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

Franco Zanini
Eletta - Sincrotrone Trieste

Elettra Sincrotrone Trieste

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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
- 100000 m^2
- 5000 hours /year
- 32 beamlines
- more than 1000 Users
- from more than 50 countries

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Partnerships & Collaborations

Elettra is part of AREA SciencePark

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

Elettra is part of CONFINDUSTRIA

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

Elettra is associated with: IAEA.org International Atomic Energy Agency

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What is a synchrotron?

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 t accelerations normal to their velocity should electromagnetic energy.[1–4] The radiation from el in a betatron or synchrotron should be emitted in a cone tangent to the electron orbit, and its spectrum extend into the visible region. This radiation has no observed visually in the General Electric 70-Mev sy tron.[5] This machine has an electron orbit radius cm and a peak magnetic field of 8100 gausses. The ra is seen as a small spot of brilliant white light by server looking into the vacuum tube tangent to the orbit

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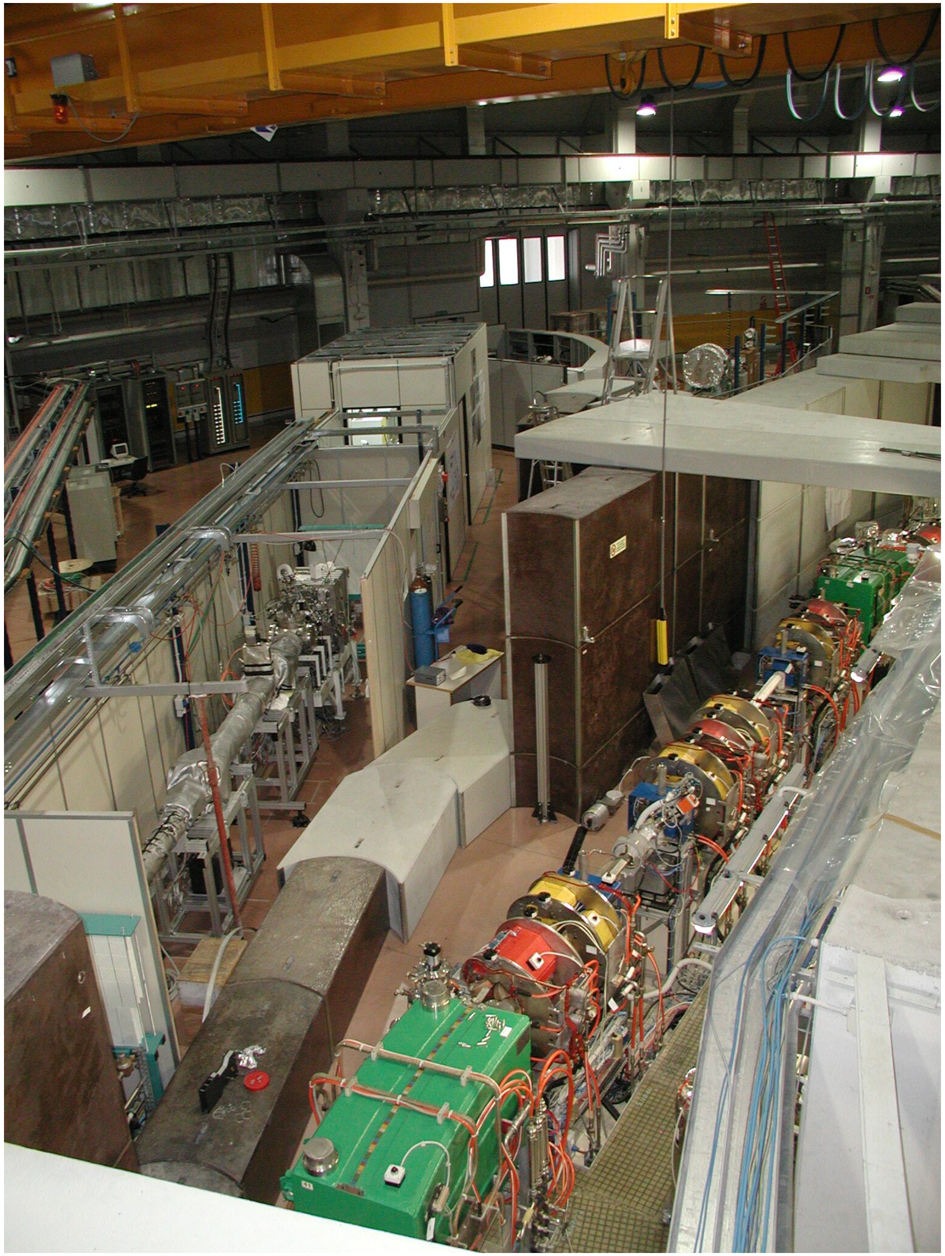
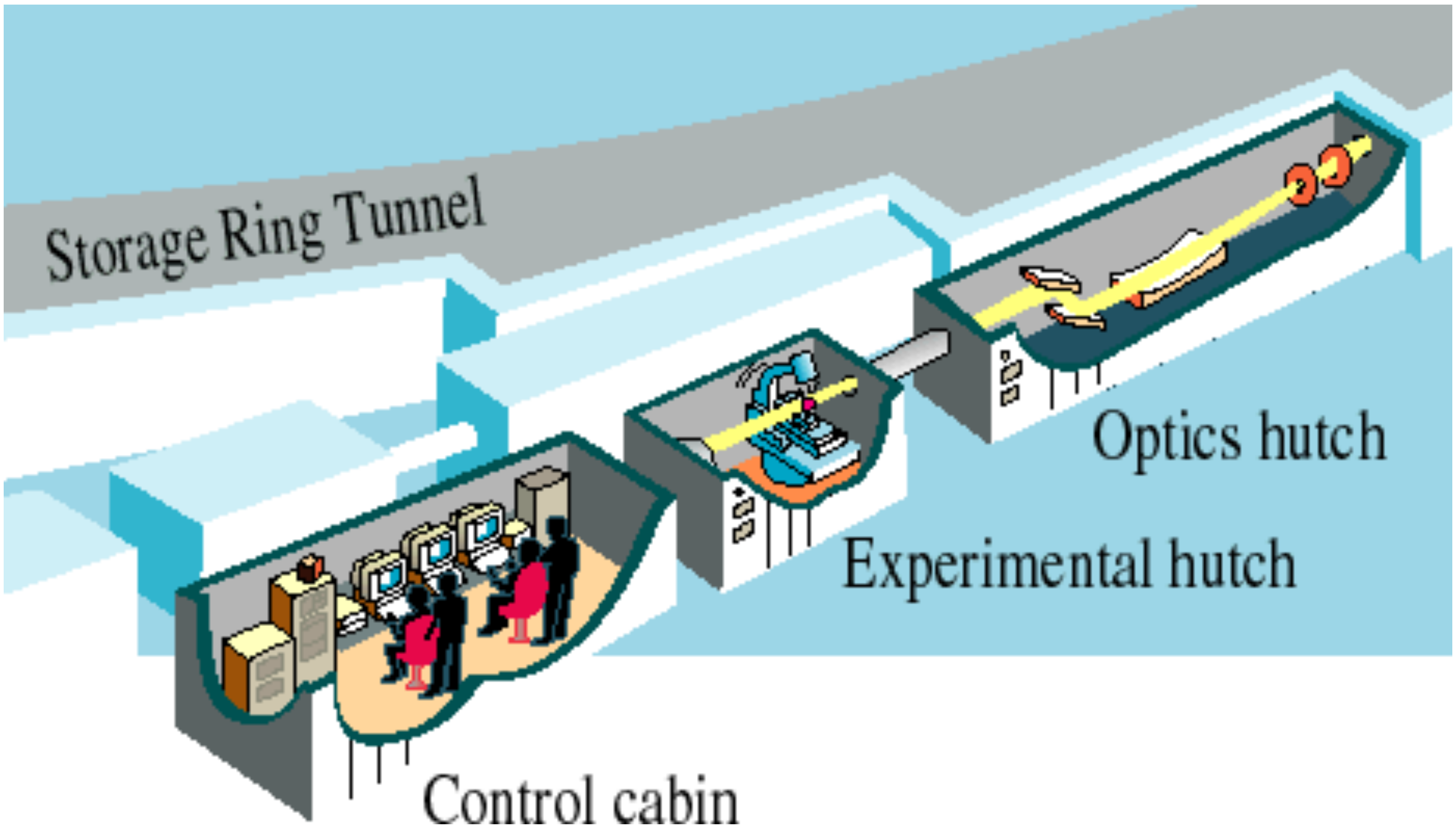
What does it look like inside?



