

# INFRARED SPECTROSCOPY

Bertucci Isabel<sup>2</sup>, Cassisa Federica<sup>1</sup>, Delpin Pietro<sup>1</sup>, Diac Francesca<sup>3</sup>, Gregoris Francesco<sup>1</sup>, Innocente Andrea<sup>1</sup>, Nascig Fabio<sup>1</sup>, Vernole Stefano<sup>1</sup>

<sup>1</sup>4LSAB, <sup>2</sup>4LSAA, <sup>3</sup>4LSUB Polo Liceale A. Einstein ISIS della Bassa Friulana, Cervignano del Friuli, Italy

\*pages9@isisbassafriulana.edu.it

## Introduction

**Infrared (IR) spectroscopy** is an analytical technique used to identify and study chemical properties of compounds. It works by measuring the **absorption** of infrared radiation by a substance. When IR radiation hits a molecule, it can be absorbed if its energy matches the **vibration frequency** of a chemical bond. Each type of bond absorbs IR radiations at characteristic frequencies, so the resulting IR spectrum acts as a **molecular "fingerprint"**, useful to recognize functional groups and analyze the structure of pure substances or complex mixtures.

## Purpose

The aim of our work is to characterize chemical variations caused by UV treatment of an epoxy resin, named **SUEX**, by comparing the IR spectra of the resin before and after UV exposure.

SUEX is a light sensitive material (**photoresist**). Once SUEX is hit by UV light, it becomes **insoluble** to a developer solution. Using a specific protective mask, we can induce permanent modifications only on specific areas of SUEX foil and wash away the unmodified part. This technique is called **optical lithography**, and it is used to fabricate **micro-electronic circuits and microchips**.

## Procedure

The 20  $\mu\text{m}$  thick SUEX sample is laminated on a silicon substrate at 60°C.

During the lithography, a 200 nm thick chrome mask on quartz is overlapped on the sample which is then irradiated with UV radiation to the power of 21 mW/cm<sup>2</sup> for 8 s with a total energy of 168 mJ/cm<sup>2</sup>.

After that, we heat the resin to 85°C for 5 minutes and then we develop it with MR600 solvent for 10 minutes. Finally, we rinse it with isopropanol to eliminate chemical residues.

Then we proceed to analyze the UV-induced chemical alterations on SUEX using IR spectroscopy. First, we acquire an optical image of SUEX in near and medium infrared by reflection and then a chemical image by transmission in the spectral range of 0.74–300  $\mu\text{m}$ . The microscope used is equipped with an FPA detector (128×128 pixels), which allows high-resolution analysis over a wide area.

The silicon substrate, opaque to visible light but transparent to infrared, allows the correct signal transmission.

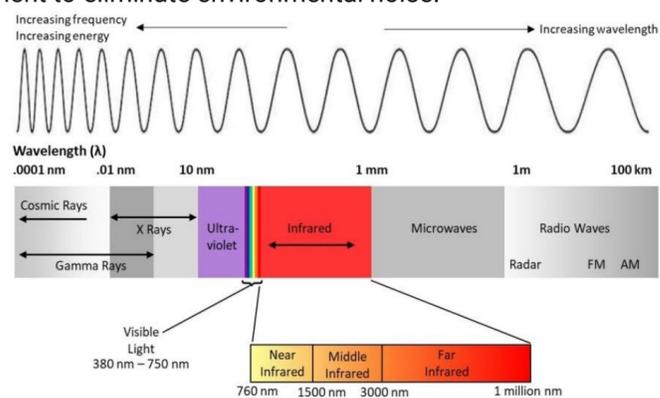
Background normalization is performed before of the measurement to eliminate environmental noise.



Chrome mask



IR microscope



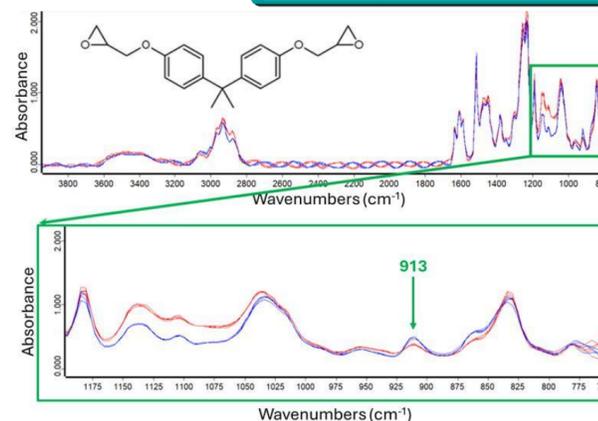
IR spectral range. Credits: www.azooptics.com

