

Nanoindentation: Measuring in the Subnanometer Range

# PI in Flexible Materials Testing

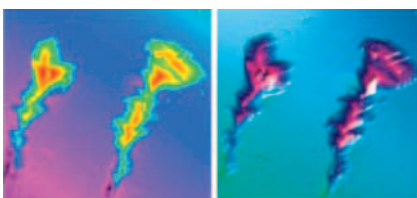


Capacitive Sensors and Parallel-Kinematic Piezo-Positioning Systems: Core Components for Nanometer-Precision Materials Testing

fracture toughness of small objects and microsystems in fields such as biotechnology. These measurements involve applying a small force to a sample using a sharp probe and measuring the resultant penetration depth. The measured value is used to calculate the contact area and hence the particular property of the sample material. Both the method of force application and the geometry of the indentation tip can be adjusted to suit the particular application.

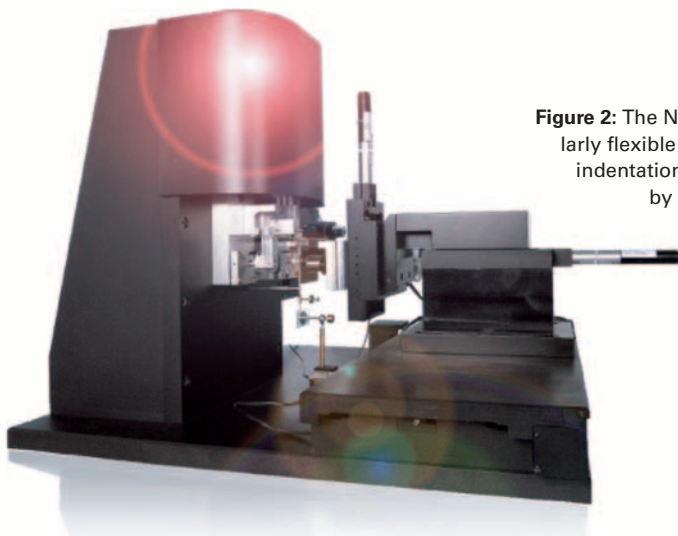
Continued on page 2

Figure 1: Scratches in a hard nano-composite layer on silicon before (left) and after (right) leveling



Nanoindentation is derived from the classical hardness test but is carried out on a much smaller scale. It can be used to determine the hardness of thin layers as well as material properties such as elasticity, stiffness, plasticity, and tensile strength, or

Contents	Page
Nanoindentation: Measuring in the Subnanometer Range	1 – 3
Motion Controller for Multi-Axis Piezo Systems	3
Lead-Free Piezo Material Enters Series Production	4
PI Ceramic on Continuous Growth	4
Picoactuator® with High Linearity	5
Phase Shifter with Picoactuator® Technology	6
Piezo Motor Helps to Monitor Railway Track Movement	6
Mini-Hexapod from PI	7
Ultra-Low-Profile Precision Rotation Stage	7
PI France – Official Partner of WONTON'11	8

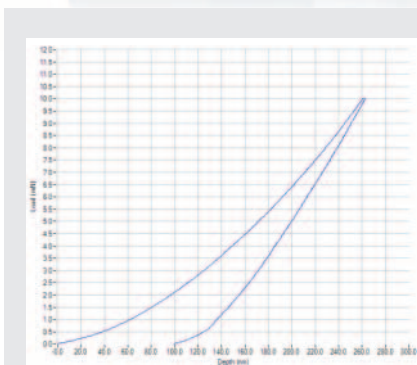


**Figure 2:** The NanoTest is a particularly flexible and powerful nano-indentation system, developed by Micro Materials Ltd.