The experimental station



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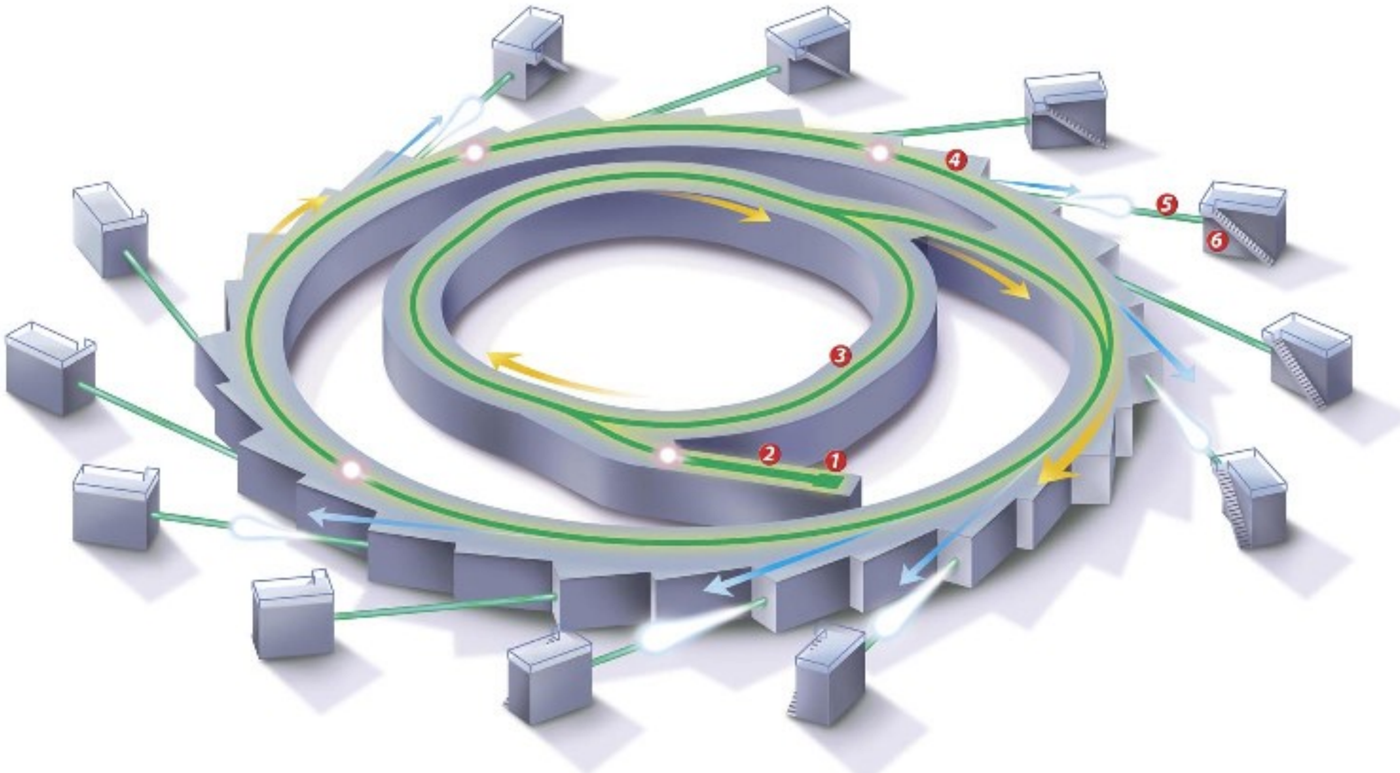
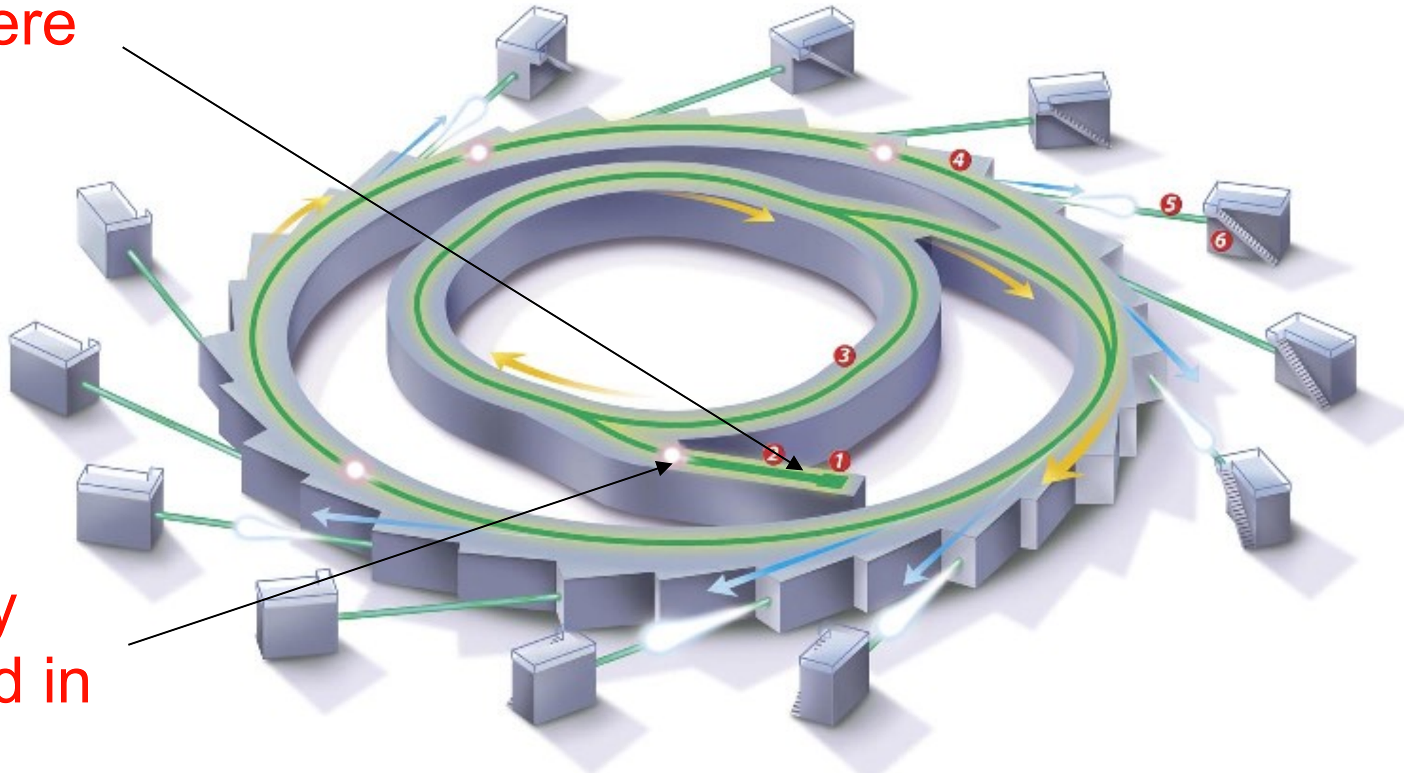
How does it work?