## Conclusions

IR spectroscopy is a useful tool to analyze chemical structure and chemical properties of matter. Here we applied IR spectroscopy to discover chemical alterations on an epoxy resin (SUEX) in an optical lithography setup. Our experiment shows how infrared spectroscopy, applied to optical lithography, allows the scientific community to obtain useful results with important practical applications.

At **SISSI-Bio beamline** of Elettra Sincrotrone TS, the IR s-SNOM Scanning Near-field Optical Microscopy has been used for the first time for studying complex cross-sectioned samples from the field of Cultural Heritage.

## Spectra Analysis



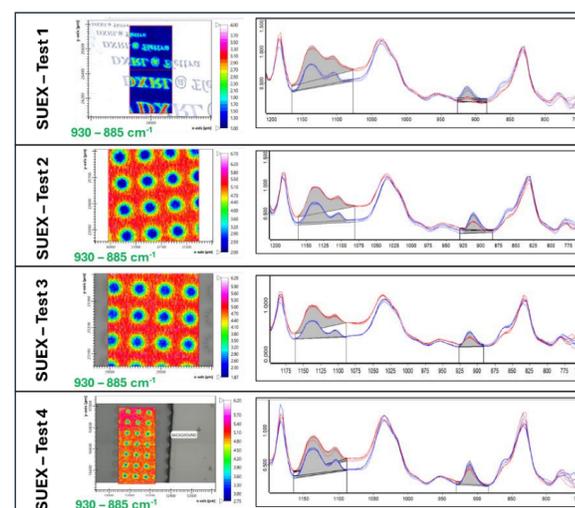
IR spectra. Credits: report CERIC PaGES9

From the previous analysis we obtain IR spectra of modified and unmodified SUEX. On Y-axis we have the absorbance and on the x-axis the wave number measured in cm<sup>-1</sup>. During the experiment, operations were conducted within the spectral range of 800 - 3800 cm<sup>-1</sup>.

We observe an increase of absorbance between 3000 cm<sup>-1</sup> and 2800 cm<sup>-1</sup>, which can correspond to the symmetric and asymmetric stretching vibrations of the C-H bonds, as highlighted in the figure below (values between 2975 and 2845 cm<sup>-1</sup>).

Another difference is evident at 913 cm<sup>-1</sup>, where we observe a drop of absorbance in UV-treated SUEX, which can be associated with the elongation of the C-O bond in the epoxy ring, suggesting a breaking of the bond due to lithographic treatment, thus explaining the lower absorbance. In addition, in the range 1160 - 1090 cm<sup>-1</sup>, a change was observed that could indicate the formation of a linear C - O - C bond.

IR spectra of SUEX can be elaborated to obtain chemical maps (shown below) where we can observe that the areas with lower IR absorbance, highlighted by the blue color, correspond to the mask unprotected regions, while areas with higher absorbance values, marked in red-orange, correspond to protected areas. Spectral variations can be analyzed by subtraction of subordinate areas (AUC Area Under Curve).



IR Chemical maps of SUEX. Credits: report CERIC PaGES9

# RAMAN SPECTROSCOPY

Bertucci Isabel<sup>2</sup>, Cassisa Federica<sup>1</sup>, Delpin Pietro<sup>1</sup>, Diac Francesca<sup>3</sup>, Gregoris Francesco<sup>1</sup>, Innocente Andrea<sup>1</sup>, Nascig Fabio<sup>1</sup>, Vernole Stefano<sup>1</sup>

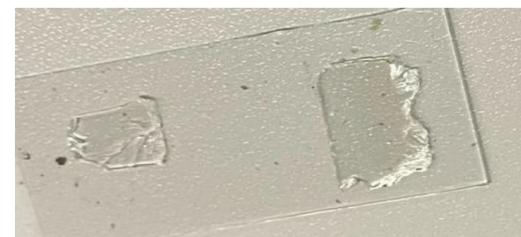
<sup>1</sup>4LSAB, <sup>2</sup>4LSAA, <sup>3</sup>4LSUB; Polo Liceale A. Einstein, ISIS della Bassa Friulana, Cervignano del Friuli, Italy

\*pages9@isisbassafriulana.edu.it

## Introduction

**Raman spectroscopy** is a **non-destructive** technique used to analyze the molecular and crystalline structure of materials in solid, liquid, or gaseous phases. The sample is irradiated with monochromatic laser light: the interaction of the light with the molecules generates **light scattering**, which can be either elastic (**Rayleigh effect**) or inelastic (**Stokes and anti-Stokes scattering**). By analyzing the inelastic components, information about molecular vibrations is obtained, which is useful to characterize materials and studying their properties.

