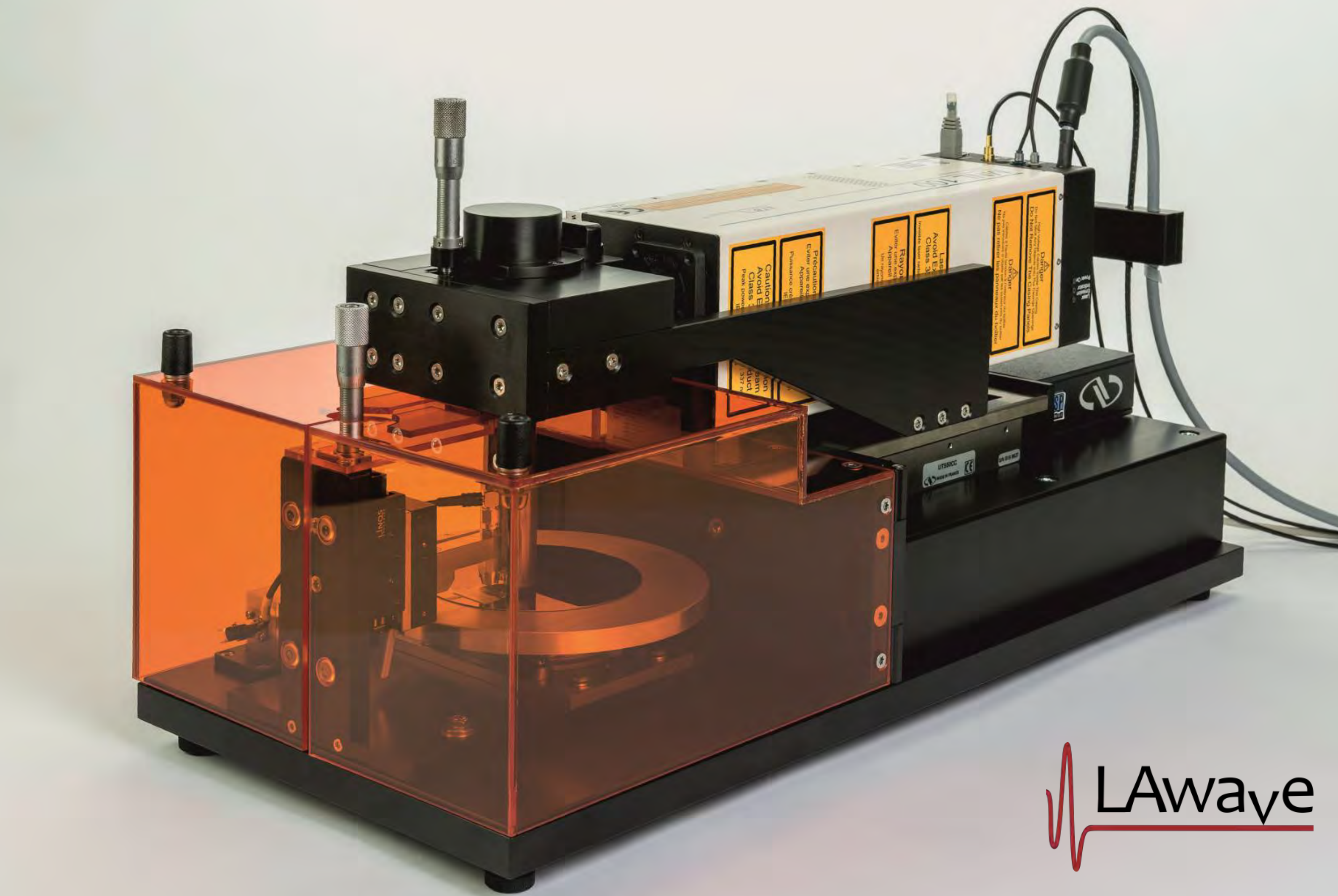
Fraunhofer

IWS Dresden

FRAUNHOFER INSTITUTE FOR MATERIAL AND BEAM TECHNOLOGY

LAWAVE – TESTING THIN FILMS AND MATERIAL SURFACES BY LASER ACOUSTICS

Non-destructive characterization by laser induced surface acoustic waves



OVERVIEW

LAwave is a measurement system that allows non-destructive characterization of films and material surfaces. It uses short laser pulses to create surface acoustic waves, measuring propagation velocity for all frequencies of the wave pulse.
The so-called dispersion curve depends on the elastic modulus (Young's modulus), density, and thickness of the surface layer.

