CHALLENGES

Real time nano CHAracterization reLatEd techNoloGiEeS

FINAL PROJECT RESULTS

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 861857

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Project Overview

CHALLENGES is a H2020 project that aims at developing multipurpose nano-optical techniques and metrological protocols for real-time characterization, capable to enable an increase of speed, sensitivity, spectral range with full cleanroom compatibility within different production environments, to improve devices performance, quality and reliability. The developed metrology technologies will enable the increase of speed, resolution, sensitivity, spectral range and compatibility within different nano-related production environments, finally improving products performance, quality and reliability, with the consequent bosting of competitiveness.

Objectives

- To provide a fully automated AFM-based tool
- To develop large sample XY piezo scanning stages
- To develop the optimum coupling solutions of light wavelengths range, AFM tip shapes and unconventional materials
- To design and demonstrate a nanoscale metrological NDT system that is compatible with production lines that need cleanroom environment
- To train a neural network capable to locate, with low-resolution hardware, relevant sites on the sample to probe with the high-resolution system, in a machine-learning framework
- To demonstrate the process-adapted nanoscale metrology for the manufacturing industry, through its use in three relevant industrial application contexts related to CMOS electronics, Silicon Photovoltaics and 2D Materials

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Expected Impacts

Laboratory-based characterisation of nanomaterials has been, and continues to be, one of the key enablers in the growth of knowledge and experience on nanotechnology and nano-enabled products. CHALLENGES will extend the scope of nano characterization beyond off-site laboratory based measurements to proper nanometrology, and is expected to have impacts on industrial production of nano-enabled materials and devices starting from the applications targeted within the project, but with impacts potentially able to be propagated to all major nanomanufacturing processes.

01 **Measurable improvement of speed by at least a factor 2 of nanoscale characterisation procedures, in comparison to already established performance and reliability for the application leading to a significant increase in industrial competitiveness**

02 **Significant reduction of the time and resources needed for nanomaterial development and upscaling, and for nanomaterials-based product development, which should be quantified with respect to established conditions for specific market sectors, with a return of investment in less than 5 years**

03 **Quantifiable enhancement of the ability to control the quality and reliability of products, with consequent improvement of product lifetime**

04 **Quantifiable enhancement of the ability to control the quality and reliability of products with associated environmental benefits**

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Results & Dependencies on other WPs

