PolarNon

hardware and software imaging solution for materials characterization

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MULTIPHOTON MICROSCOPE

The development of new non-destructive characterization and diagnostic methods for materials investigation is one of the key aspects of the modern microscopy industry. Fast defects identification and inspection can significantly decrease the cost of the final devices and components.

The PolarNon is a microscopy solution combining a specialized hardware with a dedicated software, designed to address materials properties and quality. The PolarNon microscope is developed to probe nonlinear optical responses, such as second (SHG) and third (THG) harmonic generation. This system is composed of an infrared laser source, the core of the microscope, and a sensitive sCMOS imaging detector which can be combined with a spectrometer. The PolarNon is equipped with automated translation and rotation stages that enable fully autonomous measurements.

The non-destructive, contact free and surface sensitive nature of SHG microscopy, makes it a powerful method for studying a wide range of material properties. Combined with appropriate data analysis methods, the PolarNon enables to tackle various challenges of materials science and industrial applications for quality control, such as crystal structure analysis, corrosion study, defects identification and many more.

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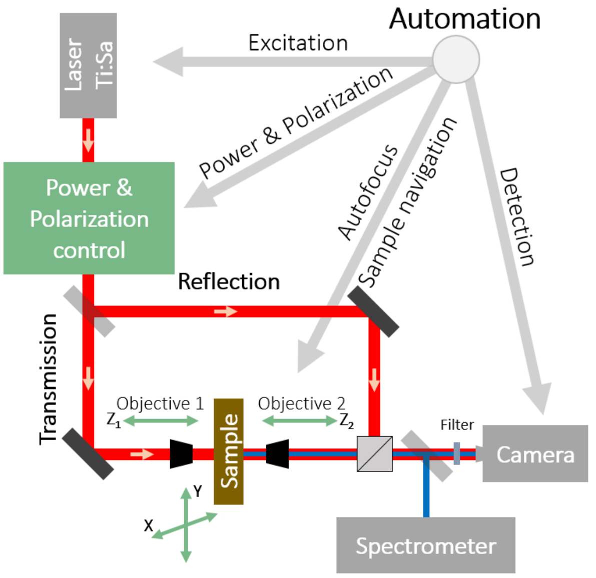
INSTRUMENT & FEATURES

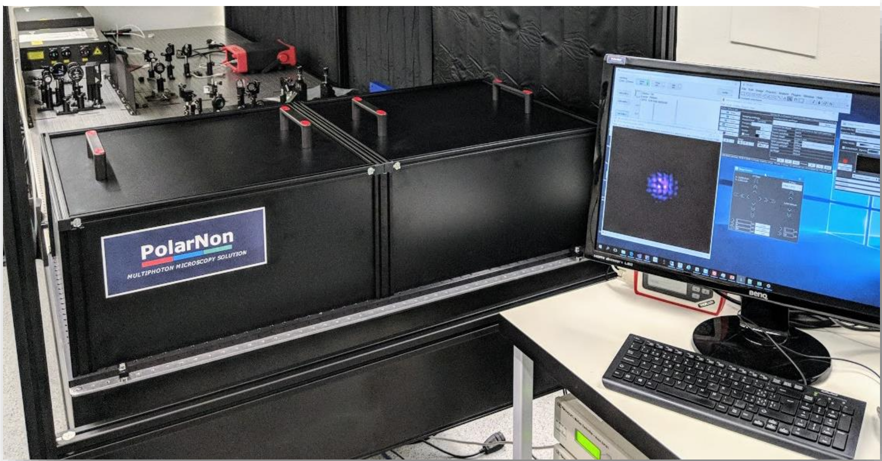
The PolarNon is designed to operate in both transmission and reflection configurations, depending on the special need of the user. It includes the following units: an excitation laser source, a power and polarization control unit, various excitation and collection objectives, a sample navigation system, a high-performance imaging detector and/or a spectrometer.