The method has a wide field of applications, ranging from PVD coatings, CVD coatings, thermal-sprayed coatings, bulk materials, and many more.
Furthermore, various properties that affect the surface and subsurface can be obtained from the elastic behavior, like porosity, cracks, delamination, hardening, and machining layers.
The measurement is sensitive to films with a thickness ranging from only a few nanometers up to several hundred micrometers with rough surfaces, and can be applied for materials as soft as polymer and as hard as diamond.

The elastic modulus is a fundamental material parameter that characterizes the mechanical behavior of the material. The stronger the inter-atomic bonds in the material are, the higher the Young's modulus is. Therefore, it is a useful indicator of the microstructure. Micro-defects and pores represent missing bonds in the material which reduce the Young's modulus and allows evaluating defect density and porosity.

HIGHLIGHTS

- non-destructive
- film thickness from a few nanometers to several hundred micrometers
- film properties from polymer to diamond
- measuring time less than one minute
- high reproducibility
- measurement of integral properties
- easy to use
- simple arrangement
- measurement area: minimum 5 × 5 mm
- few requirements for the sample geometry
- low effect of surface roughness
- complies with EN 15042-1

PHYSICAL PRINCIPLES

Surface acoustic waves are elastic vibrations propagating along the surface of the material. The elongation is in the nanometer range and completely linear-elastic, therefore, the material is not irreversibly influenced by the measurement. Additionally, the penetration depth is only in the dimension of the wavelength while the wave energy is concentrated at the surface. For higher frequencies, the penetration depth of the wave is reduced and the effect of the film on the surface acoustic wave increases. Thus, films that are considerably thinner than the wavelength still influence the wave velocity.

Figure 1 displays two examples of dispersion curves measured on silicon wafers. For the uncoated sample, the wave velocity is constant and does not depend on the frequency.
For the wafer coated with 72 nm diamond-like carbon (DLC), the velocity increases due to the higher Young's modulus of the film compared to the substrate. As both samples have the same substrate properties, their curves intercept at the same point, f = 0 MHz, corresponding to the phase velocity of silicon.

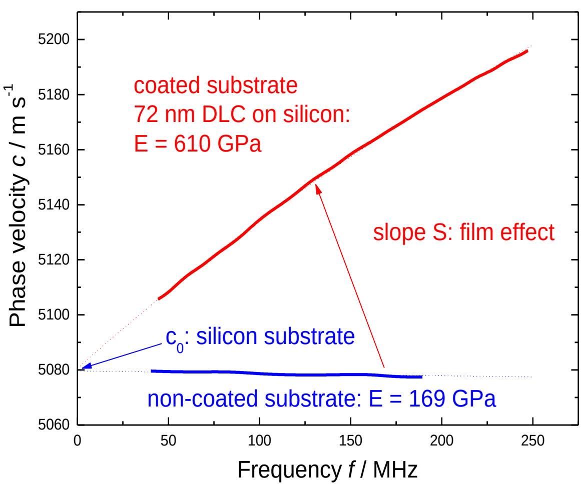


Figure 1: Effect of a 72 nm thick DLC film on the phase velocity c of the surface acoustic wave versus the frequency f. Solid lines represent measured data, dashed lines the fitted theoretical model.

After measuring the dispersion curve with the LAwave system, it is mathematically fitted to a theoretical model of wave propagation, where the material parameters of interest can be determined.