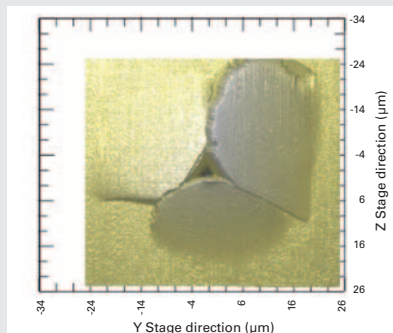
of the tip is recorded point-by-point and used to generate a three-dimensional image of the sample surface which can be evaluated with the appropriate software – also developed by Micro Materials (Fig. 4).

This combination of indenter and scanning probe microscope thus provides a quick method for assessing the sample surface. It is also possible to perform measurements at different locations without a great deal of effort.

A P-527 piezo-based nanopositioning system (Fig. 5) from PI provides the motions in the X and Y axes which are required for the scan. Its 200 x 200  $\mu\text{m}$  scanning range allows accurate statements to be made about the properties of the contact area and its surroundings. Thanks to the positioning accuracy of less than 3 nanometers a high positional resolution for the scan is guaranteed. This means, critical loads are accurately assigned to a defined position which in turn allows conclusions to be drawn about the local material properties, and the high number of closely packed measuring points make it possible to investigate very small samples (Fig. 6).



**Figure 3:** Load-displacement diagram



**Figure 4:** Surface profile around the indent point of the tip, derived from a scan with the nanopositioning system

### Hardness, Elasticity and Tensile Strength Tests

The system can be used to slowly increase the load over a defined period of time ('quasi-static indentation'), or to perform a dynamic test where the tip is accelerated towards the surface, depositing a large amount of energy on contact ('nano-impact'). The wear of materials can be assessed by moving the sample slowly to produce a scratch while applying either a constant or ramped load as the sample moves along (Fig. 1). The frictional properties of the surface are measured simultaneously. The system applies the force using electromagnetic actuation.

The NanoTest (Fig. 2) from Micro Materials Ltd. is suitable for use with a wide range of materials as it can apply forces of between 30 nN and 500 mN depending on the operating mode, and

it can measure penetration depths of between 0.1 nm and 50  $\mu\text{m}$ . This task is carried out by a high-resolution capacitive sensor, the PISeca sensor from PI. The capacitive sensor measures the penetration depth of the tip as a function of time. In static operation, the coil current and thus the load is measured at the same time, enabling a load-displacement diagram (Fig. 3) to be compiled. In dynamic operation, the load is derived from the effective acceleration of the loading head.

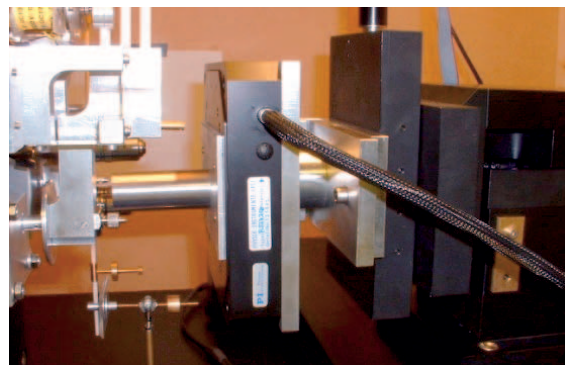
### Scanning Probe Microscopy Optimizes the Evaluation

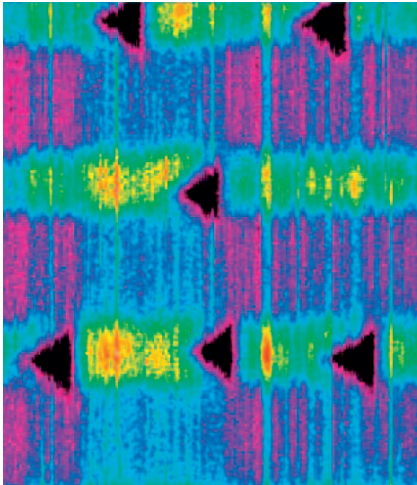
The indentation method employed by the NanoTest can be combined with an imaging technique to investigate the sample surface pre and post indentation. The indentation tip is moved row-by-row in a defined grid across the surface of the sample, that is, the sample surface is scanned. The displacement

### Nanometrology: Capacitive Position Sensors

The absolute measuring capacitive sensors operate with resolutions down to the sub-nanometer range, and provide depth information without coming

**Figure 5:** Integration of a piezo-based nanopositioning system with a 200 x 200  $\mu\text{m}$  large scanning range with the NanoTest.