The aim of our work is to use Raman spectroscopy to characterize chemical variations caused by UV treatment of an **epoxy resin of negative resist type (SUEx)**. A negative resist cross-links upon exposure to radiation and becomes insoluble to specific solvent. Raman spectroscopy allows us to identify structural alterations of SUEx molecules.



SUEx sample. Credits: A. Innocente

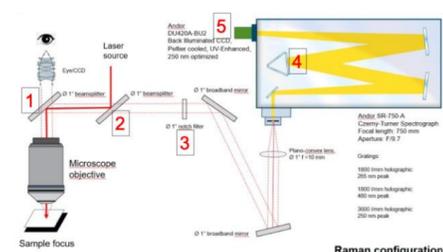
## Purpose and material

## Procedure

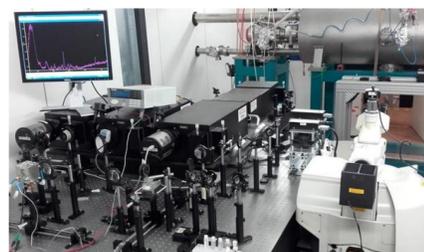
The examined resin film has a **thickness of 500 μm**. A transparent **quartz** and **chrome** mask with a thickness of **100 nm** is applied over it to protect the resin from radiation only in selected areas. The sample is then irradiated with a UV-lamp operating at 60% of its nominal power, corresponding to an **intensity of 35 mW/cm<sup>2</sup>**. However, considering the real measurement conditions, the **power per unit area** effectively incident on the sample is **21 mW/cm<sup>2</sup>**. The **exposure time** to the radiation is **55 seconds**, during which the sample is subjected to a **total energy dose of 1155 mJ/cm<sup>2</sup>**. Then we obtain a Raman spectrum of the sample using a **monochromatic light source**, which generates a light composed of a single wavelength. In our experiment, we used a **green** laser source corresponding to a wavelength of **532 nm**.



Resin with quartz and chrome mask. Credits: A. Innocente 2025



Raman configuration. Credits: F.D'Amico 2024



Raman spectrometer. Credits: F.D'Amico, et al., NIMA 2013

With reference to the picture above, the Raman spectrometer configuration is:

**experimental Beamsplitter** (1 and 2): These devices split the laser beam, reflecting 50% of the incident light and transmitting the other half.

**Notch Filter** (3): This filter selectively blocks radiation at the laser wavelength (532 nm in our case), eliminating the Rayleigh component and allowing only the Raman scattered light to pass through.

**Reflection Grating** (4): It works as a dispersive element, separating the scattered light based on its wavelength.

**CCD Detector** (5): It captures the light scattered by the grating and produces the Raman spectrum, which displays the various vibrational transitions of the analyzed sample.

## Conclusions

With this experimental procedure we determine chemical alterations on specific materials. Data analysis evidenced structural modifications on the aromatic rings of the SUEx molecule. To reach this conclusion we had to adjust Raman spectra with mathematical corrections. This result highlights the typical trade-off in theoretical-experimental fitting: an effective correction in one region can amplify discrepancies in others.



**PRP@CERIC\* Inelastic Ultraviolet Scattering with fixed-energy sources (IUVS-Offline)**

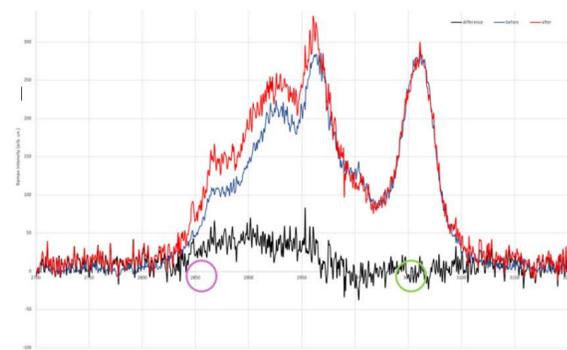
UV Resonance Raman (UVR) spectroscopy is a very valuable tool for collecting chemically specific information about a large variety of systems thanks to the simultaneous coupling of electromagnetic radiation with the electronic charge localized in particular chromophoric groups of molecules.