TECHNIQUE

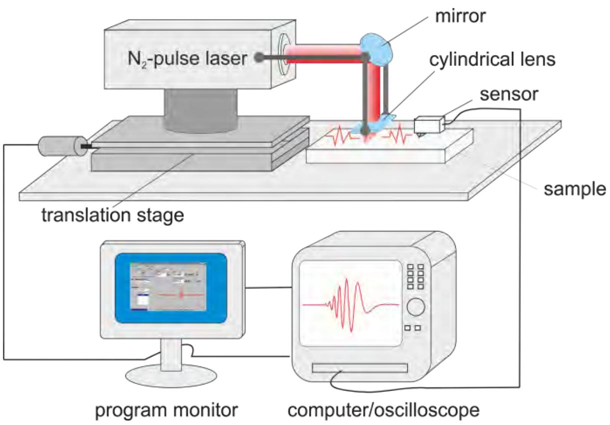


Figure 2: Schematic outline of the LAwave system

Figure 2 shows the main components of the LAwave system. A nitrogen pulse laser creates surface acoustic wave impulses. This non-contact method enables accurate positioning of the ultrasound source and reproducible creation of the wave impulses. A cylindrical lens focuses the laser beam into a line whereby an approximate plane wave is emitted. Due to the short duration of the laser pulse of 3 ns, an acoustic impulse with a wide frequency spectrum is created. After travelling through the material of interest, the ultrasonic impulse is received by an acoustic sensor that is in contact with the surface.

The acousto-electric properties of the sensor determine the frequency range of the measurement. A wideband sensor with a frequency range of 30 MHz to 200 MHz is used to measure thin films. The laser-acoustic signal is created by a piezo-electric PVDF foil connected to the surface by a steel wedge. For rough surfaces and materials with high acoustic damping, an ultrasonic standard sensor with a frequency range of 2 MHz to 20 MHz is used.
At the end, the laser-acoustic signal is amplified and recorded by an oscilloscope.

Both laser and its optical components are fixed on a translation stage and are moved together for a series of measurements. That way, the laser acoustic signals are detected at different distances x_i from the laser focus line.

The signals are Fourier-transformed and the phase spectra $\Phi_i(f)$ are calculated (i = 1 and 2). The dispersion curve c(f) can be calculated by the following relation.

$$C(f) = \frac{(x_2 - x_1)\, f\, 2\pi}{\Phi_2(f) - \Phi_1(f)}$$

The measurement of differences between two measurement locations is used to overcome the technical difficulties for the absolute measurement of the distance between laser focus and sensor.

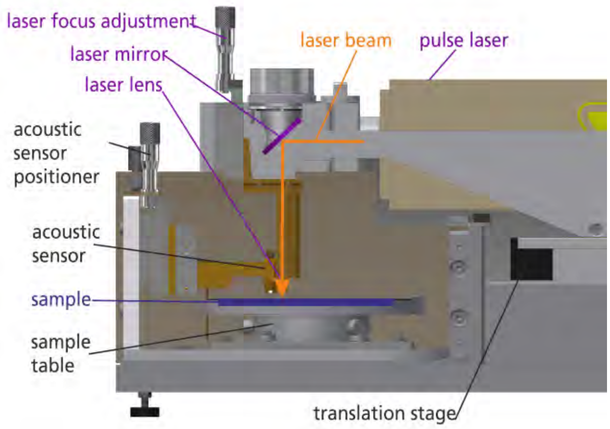


Figure 3: Cross section of the LAwave device, showing laser, optical, sample, and sensor components

Figure 3 shows the LAwave device. It consists of the following components:

- mirror to direct the laser beam to the sample surface
- cylindrical lens to focus the laser beam on the sample surface
- adjusting screw to focus the laser on the sample surface
- acoustic sensor
- manual positioner to connect the sensor to the sample surface

SOFTWARE

The measurement system includes three programs, which run on a Windows operation system.

The software that performs the measurement is shown in Figure 4 and Figure 5. It controls the laser, the oscilloscope and the translation stage during measurement. After having recorded a set of signals for different distances between the laser focus line and the sensor, the dispersion curve is calculated. An example is shown in Figure 4 for a silicon substrate coated with a diamond film.

The following data can be saved:

- dispersion curve in an ASCII file
- laser-acoustic signals in a binary file

The measuring module features a quick calculation of either Young's modulus or thickness of the film by fitting a theoretical curve to the measured curve.

For a more detailed analysis, the curve fitting software F5s and F6s are provided that allow fitting of several parameters. Figure 6 shows screenshots of the program with all parameters involved in the theory: elastic properties and density of the substrate as well as elastic properties, density, and thickness of the film. While the software F5s supports one and simple two layer systems, F6s considers up to five layers in a stack.

Data for not fitted parameters can be entered manually or taken from a data base, in which new materials can be added.

At the end of the analysis, material parameters calculated by the fit can be saved to an Excel file.