**Figure 6:** Precise positioning of indentations measuring 60 nm across and 10 nm deep  
Illustrations: Physik Instrumente (PI) and Micro Materials Ltd.

into contact with the sample. They operate without wear and hysteresis, and do not affect the application itself.

A simple physical principle is at the heart of capacitive metrology: When a voltage is applied, a homogenous electric field is produced between the plates of a capacitor. When the gap between the plates changes, the output signal from the metrology electronics changes proportionally. Capacitive position sensors are available in two different basic designs, i.e. as one-electrode or two-electrode systems. The NanoTest uses sensors with only one electrode. A conductive disc mounted on the rear of the indentation tip serves as the second electrode.

### Micro Materials in Brief

A particularly flexible nano indentation system originates from Micro Materials Ltd. based in Wrexham/UK. The Nano-Test (Fig. 2) is used as an informative testing system in various fields worldwide. It is not only used in research and development but also in production and the testing of finished products. The system can operate over a wide range of temperatures, thus simulating in-service conditions.

The current MML NanoTest system offers a range of methods for materials characterization, including nanoindentation, nano-impact and nano-scratch and wear measurements. It is in use in leading universities and industrial laboratories all over the world.

### Compact Digital Motion Controller:

## Nanometer Precision for Multi-Axis Piezo Systems

The performance of precision positioning systems equally depends on their mechanical systems and on the way they are controlled. PI has more than a decade of experience with digital piezo control technology and now introduces its 6<sup>th</sup> standard digital controller line, the E-725 three-channel motion controller.

Thanks to digital technology, users have direct access via software to numerous parameters which optimize and adapt the nanopositioning mechanics to the actual application, by improving values such as the settling time and the linearity, or compensate for system inertia during fast scans. Among other performance features, the E-725 supports the dynamic positioning of specimen in the X, Y and Z directions for applications such as tracking or atomic force microscopy.

The E-725 provides modern interfaces such as USB and Ethernet. There are many software drivers available to support its integration as a subsystem in existing structures.



The E-725 digital nanopositioning controller with optional analog inputs for the control of three axes.

The E-725 in numbers: A powerful 32 bit processor with 225 MHz ensures a servo rate of 20 kHz. The position signal has a 24 bit resolution. Digital inputs and outputs offer direct trigger signals from and to the devices in the system environment. Optionally, the individual axes can be controlled via analog

inputs. In addition, the ID chip feature enables automatic calibration and exchangeability between piezo mechanics and controllers.

With that the E-725 replaces the successful E-710 controller which set the standard in nanopositioning for many years.

## PI Ceramic on Course for Continuous Growth

Albrecht Otto, Managing Director of PI Ceramic, has good reason to be pleased: In 2010, the record result of 2007 was exceeded by almost 30 %.

"We also were affected by the recession in 2008 and 2009," says Otto, "but the markets which PI Ceramic supplies with piezo components recovered quickly." At the forefront are applications for

fast, precise and compact pump and dosing drives, primarily in medical engineering and automation. "We have meanwhile almost reached our maximum capacity," adds Otto, "both in the piezo positioner sub-assembly business with PI as well as in our own business sectors (components, ultrasonic transducers, sensors, etc). We now employ more than

Model of PIC's piezo ceramics factory and R&D center in Lederhose – on the left is the new extension to the existing building which will increase the floor space available for production and offices by 5,000 m<sup>2</sup> (53,700 ft<sup>2</sup>).