Photo credits: CERIC-ERIC

## Analysis

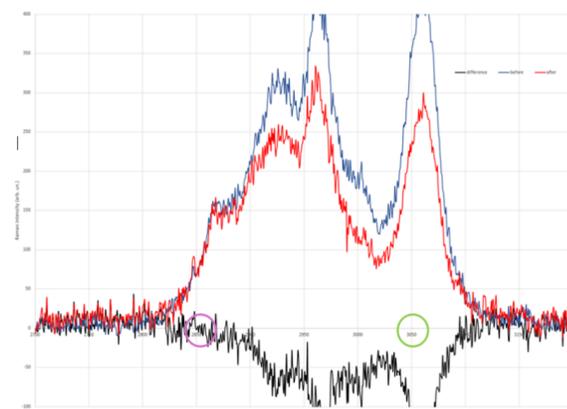
After the measurement, we proceed to compare before (red) and after (blue) Raman spectra shown below (A). By computational analysis using Avogadro software, it is possible to hypothesize that the peak furthest to the right corresponds to vibrations of the aromatic rings of the molecule. However, to maximize the difference between the two spectra on that range (3056 cm<sup>-1</sup>), we applied a correction to nullify the difference in a specific region (2850 cm<sup>-1</sup>) and, as we can observe in fig B, we obtain a more pronounced difference at the aforementioned frequency.

A



	before	after	difference	x	before	after	before plotted	after plotted	difference
A0	0	0	0	2850.691	44,78942	69,58807	47,028891	69,58807	22,559179
A1	1.05	1	1	3056.082	255,3291	265,7097	268,095025	265,70966	-2,385425

B



	before	after	difference	x	before	after	before plotted	after plotted	difference
A0	0	0	0	2850.691	44,78942	69,58807	69,243601	69,58807	0,164469
A1	1.05	1	1	3056.082	255,3291	265,7097	395,760275	265,70966	-130,05037

RAMAN spectra Credits: report CERIC PaGES9 by F. Gregoris

## Labs at CERIC

## SAXS AND WAXS

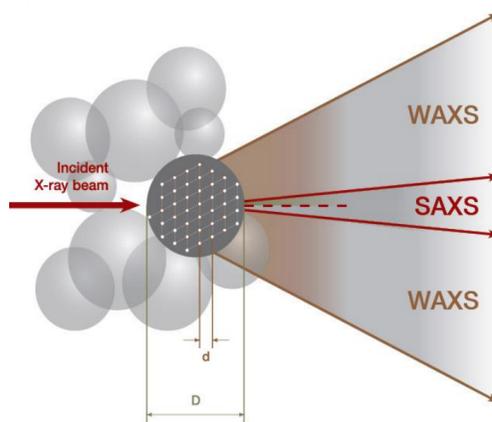
Bertucci Isabel<sup>2</sup>, Cassisa Federica<sup>1</sup>, Delpin Pietro<sup>1</sup>, Diac Francesca<sup>3</sup>, Gregoris Francesco<sup>1</sup>, Innocente Andrea<sup>1</sup>, Nascig Fabio<sup>1</sup>, Vernole Stefano<sup>1</sup>

<sup>1</sup>4LSAB, <sup>2</sup>4LSAA, <sup>3</sup>4LSUB; Polo Liceale A. Einstein, ISIS della Bassa Friulana, Cervignano del Friuli, Italy

[\\*pages9@isisbassafriulana.edu.it](mailto:pages9@isisbassafriulana.edu.it)

### SAXS

**SAXS Small Angle X-Ray Scattering** is a technique based on the scattering of X-rays at small angles ( $0.1^\circ$ – $5^\circ$ ) used to analyze structures at the nanometer scale (1–100 nm) of any type of material. It provides information on the size, shape, and distribution of particles or phases and it is ideal for studying dynamic processes (e.g., chemical transformations, changes in temperature and pressure). It requires careful sample preparation, the use of vacuum tubes, beam collimation, and calibration with a reference material.