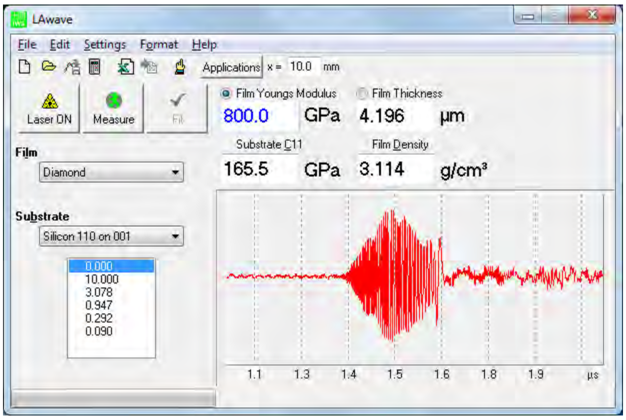


Figure 4: Laser-acoustic signal detected on a silicon substrate with a diamond film

Figure 5: Measured dispersion curve (dark blue) and fitted theoretical dispersion curve (light blue) for a silicon substrate with a diamond film. The pulse amplitude is shown as a brown line. The elastic moduli of film and substrate were calculated by fitting.

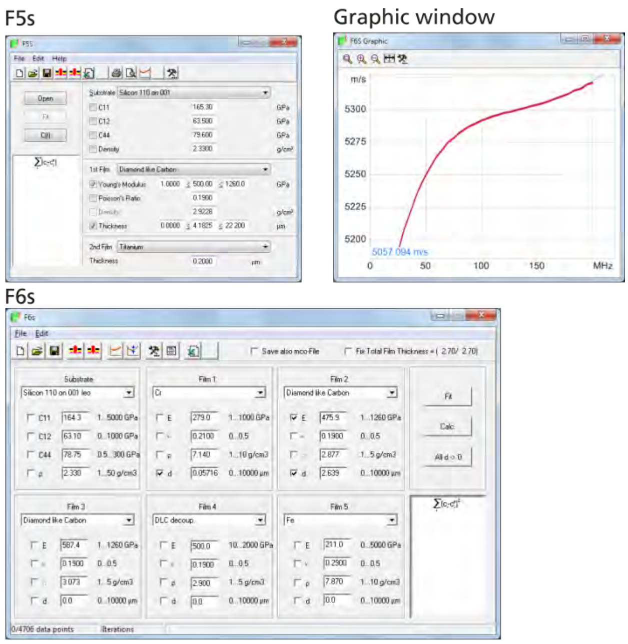


Figure 6: Screenshot of program windows for fitting single layer (F5s) and multi-layer (F6s) systems. The graphic window displays both the measured dispersion curve and the fitted theoretical model.

EXAMPLES OF APPLICATIONS

Nanometric DLC films

- film thickness: 3 nm to 100 nm
- film properties: super-hard

In case of hydrogen-free diamond-like carbon (DLC) films, the strength of the inter-atomic bonds determines both Young's modulus and hardness. A high ratio of sp^3 hybridized carbon atoms will increase the overall bond strength, which is favorable for protective coating applications. For example, DLC films with a thickness of only a few nanometers are suitable to protect the surface of computer hard disks, but it is difficult to reliably measure the mechanical properties of such thin films.

In previous studies, an empirical correlation of density and Young's modulus has been found for hydrogen-free diamond-like carbon films.
This correlation is implemented in the material data base and then considered in the fitting procedure as an additional boundary condition.

Films with thicknesses of a few nanometers change the wave velocity by only a few meters per second within a frequency range of 100 MHz. Therefore, the velocity has to be measured over a wide frequency range and with a high accuracy, which can be achieved by a long measurement distance.

In this case, a distance of 25 mm was used to measure the films with thicknesses lower than 10 nm. The values of the Young's modulus were scattered by less than 5 %. Due to the thin films investigated in this example, knowledge of the film thickness is required for the calculation of the modulus. The film thickness was measured by ellipsometry.

Figure 7 shows the values of the Young's modulus measured for DLC films with thicknesses of 3 nm to about 100 nm. Below 10 nm, the modulus decreases steadily with decreasing film thickness. This results from a very thin soft graphitic layer at the surface of the film, where the residual stress is not high enough for the carbon atoms to form diamond-like bonds.

Figure 7 also shows that a lower deposition temperature increases the Young's modulus. This result indicates that the contents of diamond bonds and hardness increase with decreasing substrate temperature.

