Applications of SR X-ray imaging to the investigation of historical samples

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Eletta - Sincrotrone Trieste
Who we are

No-profit shareholder national interest company. Shareholders: Area Science Park, Friuli Venezia Giulia Region, CNR, Invitalia. Established 28 years ago to build and manage synchrotron light sources to be open internationally.

The mission is to promote cultural, social and economic growth through basic and applied research in relevant fields, technical and scientific training, and technology transfer.
Elettra at a glance

- 400 employees
- 100,000 m²
- 5,000 hours /year
- 32 beamlines
- more than 1,000 Users
- from more than 50 countries
Partnerships & Collaborations

Elettra is part of:

- Multi-sector Technology
- 62 tenants
- 21 Research Centers

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Elettra is part of:

- General Confederation of Italian Industry
- 150000 Company
- More than 5 Million of employees

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Elettra is associated with:

International Atomic Energy Agency
A machine that generates brilliant beams of light by moving electrons through a strong magnetic field.

Radiation from Electrons in a Synchrotron

F. R. Elder, A. M. Gurewitsch, R. V. Langmuir, and H. C. Pollock
Research Laboratory, General Electric Company, Schenectady, New York
May 7, 1947

HIGH energy electrons which are subjected to accelerations normal to their velocity should emit electromagnetic energy. The radiation from electrons in a betatron or synchrotron should be emitted in a cone tangent to the electron orbit, and its spectrum should extend into the visible region. This radiation has now been observed visually in the General Electric 70-Mev synchrotron. This machine has an electron orbit radius of 3.0 cm and a peak magnetic field of 8100 gausses. The radiation is seen as a small spot of brilliant white light by the observer looking into the vacuum tube tangent to the orbit.
What does it look like inside?
The experimental station

- Storage Ring Tunnel
- Optics hutch
- Experimental hutch
- Control cabin
How does it work?
How does it work?

Electrons are generated here

And initially accelerated in the LINAC
How does it work?

Then they pass into the booster ring where they are accelerated to 99.9999% of $c$. 
How does it work?

And are finally transferred into the storage ring.
**How does it work?**

**Bending magnet**
At each deflection of the electron path a beam of radiation is produced.

**Insertion devices - produce higher intensity**

**Undulator**
Produces a very narrow beam of coherent light, amplified by up to $10^4$.

**Wiggler**
Beams emitted at each pole reinforce each other and appear as a broad beam of incoherent light.
The Techniques

- Photoelectron emission:
  - XPS
  - UPS
  - ARPES
  - XPD
  - TR

- Imaging:
  - IR microscopy
  - X-ray microscopy
  - X-ray tomography
  - Photoelectron Mic.
  - Fluorescent Img.

- Scattering:
  - Elastic
  - Inelastic
  - Magnetic
  - SAXS/WAXS

- Reflection/Emission:
  - X-ray fluorescence
  - Reflectometry
  - Reflectivity
  - NEXAFS
  - EXAFS
  - XMCD

- Absorption:
  - Infrared
  - TimeResolved

- Diffraction:
  - Crystallography
  - Powder Diffraction
  - Surface Diffraction
  - TimeResolved
Support Laboratories

CITIUS
The new Interreg project for the development of a state-of-the-art light source generating ultrashort pulses in the UV and soft X-ray spectral range. Read more...

Micro and Nano Carbon Lab
The main activity of the Micro and Nano Carbon Laboratory is the preparation and study of carbon nanotubes and several carbon based materials. Read more...

Organic OptoElectronics
The lab investigates the properties of organic semiconductors, either molecular or polymeric, and their applications. Read more...

Scientific Computing
The scientific computing team supports research activities by providing advanced algorithms, ICT services and infrastructures. Read more...

Support Lab
The Support Lab operates a machine workshop and a chemical laboratory supporting Elettra beamlines and users Read more...

Surface Science
The laboratory research activity addresses the geometrical and electronic structure as well as the chemical reactivity of a large variety of solid surfaces. Read more...

Theory@Elettra
Theory@Elettra is the theory group funded by the CNR-INFN DEMOCRITOS supporting the experimental activity performed in the laboratory Read more...

Tomolab
The TomoLab station at Elettra provides a state-of-the-art X-ray computed microtomography system based on a microfocus source. Read more...

T-ReX
The T-ReX Lab hosts a set of facilities devoted to the study of ultra-fast processes in condensed and soft matter and their applications in technology. Read more...

Structural Biology
Structural and functional studies of proteins and protein complexes involved in DNA replication and repair, autophagy and genome stability. Read more...