Mr. Albrecht Otto, Managing Director of PI Ceramic



200 staff and our production already runs multiple shifts. The time has come to expand our facilities one more time" he said at the recent ground breaking ceremony.

The extension is scheduled to be finished by the end of the year, in time for the company's 20<sup>th</sup> anniversary. It will increase PIC's production capacity by 150 % and also allow additional production lines for multilayer and leadfree ceramics.

But PIC is not only pressing ahead with large-scale production. "PI Ceramic has always been very strongly oriented towards individually adapted ceramic components," and this is where Otto sees an important factor for their success. The new production area will therefore also include lines which provide a high degree of flexibility for the production of samples and small batches under conditions similar to those of series production.

### Lead-Free Piezo Material Enters Series Production

## PI Ceramic with Pioneering Technology

PZT – lead zirconate-lead titanate – is the material which is used to produce way over 90 % of today's conventional piezoelectric elements, sensors as well as actuators. It combines stiffness, reliability and displacement with high production throughput like no other material.

The lead is permanently bound in the PZT material and therefore completely harmless, which is why piezoceramics are excluded from the relevant European directive (RoHS).



The aim is nevertheless to minimize lead compounds in commonly used components. No leadfree piezoelectric material has yet achieved the high piezoelectric constants which PZT achieves.

### Lead-free Ultrasonic Transducers

PI Ceramic has now introduced PIC 700, a lead-free piezo material which is manufactured in a block using press technology. This material is based on bismuth-sodium-titanate (BNT). The piezoelectric characteristics are promising: PIC 700 has a high coupling factor for the flexural vibration and is thus suitable as an ultrasonic transducer in the MHz range and also for sonar and hydrophone applications. The maximum operating temperature is 200 °C.

PI Ceramic is currently investigating technologies to reliably manufacture

lead-free ceramic components in series production.

"With PIC 700 we have reached an important milestone in our materials development," says Eberhard Hennig, PIC's Development Manager and responsible for its materials research. "We can therefore supply lead-free piezo components for specific high-power sound applications in addition to our conventional elements. However, actuator ceramics (layers of bulk material or multilayers) which are optimized for displacement are still a long way off," emphasizes Hennig, and refers to the research results which have been presented at specialist conferences such as ACTUATOR in Bremen. Nevertheless he is confident that PI Ceramic is one of the forerunners in this field as well.