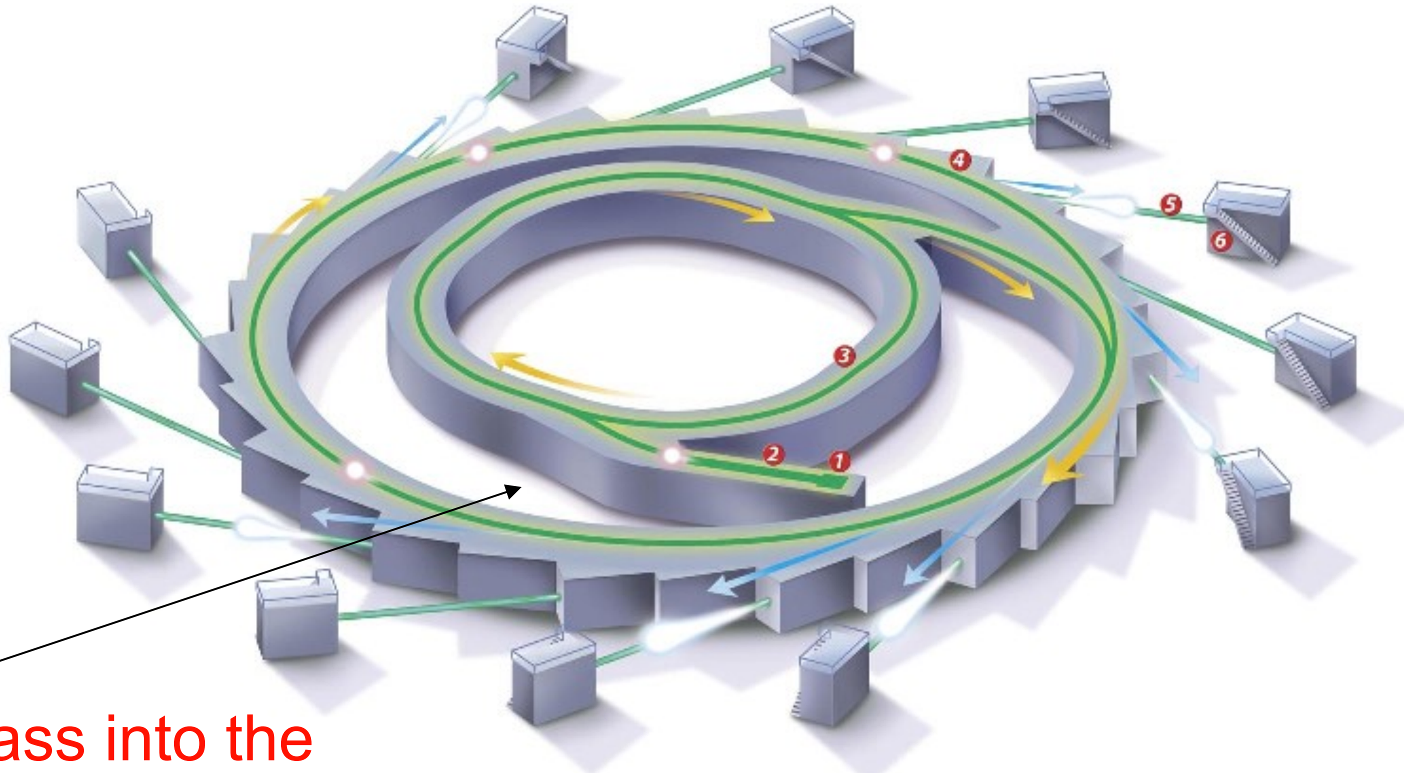
How does it work?

