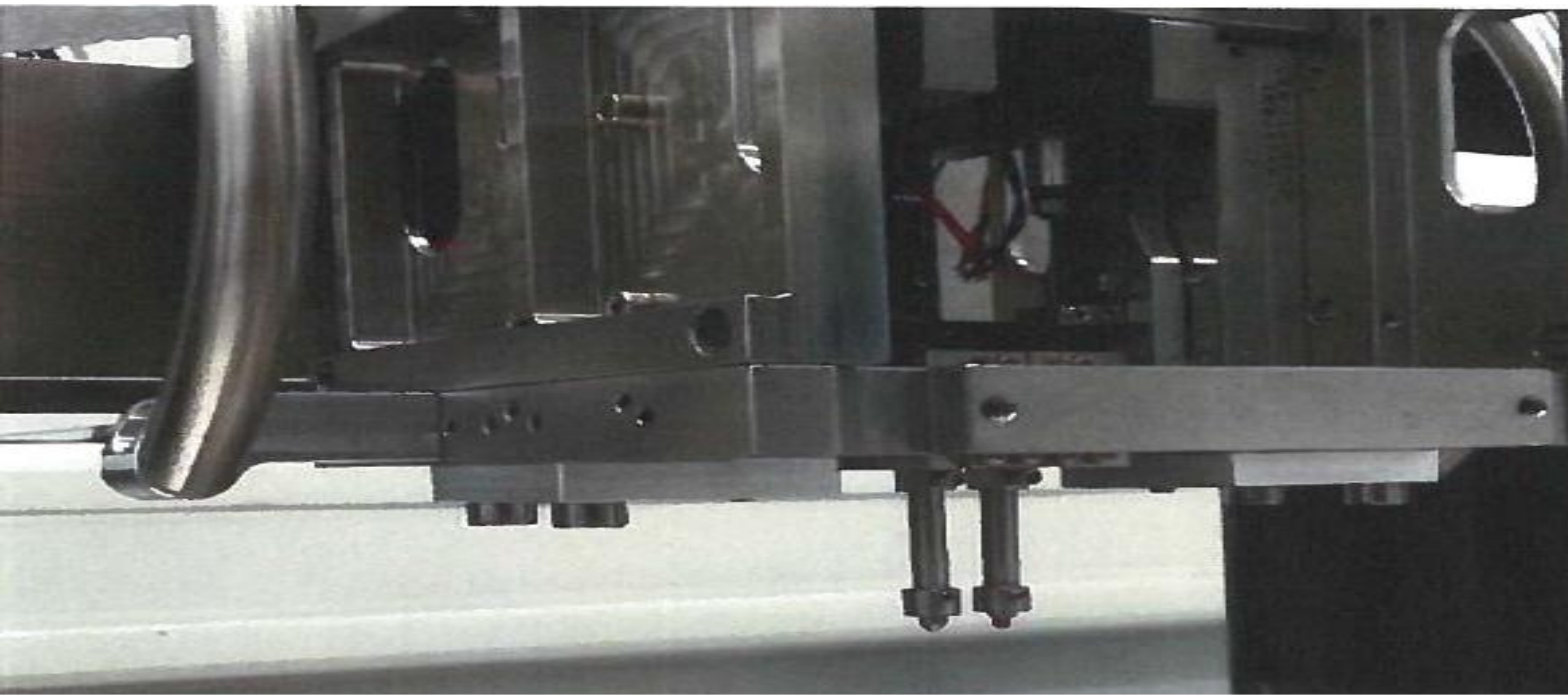
JRC Technical Systems



Installation and Commissioning of a High Temperature Ultra Nanoindentation Tester

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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) needed 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 precise 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 ≥ 1x10^7 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): ≥ 50mm 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 ≤ 0.3nm/min at room temperature and ≤ 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 μN in the whole range of test temperatures. The load resolution shall be ≤ 0.01 μN. The equipment shall offer a minimum contact load ≤ 1 μN in the whole range of test temperatures. The minimum usable load shall not exceed 100 μN in the whole range of test temperatures.
- The equipment shall provide a minimum usable displacement of 100 μm to perform Nano-Indentation testing. The displacement noise floor, under normal laboratory conditions, shall be ≤ 0.2 nm. The displacement resolution shall be ≤ 0.01 nm.
- The equipment shall provide a X-Y positioning resolution of the specimen stage ≤ 0.5μm. 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 ≤ 1 μm.
- 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 materiasl 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.