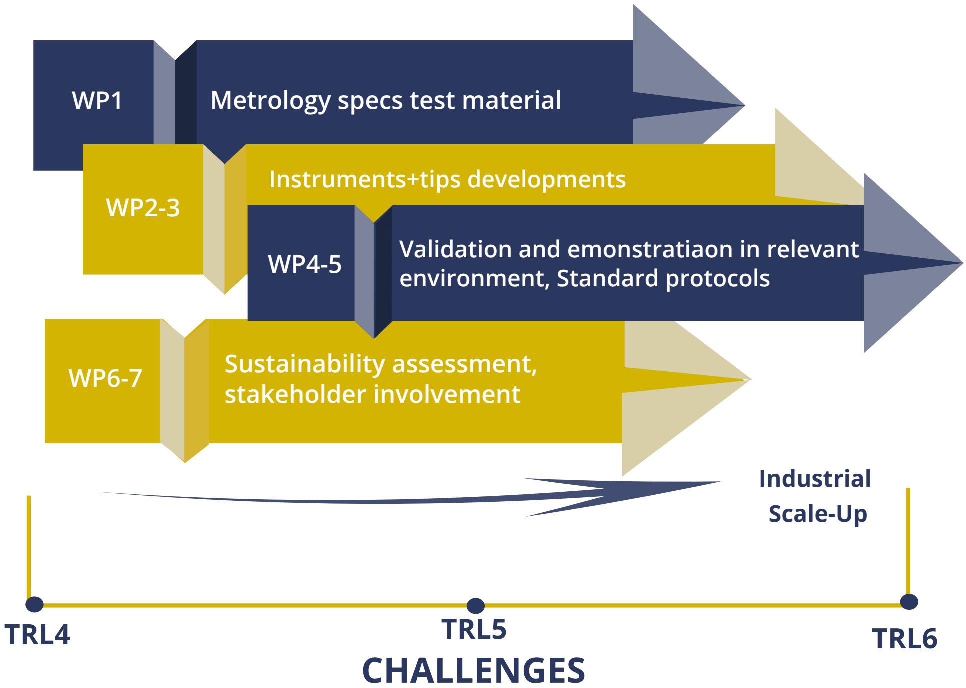

Figure 1: Progress of the technology development foreseen in CHALLENGES, leading to future industrial scale-up after the project

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Main contacts

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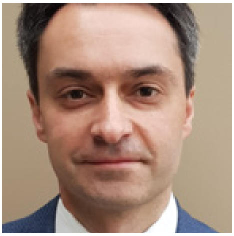
Project Results

Development and manufacturing of materials

Ivan Gordon | Department Director IMOMEC at IMEC / Energyville



CHALLENGES started with the manufacturing/acquisition of the test samples needed for the characterization work in subsequent work packages.

The choice of test samples was pivotal for the success of the project, and it has been based on the needs of the industrial partners, about manufacturing requirements and characteristics.

Two sets of test samples were benchmarked using standard industrial characterization methods, to guarantee the compliance with the expected specifications:

Samples to validate the new plasmonic materials and be able to isolate and fully characterize each physical quantity of interest ("standard samples")

Industrial semi-finished (or finished) products to be used for developing and testing the in-line metrology and adapting it to the specific requirements of each application ("industrially-relevant samples").

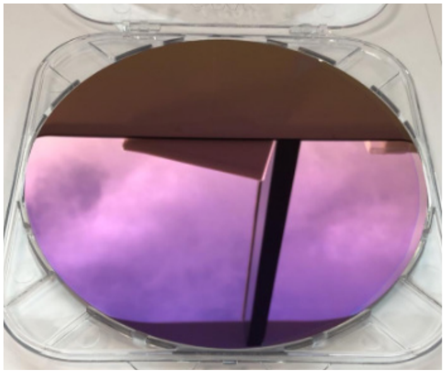


Figure 2: 200mm monolayer of grahene that is deposited on a SiO2/Si substrate.

Both standard and industrial samples were provided for the following targeted applications:

- Semiconductor Industry: CMOS electronics (representative picture)
- Photovoltaics (representative picture)
- 2D Materials

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SAPIENZA
UNIVERSITÀ DI ROMA

Development of clean room compliant tips

Daniele Passeri | Associate Professor, Department of Basic and Applied Sciences for Engineering at SAPIENZA University of Rome

One of the main results of CHALLENGES has been the development of advanced plasmonic tips, which are fully compatible with industry production lines. These tips ensure high reliability and performances in terms of plasmonic amplification, resolution and stability. Moreover, these performances have been obtained using materials which are not 'poisonous' when used in microelectronic industrial environments: these requirements inhibit the use of standard SNOM tips which employs noble metals. Therefore, CHALLENGES has implemented two different strategies:

- coating of standard glass SNOM tips with not noble metals
- manufacturing of advanced tips with or without grating systems

The approach followed required a "circular" interaction between the theoretical activities of simulation of advanced tip structures and prediction of their performances, the practical activities of industrial realization of the designed probes, and the experimental characterization and verification of their performances.

The design of advanced tips was performed on the basis of needed specifications, by UNISAP and TIBERLAB who combined different approaches, i.e., finite element analysis or first-principle calculation, for the simulation of tip-laser interaction, in order to optimize their shape and materials.

The practical realization of tips on the basis of the results of the simulation activities was carried out by SCANSENS and NANONICS.

The actual performances of the realized tips was assessed using conventional instrumentations by NANONICS and UNISAP.