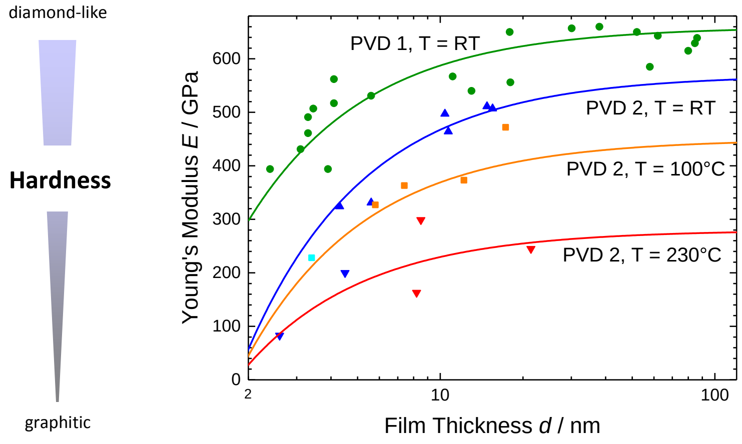


Figure 7: Young's modulus of hydrogen-free DLC coatings in dependence of coating thickness, showing the influence of deposition temperatures and different PVD technologies.

Sensor films

- film thickness: 200 nm to 600 nm
- film properties: polymeric

Polymeric films can be used to measure humidity, chemical substances, and mechanical deformation. Application of such sensors requires knowledge of their mechanical properties. Figure 8 shows the Young’s modulus and density of polyamide films measured with the LAwave method versus the implantation dose of B^+ ions.

The mechanical properties of polymers are described by the laws of viscoelasticity, making use of rheological models. In contrast to testing methods based on quasi-static deformation and the resonance vibration of the sample, the LAwave technique applies a very high deformation rate at very small deformations. Thus, the test yields the parameter for the linear-elastic behavior of the polymer, whereas the viscous flow is not stimulated by frequencies in the range of 100 MHz.

High frequency acoustic waves cannot penetrate bulk polymeric samples due to the high internal friction. However, the LAwave method can perform such tests if the polymer is deposited as sufficiently thin film on a substrate that serves as an acoustic waveguide. It has to be considered that there is a critical limit of the film thickness beyond which high frequency surface acoustic waves are completely damped by the polymer film. Depending on the properties of the polymer material, this thickness is limited to the range of 5 µm to 10 µm.

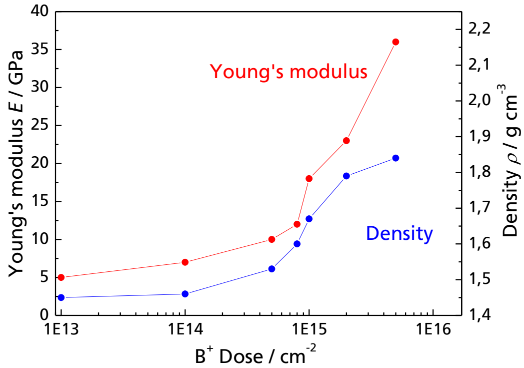


Figure 8: Young’s modulus and density of polymeric sensor films versus the B^+-ion implantation dose. Fitting the dispersion curve yielded both material parameters from one measurement.

Subsurface damage

- damage depth: up to 20 µm
- surface with high micro-defect density

Sawing a semiconductor wafer from an ingot produces a region of high defect density at the surface, which has to be removed by a careful grinding and polishing process. Determining the depth of this subsurface damage is desirable in order to optimize the machining process.

Figure 9 shows dispersion curves in the as-sawn state, after some grinding steps, and for the final polished surface of the wafer. The defective zone has the effect of a surface layer with a lower Young's modulus than the substrate. Therefore, dispersion curves decay with higher frequency. Removing this layer reduces the negative slope of the dispersion curve. For the finished wafer, a dispersion curve is measured that is parallel to the frequency axis, corresponding to a homogenous material. Hence, the slope of the dispersion curve depends on the depth of the subsurface damage.

A calibration function can be established to calculate the damage depth from the slope of the dispersion curve. Therefore, an as-saw wafer is repeatedly measured, while subsequently removing a defined portion of the damage layer by etching. This process is continued until the complete damage layer has been removed and the dispersion curve is a horizontal, corresponding to bulk silicon.
This calibration database is part of a special LAwave software version that is offered for this application.