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 compatibility with the technical requirements. The evaluation committee decided to award the contract to ST Instruments, as the company provided solid documented evidence of their technical capabilities to meet specifications beyond the mandatory technical requirements within the price constraints and offered the most competitive quotation.

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, company at present acquired by Anton Paar. The CSM High Temperature Ultra Noanoindentation Tester allows the acquisition of accurate load-displacement data up to temperatures of ≥ 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 mechanisms 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, a greater thermal stability and a faster 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. In addition, the Ultra Nanoindentation head is made of Zerodur, a ceramic glass with an extremely low thermal expansion coefficient, and the thermal drift is also minimized by preheating the nanoindentation tip with a laser, while the sample is warmed up by conventional resistance heating.

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).
- Thermal drift free: the head is made of Zerodur glass and the electronic system has a drift rate of ≈ 1 ppm/°C.
- Load frame stiffness >> 10^8 N/m
- Two independent depth and load sensors
- Ultra high resolution and very low noise:
  - Depth resolution: 0.0003 nm, noise floor: 0.03 nm
  - Load resolution: 0.001 μN, noise floor: 0.13 μN
- Perfect 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 a Ultra Nanoindentation Tester on a modular high vacuum platform including heating (≥700 °C) and cooling (≤-150 °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 (≥ 700 °C), including a Ultra Nano Hardness head with laser tip heating.
- High temperature stage (≥ 700 °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 sown to -150 °C.
- Antivibration table for vacuum chamber.
- High load range (maximum load of 100mN) and high depth range (maximum penetration depth of 100 µm) sensors and actuators for Ultra Nanoindentation.
- Berkovich indenters of diamond and tungsten carbide.
- Spherical indenters of diamond and tungsten carbide.

A dedicated AFM 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, commissioned the Ultra Nanoindentation Tester compatible only with room temperature measurements.

The second phase was delayed until 12.12.2014, due to a change in the design of the system by decision of the provider. The second phase successfully commissioned the modules needed to perform tests at low (≤ -150 °C) and high (≥ 700 °C) temperature.

Acceptance tests were carried after calibration out by measuring the modulus of a Fused Silica reference sample. The certified plane strain modulus of the reference silica is $E^* = 75.4 \pm 0.3$ GPa and the calculated indentation modulus is $E_{IT} = 73.5 \pm 0.3$ GPa. Figure 1 shows the load-displacement curve, acquired with the equipment installed at IET, 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:

$E^* = 75.3$ GPa ; $E_{IT} = 73.4$ GPa

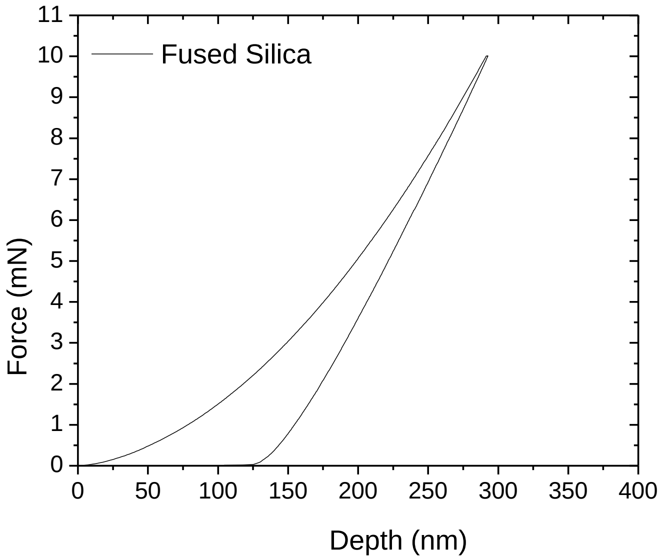

Figure 1. Load-displacement curve during Nanoindentation on a fused silica reference sample