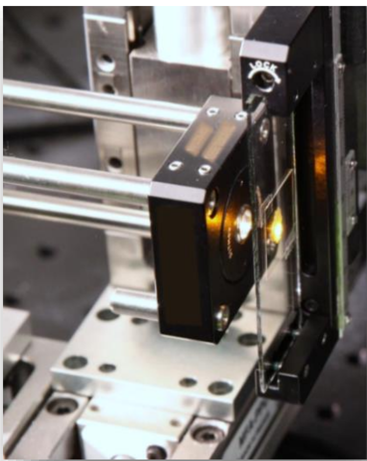
Features

- Spatially resolved SHG detection
- Submicron resolution and per-pixel data analysis
- LED/light or laser excitation
- Autofocusing module
- Domain-specific language for measurements automation
- Fully automated wavelength sweep (with autofocusing module)
- Remote control of measurements

PolarNon



SHG from nanostructures



GENERAL SPECIFICATIONS

Parameters	Range
Wavelength (nm)	from 700 to 1600
Objectives	10x, 20x, 50x and 100x
Operation	transmission or reflection
Detection	sCMOS camera, spectrometer
Power (mW)	0.1 – 1000 (at 800nm)
Polarization (deg)	0 – 360
Automation	Excitation (wavelength, power and polarization), sample translation, autofocus, detection

DETAILED SPECIFICATIONS TABLES

Excitation source

Typical av. Power* (mW)	1 – 200
Pulse Energy* (nJ)	0.0125 – 2.50
Peak Power* (kW)	0.09 – 180
Fluence** (J/m^2)	1 – 200

** Calculated for pulsed laser with repetition rate of 80MHz and pulse duration of 140fs*
*** Calculated for beam diameter of 4µm*

Detection (sCMOS or EMCCD)

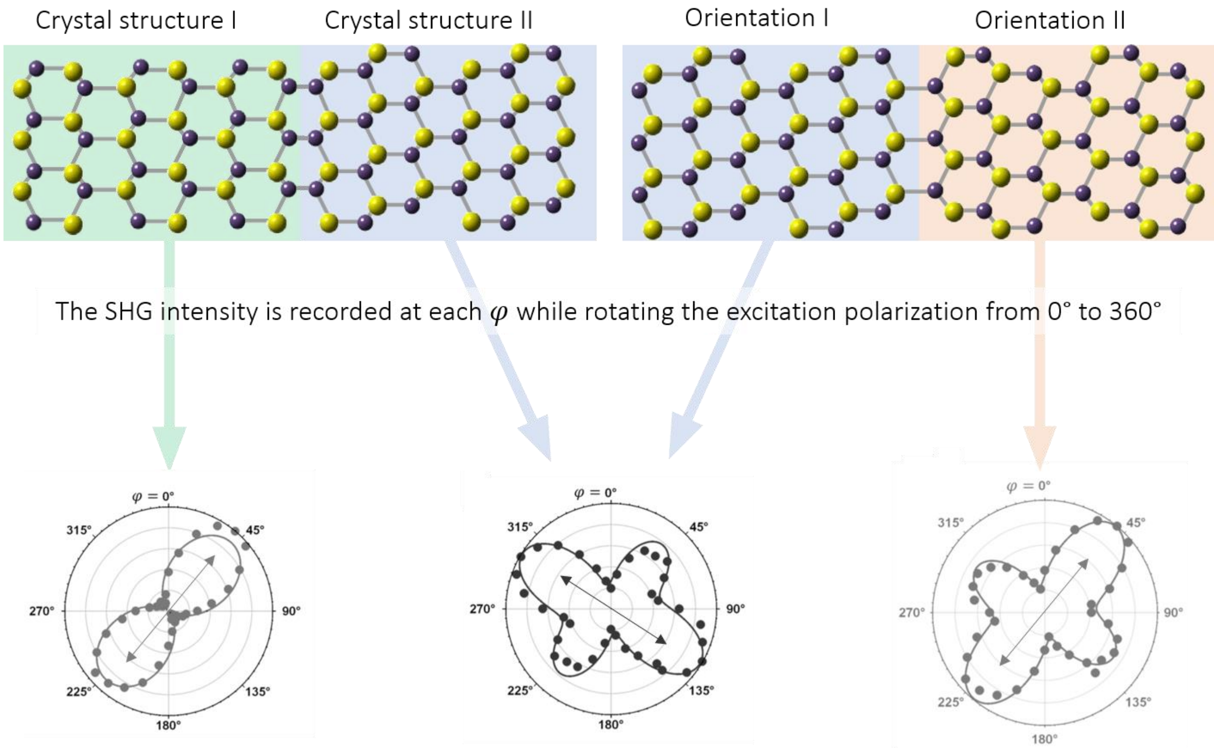
Active Pixels (WxH)	2048x2048 (4.2 MPIX)
Sensor Size (mm)	13.3x13.3
Max. Q. Efficiency (%)	82
Pixel Binning (nm)	700 to 1600
Dark Current (e$^-$/pix/sec)	0.1