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Powder Diffraction
The Powder Diffraction Laboratory is a support laboratory for MCX, XRD and XAFS beamlines, providing diffraction measurements, in Bragg-Brentano geometry, of powders, thin films, and single crystals.
Our users

Proposals submitted to Elettra in 2012 divided by country
FERMI now in operation performing its first experiments. It integrates Elettra performances in the femtoseconds range.
Our guidelines

✓ Direct access to an extensive range of facilities and techniques
✓ Feasibility Study is “free of charge”
✓ Quotations based on time, cost and performance
✓ Activities structured as a Project
✓ Continuous collaboration, sharing of results, knowledge transfer
Synchrotron light

- Tunable: possibility to select the beam wavelength
- Intense: possibility to obtain extremely fast acquisitions
- Coherent / collimated: similar to a laser beam
Ceterum censeo, munera non delendae esse

- Samples of great historical and/or commercial value
- Monitoring of restoration and conservation protocols
Artis monumentorum qui nunc vidit nullo vidit, qui mille vidit nunc vidit

- Use of several experimental techniques
- Examination of a high number of similar samples
What do we offer?

• Large portfolio of techniques
• Most techniques are non destructive or microdestructive
• Synergies between conventional labs and large research infrastructures
• Easy access to thematic networks and fundings
X-ray imaging at a 3\textsuperscript{rd} generation SR facility

- high energy photons and high flux
  \(\rightarrow\) heavy and/or bulky samples in transmission geometry
  \(\rightarrow\) tunability in a large energy range
  \(\rightarrow\) short exposure times

- small angular source size and big source-to-sample distance
  \(\rightarrow\) use of natural coherence properties of the beam

The SYRMEP beamline:

- Source size \(\sigma (h \times v) \approx 1100 \ \mu m \times 100 \ \mu m\)
- Source-to-sample distance: \(D \approx 24 \ m\)
- Beam size at sample \((h \times v) \approx 150 \ mm \times 6 \ mm\)
- Energy range: \(8 \div 35 \ keV\), Bandwidth: \(\Delta \lambda/\lambda \approx 2 \times 10^{-3}\)
SR X-ray imaging studies in archaeometry

In-situ and ex-situ experiments in a large range of applications:
  → archeaological findings and ancient artifacts identification
  → restauration techniques
  → conservation techniques …. 

The aim

to investigate the relationship between microstructural and physical properties
Absorption and Phase Sensitive (PS) Radiography

\[
\frac{\Delta I}{I}_{\text{abs}} = e^{c \Delta \mu} - 1
\]

\[
\Delta \phi = 2\pi c \Delta \delta / \lambda
\]

- \( r \ll a \Rightarrow \text{edge detection regime} \)
- \( r \approx a \Rightarrow \text{holographic regime} \)
- \( r \gg a \Rightarrow \text{Fraunhofer diffraction} \)

\[
n = 1 - \delta - i\beta \text{ : refraction index}
\]

\[
\mu = 4\pi \beta / \lambda \text{ : linear absorption coeff.}
\]

\[
c \text{ : object size // to beam direction}
\]

\[
a \text{ : object size \perp to beam direction}
\]

\[
r = (\lambda d)^{1/2} \text{ : first Fresnel zone radius}
\]
Absorption and PS Computed $\mu$-Tomography ($\mu$-CT)

Fundamental for investigation of internal features without sample sectioning:
- in many cases the sectioning procedure modifies the structures under analysis
- the sample can be studied by other experimental techniques, or
- submitted to several treatments (chemical, physical, etc...)
Conservation materials for stained glass windows - assessment of treatments, studies on reversibility, and performance of innovative restoration strategies and products

EU-Project CONSTGLASS N° 044339

Phase-contrast microtomography

Elettra Sincrotrone Trieste
Chartres - Window 37
La Passion typologique
Sample 2: glass fragment with grisaille decoration and Viacryl layer on the internal face. Evident alterations on the surface sides.
Fragments of lead or grisaille
Compact and homogeneous glass
Porous and fragmented material
Viacryl
Viacryl flakes from Bourges
(window 9, panel 4)
Viacryl flakes from Bourges (window 9, panel 4). It is evident that the Viacryl removes some original material from the glass panel.
Original glass (beginning of 20th century) bonded with epoxide resin (Epidian 53) about 30 years ago from interior, without glass dismantling. Possible phenomena: thick layer of resin on the glass surface and slight penetration of resin into the break. Break itself is soiled by external pollution. Bonding rather weak.
The same glass with epoxide resin on internal surface (on the left side of sample the resin only. The fresh break (center of glass) bonded with the same resin on 5th of August 2008).
Modern broken glass bonded (with the same resin as sample 2) on August 5\textsuperscript{th}, 2008.
Sample CAN 1a & 1b

Canterbury Cathedral.
Unknown origin.

Fragment of medieval green tinted glass with slight surface corrosion.

A mixture of microcrystalline wax (90%) and polythene A wax (10%) melted together and diluted with white spirit as painted onto the glass. Paraloid B72 was then applied.