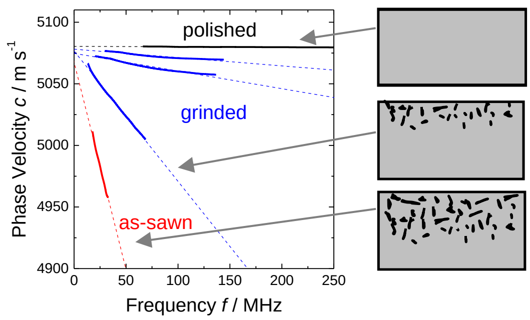


Figure 9: Dispersion curves measured on silicon wafer surfaces in the as-sawn, grinded, and polished state.

Thermal-sprayed coatings

- coating thickness: 100 µm to 600 µm
- rough, as-sprayed surfaces

Thermal-sprayed coatings often contain pores that result from the manufacturing process. These pores can significantly reduce stiffness and strength of the coating, which makes their characterization highly relevant to control both spraying process and coating properties. Conventional characterization by preparing a cross-section is time consuming and destructive, which is not an option for most components.
The LAwave method overcomes theses disadvantages by providing a fast and non-destructive method to assess the porosity of thermal-sprayed coatings.

Table 1 compares Young's moduli measured for sprayed coatings and the bulk material. The values of the coatings are sometimes considerably lower than those of the bulk material, indicating a high number of micro-voids in the coatings. The results also reveal the coating quality to depend on the spraying technology. HVOF-coatings (HVOF - high velocity oxy fuel spraying) have the highest Young's modulus, followed by suspension sprayed coating and APS sprayed coatings (APS - atmospheric plasma spraying). The results suggest that in this case HVOF-coatings are the most compact with highest mechanical strength.

Coating material	Spraying technology	Young's modulus of bulk material, GPa	Young's modulus of coating material, GPa
Al_2O_3	APS	350 - 400	68 ± 1
Al_2O_3	HVOF	350 – 400	113 ± 2
Al_2O_3	Suspension	350 – 400	101 ± 6
TiO_2	APS	250	76 ± 1
TiO_2	HVOF	250	111 ± 1
TiO_2	Suspension	250	88 ± 4

Table 1: Young's modulus of thermal-sprayed coatings compared with bulk material

The rough surfaces of the sprayed coatings often complicate the measurement. Roughness and defects also scatter ultrasonic waves of high frequencies. Therefore, measurements have to be performed in lower frequencies, where the surface acoustic waves are able to pass the material. Exchangeable acoustic sensors allow the choice of a suitable frequency range. In the case of thermal-sprayed coatings a transducer with a frequency range of 2 MHz to 20 MHz is used.

The effect of roughness on the laser-acoustic results was investigated for different thermal-sprayed coatings measured on the as-sprayed surface and after a grinding process to reduce the roughness.
Figure 10 compares the results obtained at the two surfaces with mean roughness index Ra. The slope and the coefficient of correlation of the linear regression confirm a good agreement of the results at both surfaces. As a conclusion, roughness in the range of several micrometers does not influence the results of the laser-acoustic method.