Electrons are generated here



And initially accelerated in the LINAC

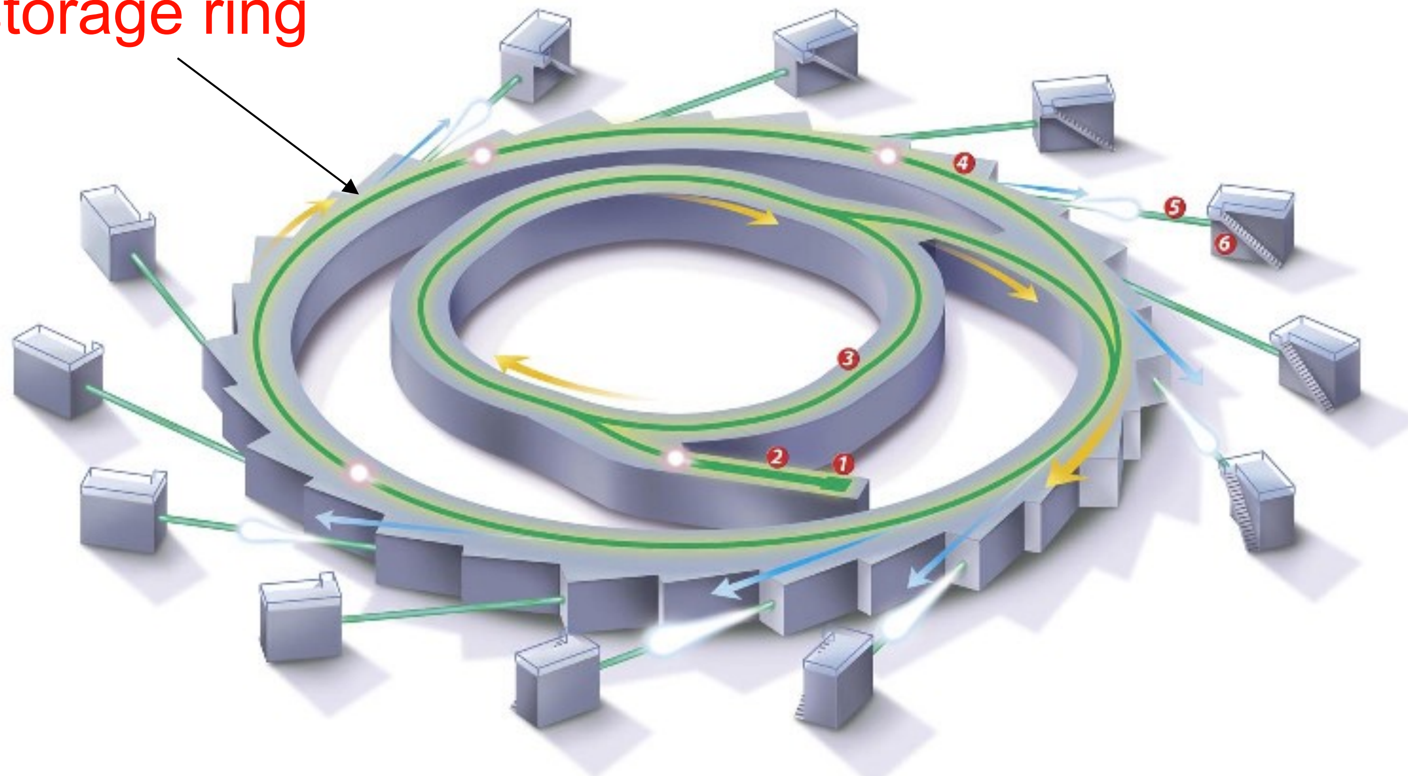
How does it work?