Credits: wiki.anton-paar.com

### WAXS

**WAXS Wide-Angle X-Ray Scattering** technique is based on X-ray scattering at wide angles (greater than  $5^\circ$ ), used to study atomic structure and crystallinity.

It reveals phase composition and atomic arrangement through unique diffraction patterns. It is suitable for monitoring structural changes under stress or temperature. It requires proper sample preparation, vacuum setup, and calibration with a standard material.

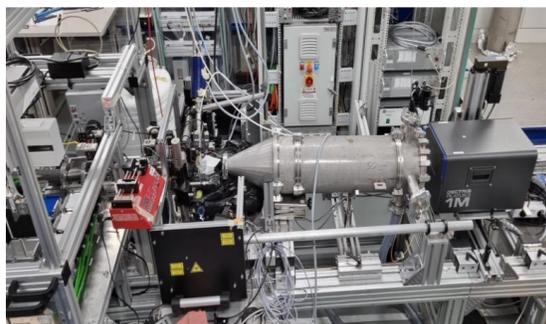
### Experimental SAXS/WAXS Setup

From an experimental standpoint, to correctly perform a SAXS measurement it is required to follow a precise operational sequence. Procedurally, the main components of the experimental setup must be checked in the following order:

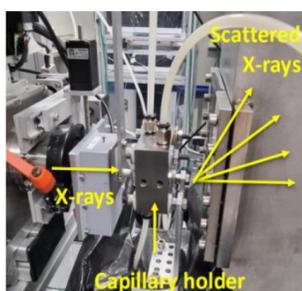
1. the sample must be properly prepared and positioned in the designated location within the experimental apparatus;
2. a slit system is used to adjust the size of the beam, regulating the shape and intensity of the incident X-ray beam;
3. vacuum tubes are employed to prevent air molecules from contributing to X-ray scattering, which would lower the signal quality;
4. a detector collects the scattered X-rays, generating the scattering signal.

Before analyzing the actual sample, it is necessary to use a calibrant: a known reference material, to calibrate the instrument.

This step ensures the accuracy and reproducibility of the acquired data.

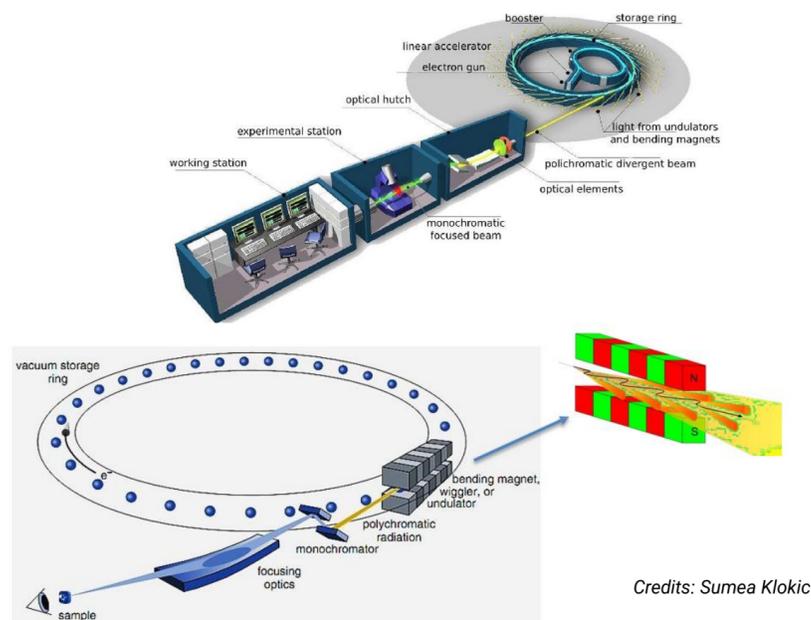


SAXS Experimental setup  
Credits: Sumea Klokic



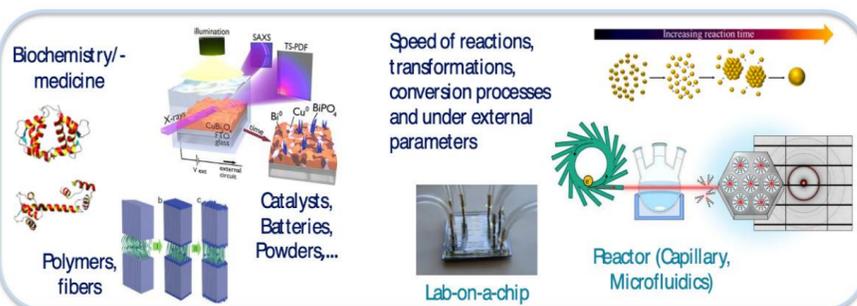
see also [Small Angle X-ray Scattering \(SAXS\) - Ceric](#)