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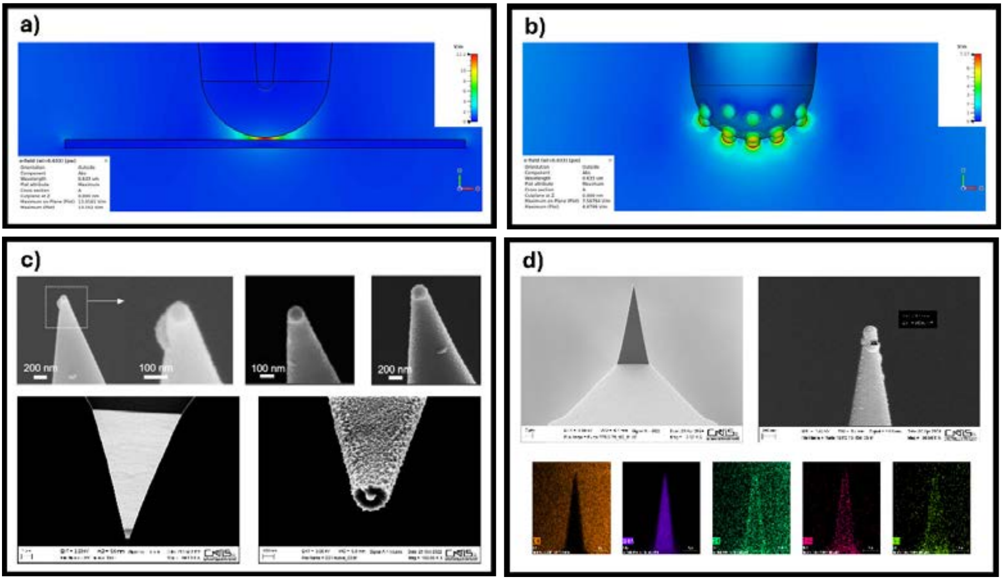

Figure 3:

- *(a) Electric field simulation for a TiN-coated Si probe on a Si substrate in the x-y plane.*
- *(b) 3D electric field simulation for a TiN-coated Si probe with surface roughness.*
- *(c) SEM images of TiN-coated tips from the first and second optimization iterations.*
- *(d) SEM and EDX analysis of TiN-coated tips from the final optimization iteration.*

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Instrumentation development

Aaron Lewis | Founder of Nanonics Imaging

We developed the two main components of the instrument:

- AFM components (NANONICS);
- spectroscopy components for Raman and mid-IR (SOL Instruments).

1.The AFM components

Were able to provide the following advantages in terms of capabilities and optical integration:

- Measurement of phase and amplitude
- Normal and lateral force
- High resolution stage
- Fully integrate with upright microscope
- Inverted microscope if needed
- Optical, Electrical and Thermal measurement without artifact of feedback

2.The spectroscopy components

Spectrometer developed by SOL instruments is designed to solve a wide range of routine and research tasks related to registration and processing of spectra, quantitative and multi-component analysis, as well as kinetic measurements.

The spectrometer features:

- high optical throughput and sensitivity of illumination and detection channels
- high spectral resolution. Spectral resolution of Raman spectra - 0.16 cm-1 (excitation wavelength 532 nm, Echelle grating 75 l/mm, 45-th order)
- possibility to operate with the laser wavelengths necessary to trigger the plasmonic resonance on the AFM tips
- high long-term spectral stability (there is the integrated the reference light source for spectral calibration and validation)
- far field confocal spatial resolution that is the closest possible to the theoretical diffraction limit
- fully automated device