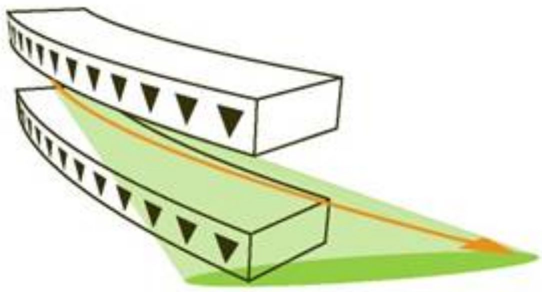
Then they pass into the booster ring where they are accelerated to 99.9999% of *c*

How does it work?

And are finally transferred into the storage ring



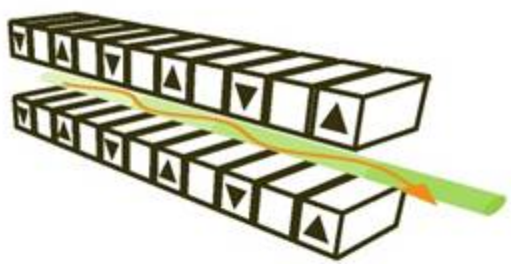
How does it work?

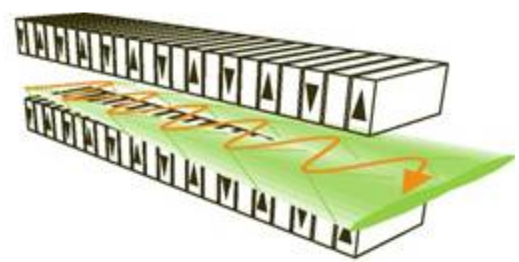


Bending magnet

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

Insertion devices - produce higher intensity





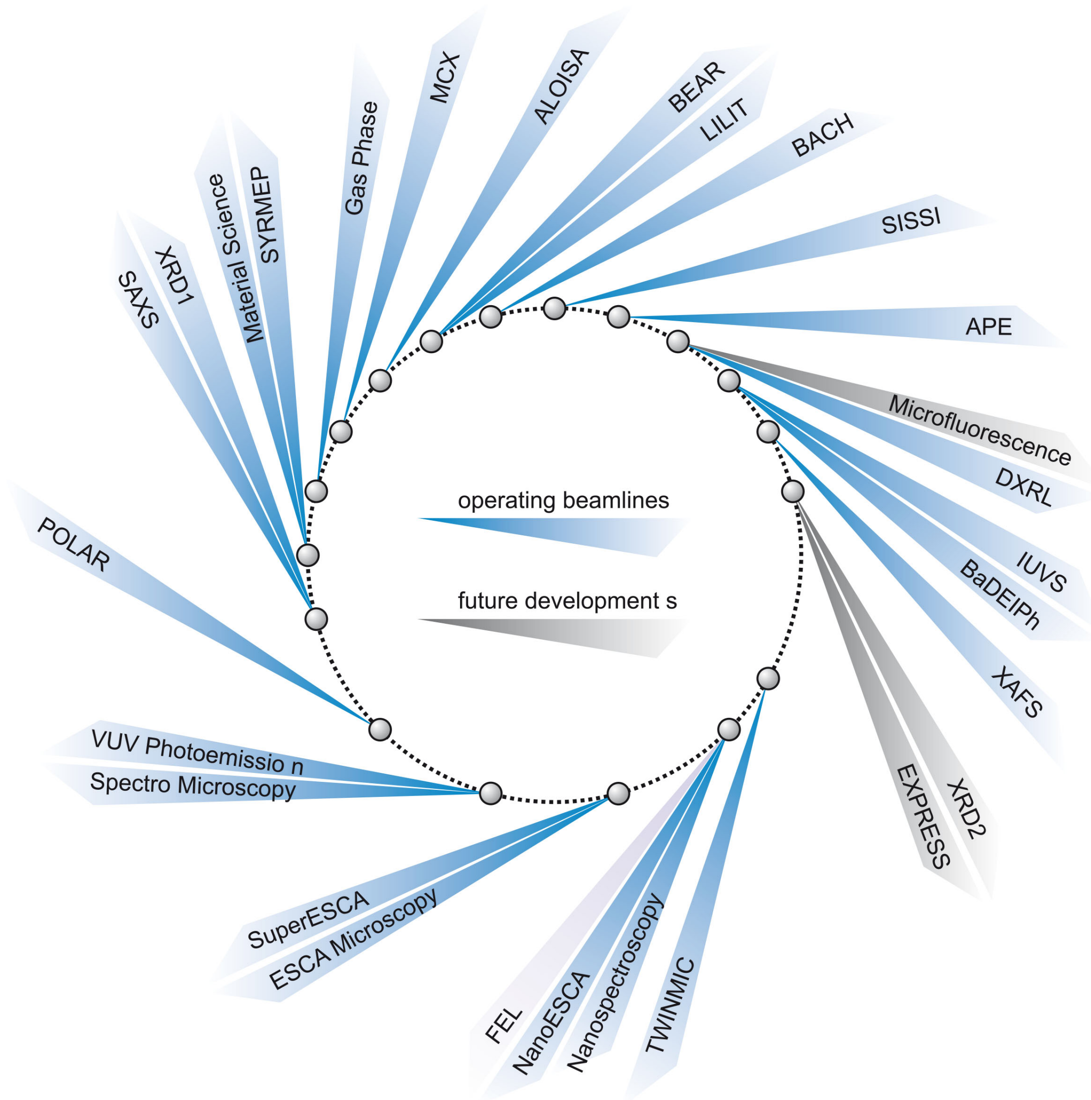
Undulator

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.