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 2 shows a SEM cross section of the sample and Figure 3 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 10 s, with loading and unloading rates of 20 mN/min. Table 1 summarises the measured mechanical properties. The results show a lower modulus of the coating and a much higher hardness as compared to the base material.

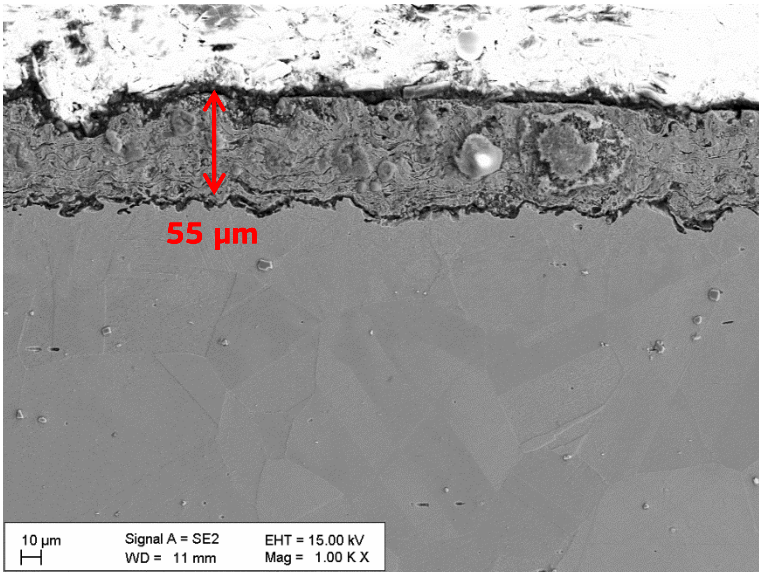

Figure 2. SEM image of the NiCrAlY-coated 800 HT alloy sample.

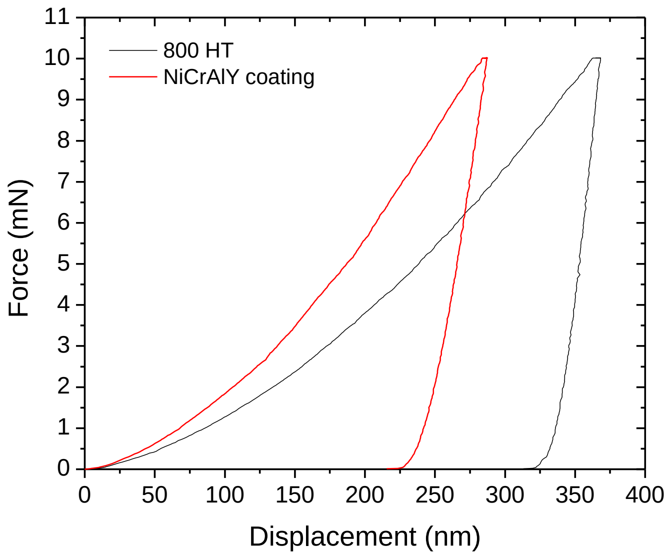

Figure 3. Load-displacement curves during Nanoindentation on a 800 HT stainless steel sample coated with NiCrAlY

Table 1. Measured hardness and moduli of a 800 HT stainless steel sample and its NiCrAlY coating

	800 HT	**NiCrAlY coating**
H_{IT} (MPa)	3118.9	5551
E_{IT} (GPa)	185.13	168.27
E* (GPa)	203.44	184.91

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 and the instrument is therefore fully operative. 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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