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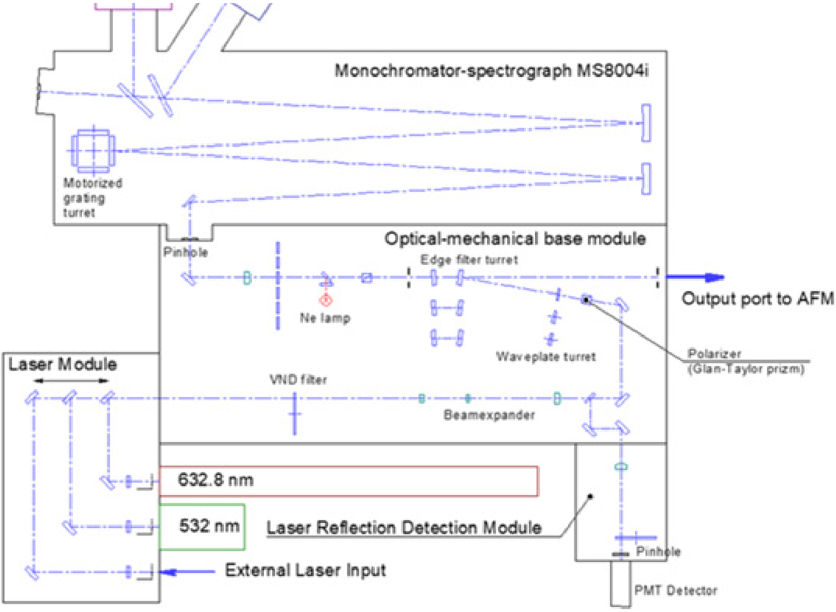

Figure 5: Spectrometer structure

Working spectral range of the system:

- Excitation (Probing) wavelength range: - 530 – 800 nm;
- Detection wavelength range: - 530 – 1700 nm.

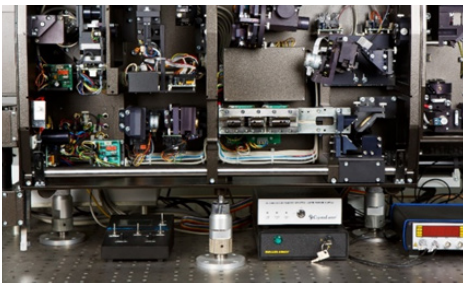

Figure 6: Spectrometer for Raman and mid-IR

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Application and validation

Roberto Balboni | Researcher at CNR-IMM

Results achieved:

Definition of specifications of test samples for the validation activities (linked to WP1)

- Definition of material for different analyses
- New 'standard' materials (SiGe epitaxial layers on Si, prepared by CEA)

Validation with TEM and X-Ray based characterization techniques:

- TEM sample preparation by FIB: design and realization to preserve strain configuration
- TEM imaging and strain analysis on DIVA structures, Photovoltaics samples and SiGe standards
- X-ray analysis crystal quality and strain measurements on Photovoltaics samples and laser annealed Si crystalline samples from ST-C

Validation with Optical-based characterization techniques:

- Raman analysis of strain, crystal order and defect analysis on DIVA samples, Photovoltaics samples and laser annealed Si crystalline samples and SiGe standards
- PL measurements for Crystal quality and Band-gap analysis on on DIVA samples and Photovoltaics samples

Plasmonic, TERS and TEPL measurements with standard and new tips:

- TERS strain analysis on DIVA samples
- TERS and TEPL Crystal quality and defect analysis on Photovoltaics samples and laser annealed Si crystalline samples and SiGe standards

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Development of measurement protocols

Reiner Stosch | Head Of The Department Of General And Inorganic Chemistry at Physikalisch-Technische Bundesanstalt

The main objective was to set up a framework for establishing metrological support of the CHALLENGES methodologies by identifying and qualifying industrial samples as calibration specimens.

A subset of test samples was selected and used for a statistically strong metrological basis. For traceability and validation purposes, an appropriate set of samples was also selected from outside the consortium to support the metrological work within the consortium. In particular, appropriate SixGe1-x epitaxial samples of different composition have been identified and showed a compositional dependence of strain/stress parameters. The latter corresponds to wavenumber shifts of relevant bands in Raman spectroscopy.