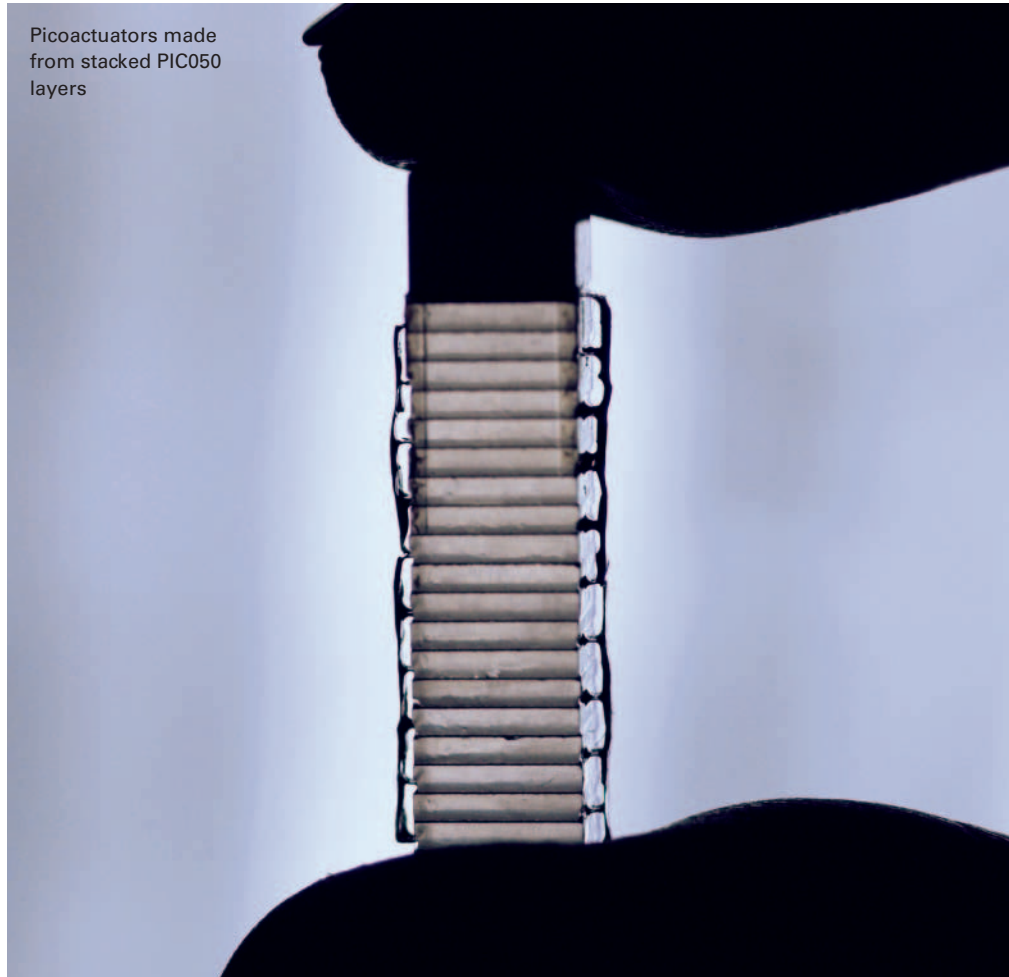
**Picoactuator® – “Good Things Come in Small Packages”**

# Lead-Free Piezo Actuator with High Linearity for Higher System Bandwidth

In precision positioning, the application requirements determine which actuator material is used. PZT is usually the material of choice due to its large displacement and high stiffness. The displacement of PZT material is associated with a hysteresis effect, however, which is caused by polarization effects in the crystal lattice. This hysteresis means that, the displacement not only depends on the applied voltage but also on the history i.e. if previously a higher or lower voltage was applied. For PZT the uncertainty is on the order of 10 % when operated in voltage control mode. Several orders of magnitude higher precision is available when operated in closed loop mode with a position feedback sensor and servo circuit.

The new Picoactuator® material enables PIC to provide longitudinal and shear piezo actuators which exhibit highly linear behavior in open loop mode. Picoactuators® are configurable up to heights of 20 mm and maximum travel of +/-3 µm. The main differences between this type of actuator material and conventional PZT can be summarized in one sentence: Good things come in small packages – picoactuators have a linearity which is 10 times better and a displacement which is 10 times smaller (see Table).

The advantage of high linearity in open loop mode (no additional sensor and servo electronics required) allows the design of smaller actuators with higher bandwidth and dynamics – up to one order of magnitude higher than those operated in closed-loop.



Possible applications are to be found in scanning force microscopy as the sample scanner in the XY plane or in the Z-direction with several 100 Hz scanning frequency. High-resolution microscopy

provides yet another application, where conventional piezo scanners are equipped with picoactuators to reduce deviations from the planar scan to below 1 nanometer in the Z-axis.

Approximate information to represent the orders of magnitude	Picoactuator®	PICMA® (Multilayer PZT)	PICA (PZT stack actuator)
Nonlinearity as % of displacement	1	10	10
Operating voltage in V for nominal displacement	+/-500	+100	+1000
Expansion for longitudinal actuators as % of the active actuator length	+/-0.005	0.1	0.1
Expansion for shear actuators as % of the active actuator length	+/-0.010	–	0.2
Lead-free	Yes	No	No

### Phase Shifter with Picoactuator® Technology

## Precision without Position Sensor



Phase shifter for an industrial interferometer, 5  $\mu\text{m}$  travel, equipped with high-linearity piezoelectric Picoactuators®.