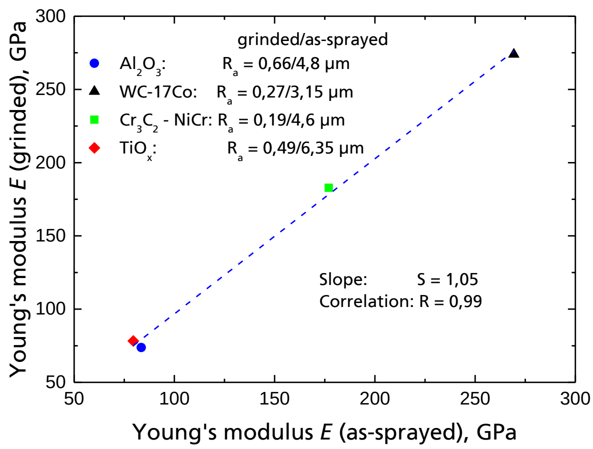
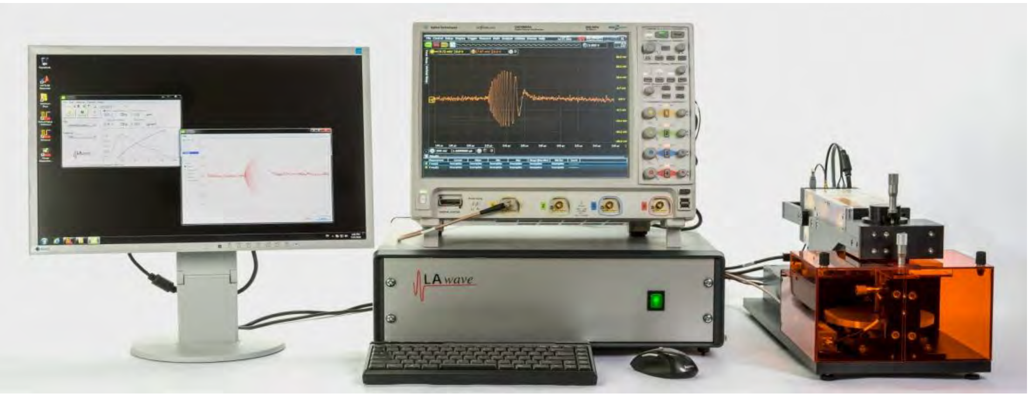


Figure 10: Comparison of Young's modulus of different thermal-spayed coatings measured with LAwave on the rough, as-sprayed surface and the grinded surface

Technical Specification LAwave 2G

Pulse laser with protective shield	Type Safety class without shield Wavelength Pulse energy Pulse duration Pulse repetition rate	MNL 103-LD 3B 337 nm $\geq$ 90 µJ 3 ns 10 Hz
Digital oscilloscope with integrated PC	Type Sampling rate Bandwidth Operating System	DSO Infiniium 10 GSa/s > 300 MHz Windows 7
Translation stage	Type Travel range Unidirectional repeatability On-axis accuracy	Newport UTS50CC Max. 50 mm 1 µm 5 µm for 50 mm
Repeat accuracy of phase velocity measurement	Measuring distance: 10 mm Measuring distance: 50 mm	$\Delta c/c \leq 1 \cdot 10^{-4}$ $\Delta c/c \leq 2 \cdot 10^{-5}$
Sample table	Diameter	Standard: 150 mm (standard) Optional: customized table
Acoustic sensor	High frequency range Low frequency range (optional)	30 MHz – 200 MHz 2 MHz – 20 MHz
Software	Measurement software Evaluation/Fitting	LAwave F5s (1 layer), F6s (up to 5 layers)
Dimensions (width × depth × height), weight	Laser device Oscilloscope & connection box	0.25 × 0.60 × 0.25 m³, 26 kg 0.52 × 0.50 × 0.55 m³, 30 kg



CONTACT ADRESSES WORLDWIDE

Germany

Fraunhofer IWS Dresden
Winterbergstrasse 28
01277 Dresden
Phone +49 351 83391 3096
Fax +49 351 83391 3300
www.iws.fraunhofer.de
martin.zawischa@iws.fraunhofer.de
stefan.makowski@iws.fraunhofer.de

USA

Fraunhofer USA
Center for Coatings and Diamond Technologies
1449 Engineering Research Ct.
East Lansing, MI 48824, USA
Phone +1 517 432 8709
Fax +1 517 432 8167
www.ccd.fraunhofer.org
ccd-info@fraunhofer.org

JAPAN

TOYO Corporation
1-6, Yaesu 1-chome,
Chuo-ku, Tokyo 103-8284, Japan
Phone +81 3 3279-0771
Fax +81 3 5205-2030
www.toyo.co.jp
egawam@toyo.co.jp

CHINA

GermanTech Co. Ltd.
Room 1408, Tower B, Techart Plaza
No.30 Xue Yuan Road, Haidian District,
100083 Beijing, P.R.China
Phone +86 10 82867920/21/22
Fax +86 10 82867919
www.germantech.com.cn
contact@germantech.com.cn

TAIWAN

Scientek Corp.
4F, No. 22, Taiyuan St. Chupei
Hsinchu Hsien
Taiwan 302
Phone +886 3 5526201 611
Fax +886 3 5526230
www.scientek.com.tw
jason.chao@scientek.com.tw