Using conventional and new characterization techniques and standard samples, we were able to assess the reliability of novel approaches proposed in CHALLENGES, in particular linked to baseline precision and accuracy of lab instrumentation.

The results underpin the choice of Si WP6 provides the main elements to set the stage for the after-project exploitation of the solutions proposed by CHALLENGES, including an assessment of sustainability both from the economic and the environmental points of view.

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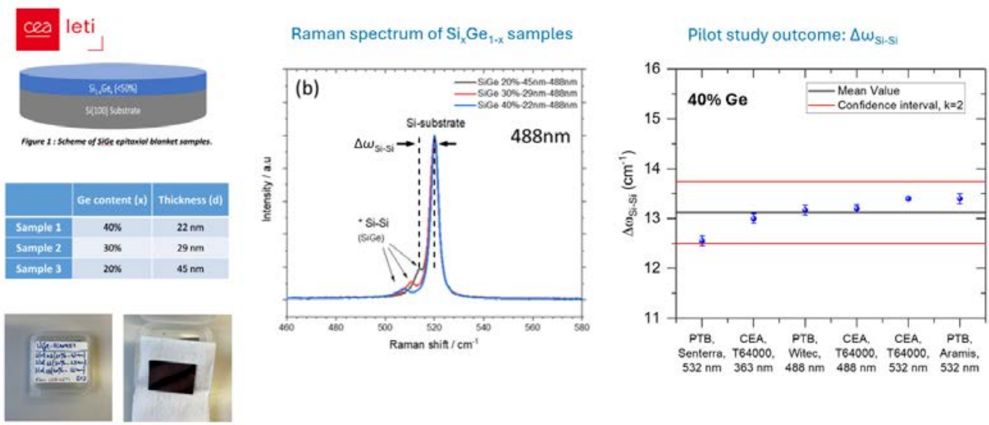

Figure 7: The figure shows SiGe layers deposited on Si substrates. The Ge content and thickness can be varied, resulting in different strain/stress values that are correlated with Raman shifts as can be seen in the middle. A pilot round robin activity rvealed a good agreement of 6 participating installations.

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Exploitation and environmental impact

Lisa Bregoli | EU project expert and manager at Warrant Hub (Tinexta Group)

The main elements to set the stage for the after-project exploitation of the solutions proposed by CHALLENGES, including an assessment of sustainability both from the economic and the environmental points of view.

Key Exploitable results:

We have developed exploitation routes for the Key exploitable results (KER), following two different strategies which depend on the type of interest:

✓ EXPLOITATION of technologies and tools as **TECHNOLOGY PROVIDERS:**

- Plasmonic tips made of not noble metals materials (SCANSENS)
- Automated AFM-based tool, optimized for plasmonic enhancement of optical signals in industrial production environments (NANONICS)
- Raman and Luminescence spectrometer (SOL)
- Machine learning algorithms between tip enhanced tools and far field metrology tools (NOVA)
- Data management system for Raman spectrometers (TIBERLAB)

Figure 8: Exploitation of technologies and tools as Technology providers

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✓ EXPLOITATION of technologies and tools as **ENDUSERS:**

- New insights into the formation and reorganization of porous silicon for the growth of epitaxial silicon wafers for photovoltaic devices (IMEC)
- Evaluation of TERS technic to characterize advanced semiconductor device and materials and round robin between partners on standard Raman technics on CEA's samples (CEA)
- SI-traceable Raman spectroscopic procedure to measure and quantify stress/strain at the surface of semiconductors and 2D materials and experimental set-up for SI-traceable Raman-band measurement and Raman spectrometer calibration (PTB)
- Introduction of the Raman spectroscopy analysis into the PV field (AMAT)
- Test Raman spectroscopy analysis on advanced CMOS structures and evaluate calibration protocol for quality industrial requirements (ST_C)
- Industrial in-line Quality Control method for graphene wafer production line (GRAPHENEA).