Dummy test sample using XII/XIII century glass to replicate the condition of the glass surface and the methods used on the original glass during the 1970’s conservation treatment, although the wax has been applied in several layers to obtain thickness required for the tomo analysis.
The wax was applied in 3 separate layers and left for three days. The Paraloid B72 was mixed together with raw umber pure powder pigment and this was applied with a small brush on the wax.
No traces of net interface between the different wax layers and between wax and Paraloid. The air bubbles in the Paraloid are due to the evaporation of the solvant.
The wax was mixed together with raw umber pure powder pigment and applied in 3 separate layers and left for three days. The Paraloid B72 was applied with a small brush on the wax.
Sample CAN 1b

No traces of net interface between the different wax layers and between wax and Paraloid. The air bubbles in the Paraloid are due to the evaporation of the solvant. The wax does not fill irregularities in the glass surface.
The organ by Lorenzo da Pavia

Organ by Lorenzo Gusnasco (1494)

Pipes made with rolled and glued carton

Structural characterization of the paper pipes to define strategies for restoration, conservation and possible substitution

Instrument of great historical and artistic relevance
The organ by Lorenzo da Pavia

Correspondence between Lorenzo Gusnasco and Isabella d’Este
The organ by Lorenzo da Pavia

Slice at the wood foot position
The organ by Lorenzo da Pavia

- Ten 0.25 mm layers
- Good quality of the external layer
- Evident degradation of the layers adhesion
The organ by Lorenzo da Pavia

• Possibility of wood species characterization
• Presence of larvae
Computed tomography (CT) is a unique tool for characterization of bowed stringed instruments. Sirr and Waddle are the authors of the first works where clinical CT has been applied to the study of violins. Internal damage or repair invisible at visual inspection were detected in historical instruments.

S.A. Sirr, J.R. Waddle, Radiology, 1997, 203, 801
The main limitation in the application of clinical CT to the structural analysis of bowed instruments, however, is the **limited spatial resolution** of commercial instruments, (0.4x0.6x0.6 mm$^3$). Every defect with lateral dimensions smaller than this value **cannot be detected** with state-of-the-art hospital instruments.

S.A. Sirr, J.R. Waddle, Radiology, 1997, **203**, 801
The main limitation in the use of synchrotron radiation is related to the reduced dimensions of the samples under investigation. The development of new X-ray detectors designed for the particular characteristics allows the researchers to overcome this kind of problems.
PICASSO (Phase Imaging for Clinical Application with Silicon detector and Synchrotron radiation) has been developed by the Istituto Nazionale di Fisica Nucleare (INFN). It is a silicon microstrip detector in “edge-on” configuration.

The aperture of each pixel is determined by the strip pitch (0.05 mm, H) and the sensor thickness (0.3 mm, V). The detector is operated in single-photon counting and it is read out by a high-rate electronics based on the Mythen-II application-specific integrated circuit (ASIC). Each pixel is wire-bonded to one channel of the circuit and its signal is processed individually throughout the read-out electronics. The single-photon counting approach allows to maximize the contrast resolution (preserving the quantum nature of the information carried by the photon beam) and to overcome the limitations in the dynamic range, which are typical of CCDs and flat panels.
Transaxial μCT scan of a student violin taken at SYRMEP
(E = 23 keV, acquisition time = 1s, 3600 images)
Transaxial CT taken with a state-of-the-art clinical instrument (Toshiba Aquilion, helical scan 120 kVp, 512x512 matrix, 0.5 mm slice thickness, 0.5 s exposure time, 0.485/0.485 pixel spacing, Torax protocol)
Detail showing the bass bar and the glue used to attach it to the front plate.
Peter Herresthal and his Giovanni Battista Guadagnini (1753)
The experimental hutch
The planar image

Original notch of sound hole

Present notch of sound hole
- The original top plate is very thin, especially on the left side;
- The grain of the patch below the top plate is not straight (probably obtained by cutting and not by splitting);
- Warped shape of left rib due to the chin rest position.
- Thickness of top and back plate
- Bass bar is glued on a patch;
- Two patches on the right part of top plate.
- Two patches are glued on the top plate;
- The grain of the patches is good;
- Thickness of the back is very thin (mm)
In principle, it is impossible to reconstruct, with the usual experimental and mathematical tools, an object with lateral dimensions smaller than the FOV of the detector. This is the case of most musical instruments. We overcame this limit with local area tomography techniques, with a continuous scan where every image corresponds to an angular range and not to a single position.

This approach allowed us to analyze a violin at level of cellular structure, visualizing in detail the external varnish layer. In an absolutely non-invasive way.

Virtual section of the front plate obtained with local area tomography
The Divje babe flute, Mousterian, about 50000 years old, Slovenia

In collaboration with National Museum of Slovenia, Ljubljana
These evidences cannot conclusively prove a Neanderthal origin for this sample but show that the carnivores did not produce many of the features on the “flute”.
The Lonche mandible

Upper Pleistocene, about 6500 years old, Slovenia

Left canine shows presence of beeswax inside a vertical crack

Earliest known evidence of therapeutic dental filling

In collaboration with the Natural History Museum of Trieste