Picoactuators® are piezoelectric drives which differ from classical PZT ceramics in that they are constructed from a crystalline, lead-free material. They feature a particularly high motion linearity enabling precision positioning without feedback sensor and closed-loop control.

Since picoactuators provide reduced displacement compared to conventional piezoceramics, ideal fields of application are those which combine high dynamics, small travel and high precision requirements.

In laser metrology, phase shifters are used to adjust optical path lengths, for example. They move optical systems by about a wavelength with a linearity of around 1 % (3 nanometers at a path length of 300 nm), which means that travel of a few micrometers is more than sufficient. The open-loop operation simplifies the mechanical design of the positioner and its electronic control, and increases the system bandwidth compared to a closed-loop servo which will add phase lag or delay.

### Direct Drives Increase the Measuring Speed of Tachymeters:

## Piezo Motor Helps Monitor Railway Track Movement

A great many factors come into play when selecting a drive system for a portable precision instrument: The installation space available, the velocities, the accelerations and positional accuracy required, the energy consumption and the reliability of the motorization selected. When increased demands are placed on an application, PLine® ultrasonic piezomotors are the obvious practical alternative to classic DC stepper motor combinations. Thanks to the employment of piezomotors, the performance of the geodesic measurement systems from Leica Geosystems AG has increased drastically (see PI Newsletter 40). They showed what they were capable of when monitoring the track of the Munich-Salzburg railroad as a road tunnel was being constructed underneath it.

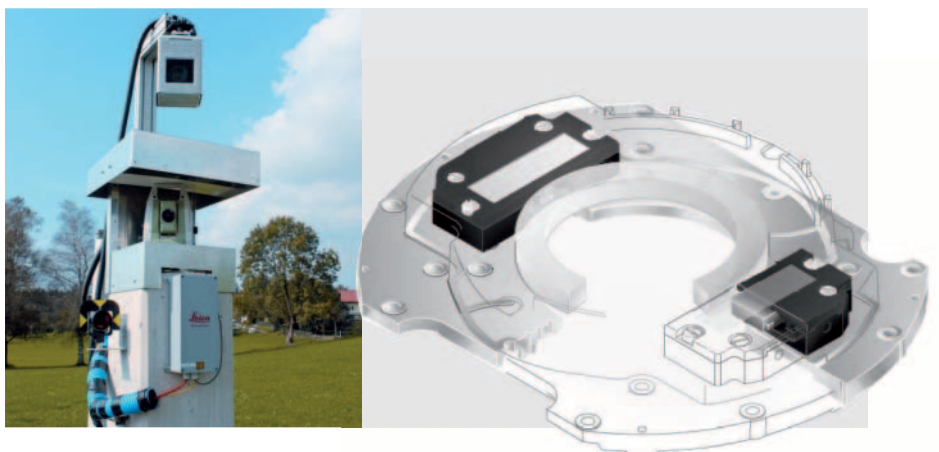
The engineering company "ing Traunreut GmbH" was here tasked with installing a monitoring system to safeguard the position of the track. The individually combinable measurement sensors from Leica Geosystems in conjunction with the matching software for monitoring, analysis and web-based data transmission proved to be just what was needed.

### Technical Progress Helped Along by Ultrasonic Piezomotors

Two tachymeters used for monitoring measure the change in position of the track and the motion of the overhead line masts. For this purpose, among others, the Leica TM30 monitoring sensor which uses the piezomotors was employed. The advantages which resulted are convincing: The speed of the tachymeter has increased to around 200 gon/sec and the measurements are now almost silent because no reduction gears are required. The high velocity

and the dynamic start and stop behavior allow a shorter time span between the measurement cycles. In addition the drives support a very high angle accuracy of up to 0.5". The tiny step sizes support high-precision measurements as are necessary for the monitoring.