Microscope system

Objectives	Typically used are 10x – 100x microscope objectives Can be adapted for any other types of customized objectives	
Sample Translation Stages	Travel Range (mm)	25
	Speed (mm/s)	2.5
	Minimum Incremental (nm)	100
	Typical Accuracy (nm)	± 20
	Drive Type	DC Motor
Polarization and Power (Rotation Stages)	Travel Range (deg)	360
	Max Speed (deg/s)	20
	Min Increment (mdeg)	20
	Typical Accuracy (nm)	± 20
	Drive Type	DC Servo

POLARIZATION-RESOLVED SECOND-HARMONIC GENERATION

The working principle of the PolarNon is based on the imaging and postprocessing analysis of the optical second-harmonic generation (SHG). The SHG signal is extremely sensitive to any break of symmetry in the crystal structure, which makes SHG microscopy an efficient tool for inspection of defects, cracks, crystal structure and orientation. Depending on the polarization state of the excitation beam, the SHG response will vary for regions with different types/orientations of the crystal structure. The figure below illustrates how different types and orientations of crystal structures respond to the polarization of the excitation light. During the measurements, the SHG intensity is recorded with the sCMOS or EMCCD camera.



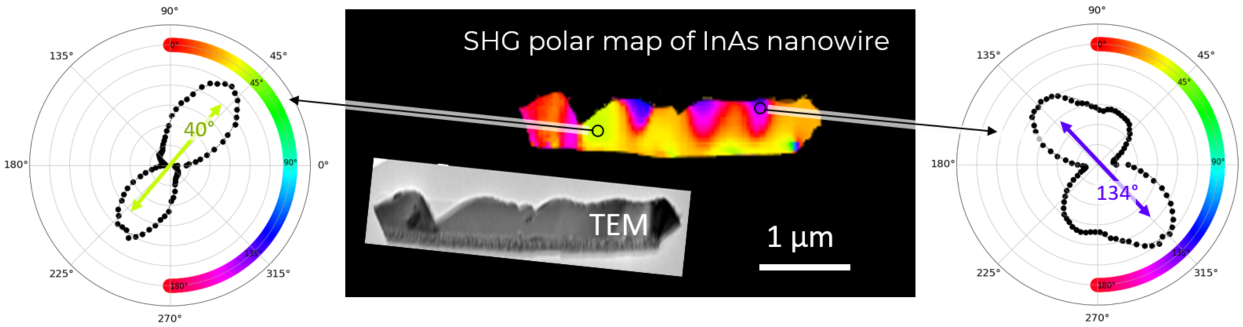
different shapes and orientation of SHG polar anisotropy pattern