### How X-rays are generated



Credits: Sumea Klokic

X-Rays synchrotron radiation are generated at the synchrotron ELETTRA by accelerating electrically charged particles (electrons) at near-light speed and by deviating their trajectory using bending magnets.



Credits: Sumea Klokic

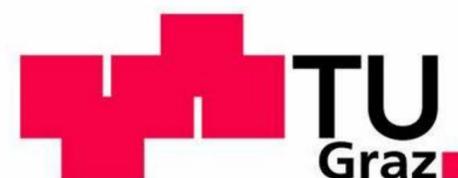
### What for

### Labs at CERIC

The highflux **SAXS beamline** of TU University of Technology of Graz, located at Elettra Sincrotrone Trieste, was mainly intended for time-resolved studies on fast structural transitions in the sub-millisecond time region in solutions and partly ordered systems with a SAXS-resolution of at least 100 nm in real-space.

However, increasingly also grazing-incidence (GISAXS) measurements are performed to study self-assembly processes on surfaces or to perform structural characterizations of thin films.

Additionally, SAXS measurements can be coupled to simultaneous Differential Scanning Calorimetry (DSC) and Wide Angle X-ray Scattering (WAXS) recordings.



## PaGES<sup>9</sup>

### Planning, Management and Implementation of a scientific experiment in an international research infrastructure

Bertucci Isabel<sup>2</sup>, Cassisa Federica<sup>1</sup>, Delpin Pietro<sup>1</sup>, Diac Francesca<sup>3</sup>, Gregoris Francesco<sup>1</sup>, Innocente Andrea<sup>1</sup>, Nascig Fabio<sup>1</sup>, Vernole Stefano<sup>1</sup>

<sup>1</sup> 4LSAB, <sup>2</sup> 4LSAA, <sup>3</sup> 4LSUB; Polo Liceale A. Einstein, ISIS della Bassa Friulana, Cervignano del Friuli, Italy

[\\*pages9@isisbassafriulana.edu.it](mailto:pages9@isisbassafriulana.edu.it)

#### The project

The PaGES project gives high school students the opportunity to have the experience of an innovative educational context. By carrying out all the steps of a scientific experiment, students will acquire the basic tools for planning, management, execution, evaluation and dissemination of the results of a research project. They also will learn the basic elements to develop their entrepreneurship. Through the PaGES project, pupils from different scientific high schools of the Region Friuli Venezia Giulia, Italy, have had the chance to gain the know-how and direct experience of an international research environment and have actively participated in the process of shaping a research idea into a factual experiment, through to the communication of its results and the presentation of the projects' outcomes to the community to which they belong.

#### Network of partners

**CERIC**

Central European  
Research Infrastructure  
Consortium



BUONARROTI



Innovare  
attraverso  
la Scienza,  
Costruire  
insieme

Scopriamo  
per Tutti

Agire sul  
piccolo  
pensando  
in grande

Rigore  
Tenacia  
Passione  
Curiosità

#### Thanks to

Marina Coricciati  
**Project Manager & operational support**  
Andrea Santelli  
**Chief Administrative Officer**  
Nicoletta Carboni  
**Press & Communications**

We wish to thank all the people that made PAGES9 possible:

CERIC-ERIC staff and the researcher team  
Elettra Sincrotrone Basovizza TS  
School partners of Friuli Venezia Giulia



REGIONE AUTONOMA  
FRIULI VENEZIA GIULIA

**Researchers team**  
Benedetta Marmioli  
Chiaramaria Stani  
Francesco D'Amico  
Sumea Klokic

Progetto finanziato con Bando Regionale delibera 564 del 22 aprile 2022

Dietro ad ogni problema,  
c'è un'opportunità  
Dedicato a Emanuele (1998-2018)