Measurement pillar with webcam and TM30 monitoring sensor. The rotations are generated by two ultrasonic motors which are tangentially preloaded. The friction ring is pivot-mounted. (Photos: ing Traunreut / Leica Geosystems AG / Physik Instrumente (PI))





The compact M-811 Miniature Hexapod: Vacuum compatible, large travel ranges in six axes and an excellent positioning resolution.

**Mini-Hexapod from PI:**

## Six-Axis Motion in Vacuum up to $10^{-6}$ hPa

Positioning applications within vacuum chambers have high demands on the components used: The installation space is restricted and at the same time, a high degree of flexibility is needed in positioning objects so that the vacuum chamber does not need to be opened frequently causing contamination and loss of operating time.

The new M-811.STV Miniature Hexapod offers the perfect solution. Based on the even smaller M-810 Hexapod, this new design provides twice the stiffness in the horizontal axes. Despite to its compact size, with a diameter of only 130 mm and a height of 115 mm, the M-811.STV offers large travel ranges of up to 34 mm in the XY plane and of 13 mm in the Z

direction. What makes this Hexapod so versatile is the large tilting angles of  $20^\circ$  around the X and Y axis and up to  $40^\circ$  around the vertical axis as well as the pivot point which can be freely defined by the user.

The Mini-Hexapod reliably positions loads of up to 5 kg and achieves velocities of up to 10 mm/s. Each individual strut has a positioning resolution of 40 nm; complex multi-axis positioning tasks can be accomplished with submicron repeatability.

Like all Hexapod models from PI, the Mini-Hexapod is commanded by a powerful digital controller via Ethernet. All positions are conveniently given in Cartesian coordinates.

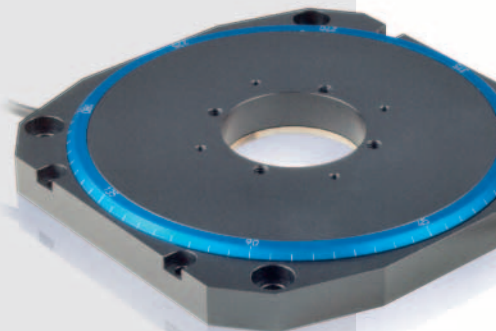
Parallel-kinematics systems have several advantages over stacked multi-axis positioners. All six actuators act on a joint platform, which keeps the moved mass low. Moreover, there is no summation of the lateral runout and tilt errors of the individual axes. The pivot point can be selected as desired via software commands and remains independent of the movement.

## Ultra-Low Profile

### Position Resolution of a Few Microradians thanks to Ultra-Low-Profile Precision Rotation Stage

One of the ultra-lowest profile stages on the market, the M-660 rotation stage, is now strengthened by a version with more than the eightfold position resolution of just  $4 \mu\text{rad}$ , that is  $0.00023^\circ$ . This offers additional possibilities for its application, for example in accurate inspection tasks. The new M-660.45 stage has a clear aperture of 35 mm in diameter. With just 15 mm in construction height it offers a particularly space-saving integration for example in already existing settings.

The drive with the PILine® piezomotor technology ensures the compact construction height as well as the high velocity of up to two rotations per second. The stage reaches a maximum torque of up to 0.3 Nm regardless of the direction of motion, and it is designed for a maximum load of 1 kg. The PILine® motors transmit the force directly onto the runner at the moving platform and thus keep the position stable, even when powered down.



M-660 rotation stage with ultrasonic piezomotors for an extremely low profile installation space

#### 4<sup>th</sup> Workshop on Nanotube Optics and Nanospectroscopy

## PI France – Official Partner of WONTON'11

The newest progress of the optical studies on nanotubes and graphene are the focus of this workshop which takes place in Bordeaux, France, from May 29 to June 1, 2011. PI France supports the organizer of the symposium, the University of Bordeaux, as an industry partner.