flipped orientation of SHG polar anisotropy pattern

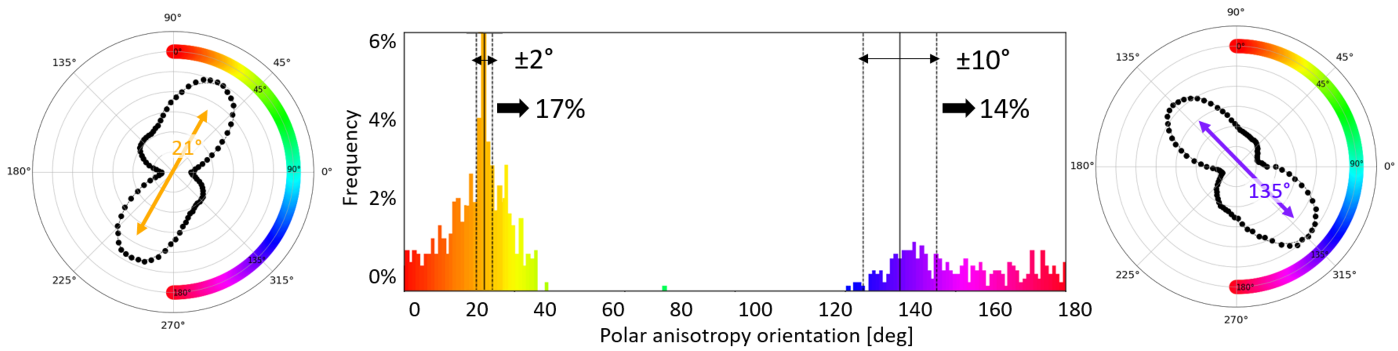
DATA PROCESSING AND PER-PIXEL ANALYSIS

To simplify the user experience, the PolarNon incorporates a suitable image analysis method to proceed raw SHG data. By processing the polarization-dependent SHG intensity I(φ), a resulting stack of polarimetric SHG images is precisely reconstructed. Each pixel contains information about the SHG polar anisotropy shape and orientation. This simple and rigorous visualization approach enables to identify critical correlations between the recorded signal and the material properties. As the sample is scanned by the focused incident laser beam, each pixel signal results from a nonlinear coupling at the overlap between the material and the diffraction-limited spot at this particular pixel position. This subdiffration pixel size accuracy allows to access a submicron sensitivity. Further analysis of the polarimetric images stack allows to extract a frequency histogram to quantify the different crystal phases and orientations present in the material.

The resulting SHG polar map provides information about the crystal phases and orientation. Each pixel contains a local polar anisotropy pattern. It's specific orientation, from 0° to 180°, is represented with the color scale.



The frequency histogram of the polar map represents the orientation distribution of the polar anisotropy patterns. It enables to quantify material phases according to the user's wishes.



The example shows that 17% of the InAs nanowire contains polar anisotropy pattern oriented with 21°±2°, and 14% with 135 °±10°.

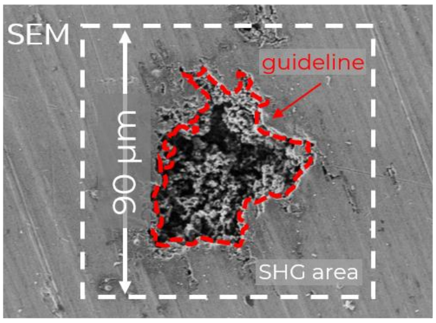
APPLICATION EXAMPLES

SHG microscopy is already well known, and frequently used in the biomedical field. The development of the method over the past decades has, however, made it possible to expand the possible application. Thanks its high sensitivity to surfaces and crystal structures, SHG microscopy has become a promising method for studying various types of materials. Some application examples are presented below.

