed. Single indents were evaluated and faulty indents were removed. A hardness mapping and Young's modulus mapping was presented, concluding that the functionality and capabilities of the equipment to perform indentation test matrices and automated measurements was sufficiently demonstrated.

**Test 2:** High temperature indentation tests on 316L bulk sample.

- Number of indents: 7 (single indents)
- Test temperature: room temperature, 600°C and 700°C
- Indenter material: Tungsten Carbide
- Test parameters:
  - Load controlled
  - Loading rate: 120mN/min
- Max load: 30mN
- Pause: 120s
- Unloading rate: 120mN/min

Figure 7 shows the loading and unloading curves recorded at 600 °C and 700 °C, proving the capabilities of the equipment to carry out high temperature indentation tests up to 700°C as required.

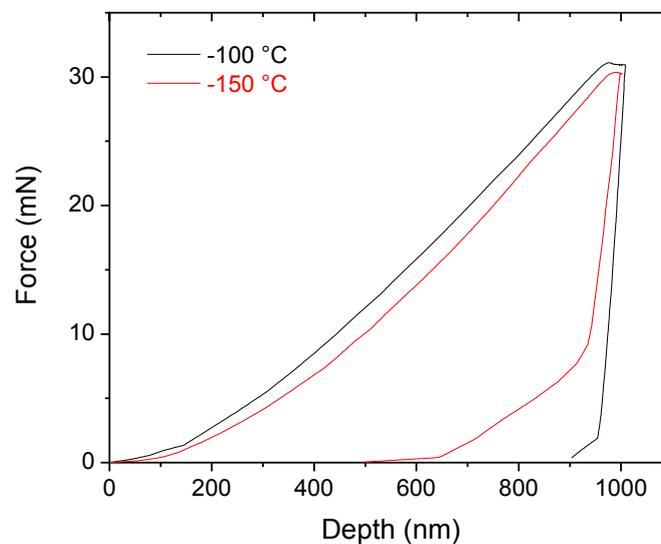


**Figure 7.** Load-displacement curves during Nanoindentation on 316L stainless steel at 600 °C and 700 °C

**Test 3:** Sub-zero test on Cu bulk sample at -100°C and -150°C

- Test temperature: -100°C, -150°C
- Indenter material: Diamond
- Sample material: Cu
- Test parameters:
  - Load controlled
  - Loading rate: 120mN/min
- Max load: 30mN
- Pause: 10s
- Unloading rate: 120mN/min

Figure 8 shows the load-displacement curves recorded at -100 °C and -150 °C. The tests have proven the capabilities of the equipment to carry out sub-zero indentation tests as requested in contract 111171.



**Figure 8.** Load-displacement curves during Nanoindentation on Cu at -100 °C and -150 °C

### 3. Conclusions

The delivery, installation, commissioning and acceptance testing of a High Temperature Ultra Nanoindentation Tester, coupled to a dedicated Atomic Force Microscope, have been successfully completed on 22.04.2014. The system was able to perform fully automated measurements. The temperature range, vacuum level, thermal stability and noise levels of the equipment were tested and met the technical requirements. The delay in submitting some of the parts, in particular the temperature-controlled tip and stages was due to the decision of the company to modify the initial design during production in order to deliver a more performing instrument.



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