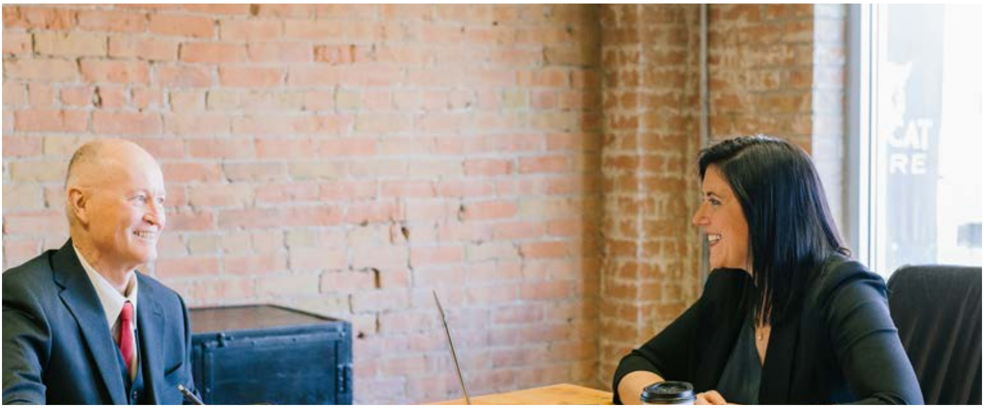

Figure 9: Exploitation of technologies and tools as Endusers

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Dissemination, communication and training

Isella Vicini | beWarrant CEO | Director of the European Funding Development Business Unit of Warrant Hub (Tinexta Group)

Dissemination, communication and training have the aim to implement full exploitation of project results during the life of the project though engagement with all stakeholder groups, to ensure that the project expected impacts are met during and after the project thought a strategic use of all CHALLENGES results.

CHALLENGES 1st Training School on real time nano characterization related technologies

21-22 September 2022

The School was held both remotely and in presence, organized by University of Sapienza and beWarrant (Tinexta Group).

Figure 10: CHALLENGES 1st Training School on real time nano characterization related technologies

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beWarrant
TINEXTA GROUP

CHALLENGES 2nd Training School on real time nano characterization related technologies

12-13 February 2024

The School was held both remotely and in presence, organized by University of Sapienza, CEA-Leti and beWarrant (Tinexta Group).

Figure 11: The CHALLENGES 2nd Training School on real time nano characterization related technologies
Video part 1 >>
Video part 2 >>

CHALLENGES Project Video

Figure 12: The CHALLENGES Project Video. Link to the project video >>

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CHALLENGES Project | ALTECH workshop at the e-MRS conference spring 2024 in Strasbourg

Figure 13: The CHALLENGES section is kicking off, within the ALTECH workshop at the e-MRS conference spring 2024 in Strasbourg.

CHALLENGES Project interviews

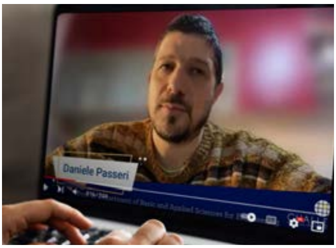

Figure 14: CHALLENGES Project | interview to Daniele Passeri (UNISAP).

[Link to the interview >>]

Figure 15: CHALLENGES Project | interview to Amaia Zurutuza and Alba Centeno (Graphenea)

[Link to the interview >>]

Figure 16: CHALLENGES Project | interview to Ivan Gordon (IMEC)

[Link to the interview >>]

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Partners

SAPIENZA UNIVERSITÀ DI ROMA

imec embracing a better life

Graphenea

life.augmented

IMM Institute for Microelectronics and Microsystems National Research Council of Italy

tiberlab

cea leti

NOVA PROCESS INSIGHT

NANONICS IMAGING Ltd.

ScanSens You can Scan You can Sens!

APPLIED MATERIALS

PTB Physikalisch-Technische Bundesanstalt Braunschweig und Berlin

beWarrant TINEXTA GROUP

SOL instruments

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