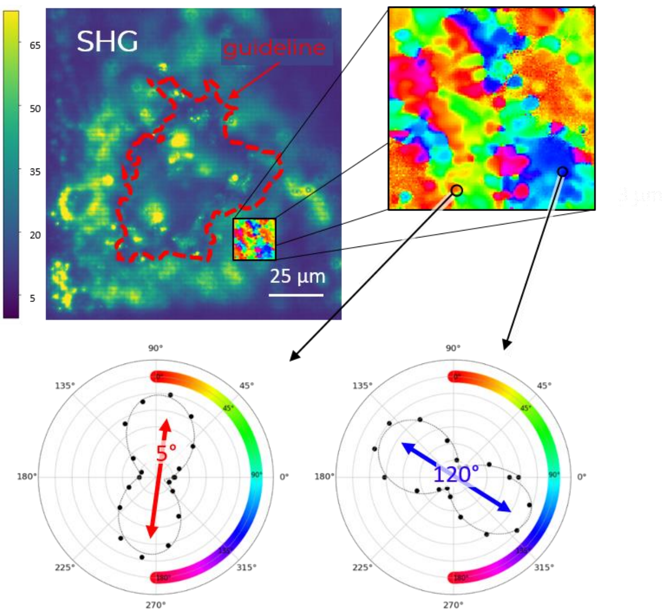
Investigation of corrosion in aluminum

The mechanisms of formation of anodic aluminum oxide layer as well as the composition of the resulting corrosion products are complex, and still await full understanding. Here, the PolarNon was successfully used to study the applicability of SHG microscopy to detect and study anodic corrosion in aluminum alloy. The SHG anisotropy measurements provide useful information about the corresponding oxide complex.

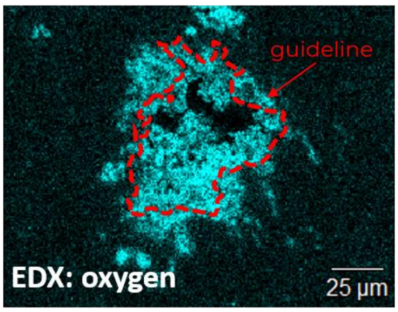
SEM image of a deep corrosion pit in aluminum alloy



SHG intensity and SHG polar anisotropy measured over the same corrosion pit



EDX showing the oxygen distribution in corresponding aluminum alloy

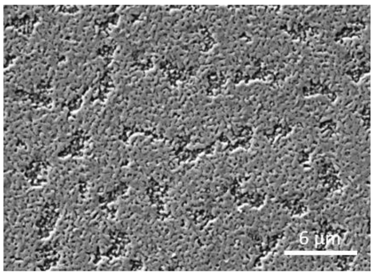


The SHG intensity map combined with the SHG polar anisotropy measurements provide information about the complexity of oxides, oxihydroxides and hydroxides arrangement

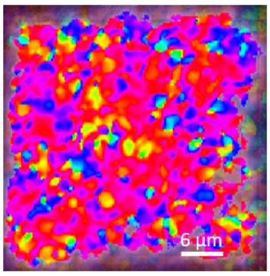
Crystal structure characterization of SiC wafers

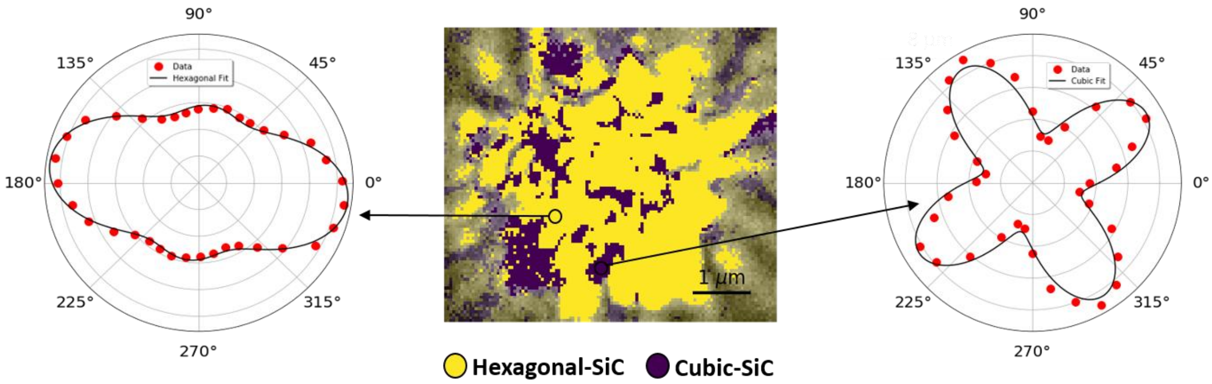
Silicon Carbide (SiC) has become a highly promising material for a wide range of electronic applications like high-temperature, high-frequency and high-power devices. Nevertheless, the high defect density and the difficulty to grow single polytypes limit its widespread use. In this application, the PolarNon was used to study SiC crystal structures, and to differentiate hexagonal and cubic polytypes thanks to its high sensitivity to materials symmetry.