After the United States (2005), Canada (2007) and Japan (2009), Europe hosts the fourth WONTON workshop. Established and upcoming researches report and discuss the newest findings in nanotube optics and nanospectroscopy. The workshop not only offers highly qualified papers but is also a platform for exchange for the international participants.

The speakers offer interesting news in their abstracts and posters, the following topics will be presented among others:

- Light absorption, emission, and scattering
- Raman spectroscopy
- Carrier interactions, band structure and optical spectra
- Excitons in carbon nanotubes
- Spectroscopy of individual nanotubes
- Magneto- and electro-optics
- Nanotube-based optoelectronic devices and electroluminescence
- Growth, purification and separation of nanotubes for optical studies
- Spectroscopy of graphene

Find out more on:  
<http://www.rdv-routedeslasers.com/wonton11/index.php>



## Tradeshows 2011

Semicon West	July, 12 – 14	Moscone Center, San Francisco California, Booth #6342
SPIE Optics & Photonics	August, 21 – 25	San Diego Convention Center, San Diego California
Nanotech	June, 14 – 15	Boston Hynes Convention Center
Canadian Light Source	June, 24 – 25	Saskatoon SK, University of Saskatchewan
ICO -22	Aug, 15 – 19	Puebla Mexico

### Imprint

Publisher:  
 Physik Instrumente (PI)  
 GmbH & Co. KG  
 Auf der Roemerstrasse 1  
 D-76228 Karlsruhe  
 Reprint only with permission and citation of source.  
 Documents are made available.

**Editor:** Dr. Karl Spanner

**Editorial Staff:** Steffen Arnold, Stefan Vorndran, Sandra Ebler

**Creation & Design:**  
 Regelmann Kommunikation,  
 Pforzheim

**Production:**  
 Systemedia, Pforzheim

### USA

**PI (Physik Instrumente) L.P.**  
 16 Albert St.  
 Auburn, MA 01501  
 Tel. +1 (508) 832 3456  
 Fax +1 (508) 832 0506

5420 Trabuco Rd., Suite 100  
 Irvine, CA 92620  
 Tel. +1 (949) 679 9191  
 Fax +1 (949) 679 9292  
 info@pi-usa.us  
 www.pi-usa.us

### JAPAN

**PI Japan Co., Ltd.**  
 Akebono-cho 2-38-5  
 Tachikawa-shi  
 Tokyo 190  
 Tel. +81 (42) 526 7300  
 Fax +81 (42) 526 7301

Hanahara Dai-ni Building, #703  
 4-11-27 Nishinakajima,  
 Yodogawa-ku, Osaka-shi  
 Osaka 532  
 Tel. +81 (6) 6304 5605  
 Fax +81 (6) 6304 5606  
 info@pi-japan.jp  
 www.pi-japan.jp

### UK & IRELAND

**PI (Physik Instrumente) Ltd.**  
 Trent House, University Way,  
 Cranfield Technology Park,  
 Cranfield, Bedford MK43 0AN  
 Tel. +44 (1234) 756 360  
 Fax +44 (1234) 756 369  
 uk@pi.ws  
 www.physikinstrumente.co.uk

### ITALY

**Physik Instrumente (PI) S. r. l.**  
 Via G. Marconi, 28  
 20091 Bresso (MI)  
 Tel. +39 (02) 665 011 01  
 Fax +39 (02) 610 396 56  
 info@pionline.it  
 www.pionline.it

### FRANCE

**PI France S.A.S.**  
 244 bis, avenue Marx Dormoy  
 92120 Montrouge  
 Tel. +33 (1) 55 22 60 00  
 Fax +33 (1) 41 48 56 62  
 info.france@pi.ws  
 www.pifrance.fr

### CHINA

**Physik Instrumente (PI Shanghai) Co., Ltd.**  
 Building No. 7-106  
 Longdong Avenue 3000  
 201203 Shanghai, China  
 Tel. +86 (21) 518 792 98  
 Fax +86 (21) 687 900 98  
 info@pi-china.cn  
 www.pi-china.cn