SEM image of SiC grown with various defects and polytypes



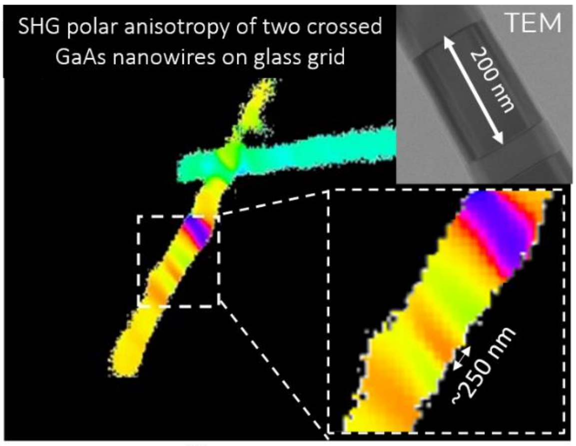
SHG polar anisotropy showing various polar plots orientation



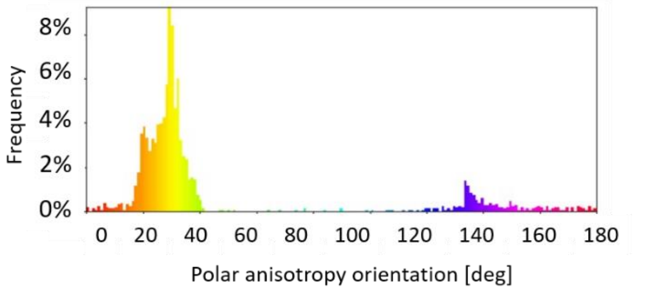


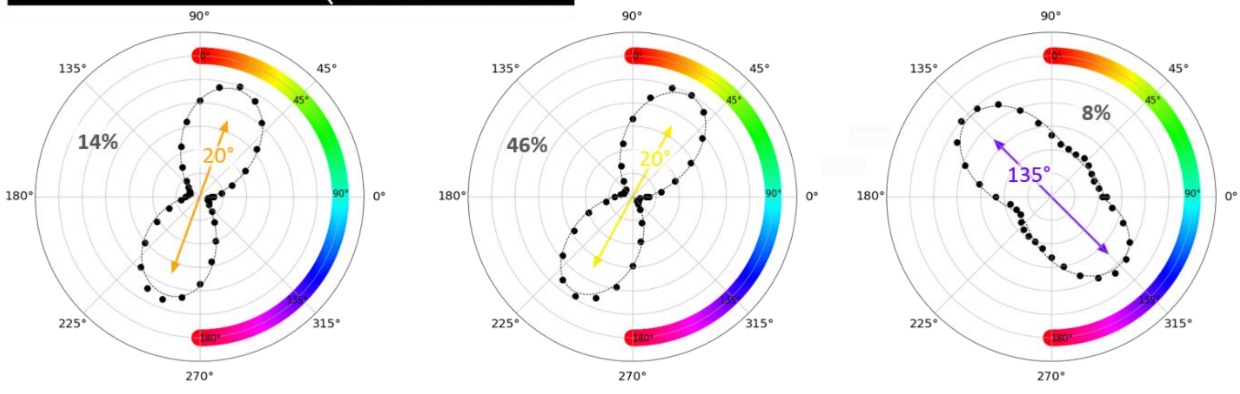
Crystal structure in III-V nanostructures

Semiconductor nanowires attract a growing research interest due to their unique optical and electronic properties. Their crystal structures, and orientation are highly dependent on the growth parameters. GaAs nanowires can exhibit wurtzite ($6mm$ symmetry) and zinc-blend ($\bar{4}3mm$ symmetry) crystal phases or a mixture of both. In addition to that, twinning can occur during the growth. This results in segments of the same crystal phase with different orientations along the growth axis. SHG microscopy has the advantage of being extremely sensitive to structural properties of the nanowires due to its dependency on the light polarization. Here, the PolarNon was used to detect the wurtzite and zinc-blend phases in GaAs nanowires and to distinguish twins.



The pixel-by-pixel polar anisotropy analysis of the zoomed area provides specific information about the crystal structures and orientation. Crystal phases for each region of interest can be revealed and quantified accurately.

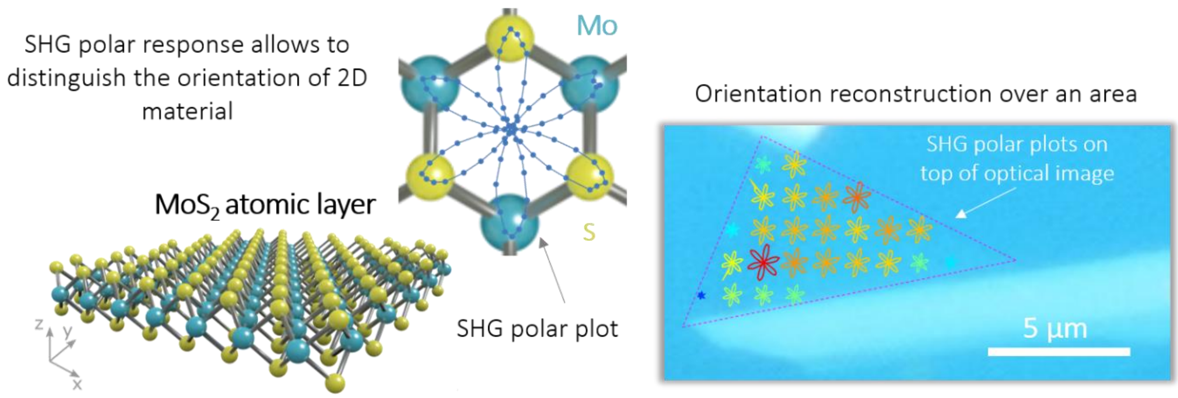


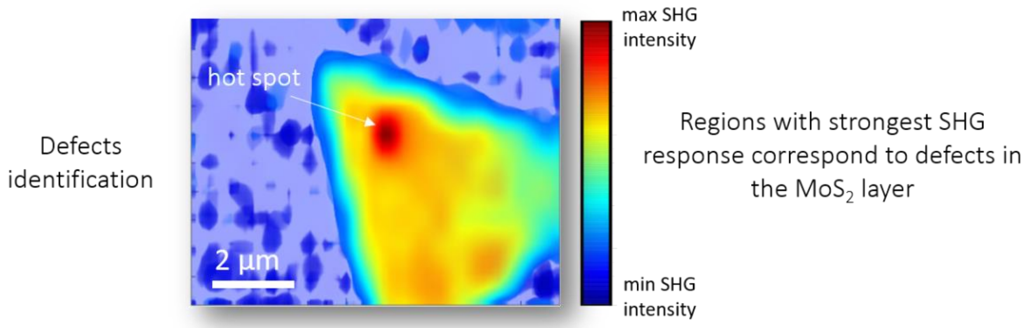


Twinned segments, and crystal phases can be identified based on their polar anisotropy signature. The histogram analysis shows that the NW is composed of 14% with polar orientation of 20°, 46% with 29°, and 8% with 135°.

2D materials characterization

The SHG polar anisotropy can be used as a sensitive tool for crystal structure orientation identification and defects inspection in transition metal dichalcogenide monolayers (TMDC), for example MoS_2, hBN, WS_2, etc. By varying the polarization of the excitation laser light simultaneously with the SHG polarization (Polarization-In, Polarization-Out), the positions of the atoms in the monolayer was determined.





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