Elettra today



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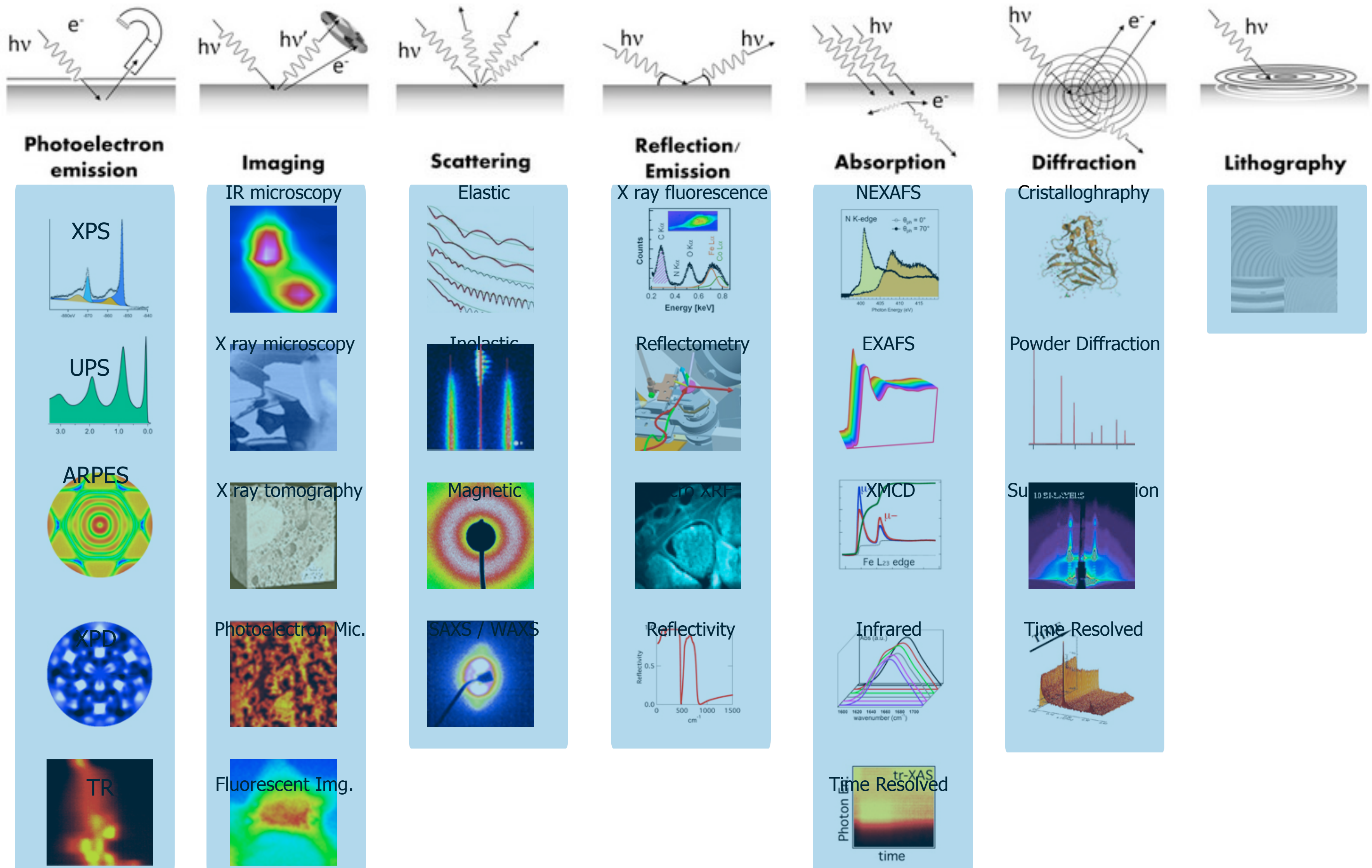
The Techniques



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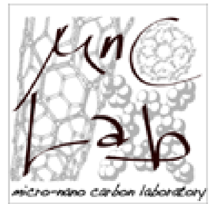
Support Laboratories

CITIUS

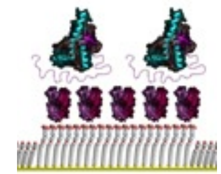
The new Interreg project for the development of a state-of-the-art light source generating ultrashort pulses in the UV and soft X-ray spectral range.
Read more...

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...

NanoLab

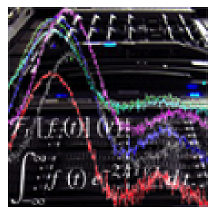


The lab carries out research on surface confined bio- molecules and self- assembled monolayers using atomic force microscopy.
Read more...

Organic OptoElectronics

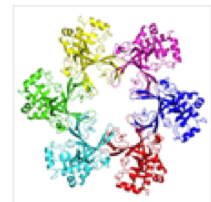
The lab investigates the properties of organic semiconductors, either molecular or polymeric, and their applications.
Read more...

Scientific Computing



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

Structural Biology



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

Support Lab

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

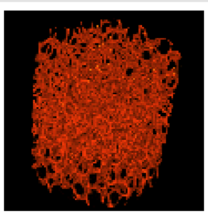
Surface Science

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

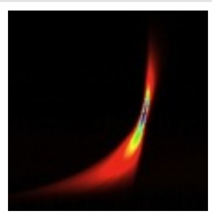
Theory@Elettra is the theory group funded by the CNR-INFM DEMOCRITOS supporting the experimental activity performed in the laboratory
Read more...

Tomolab



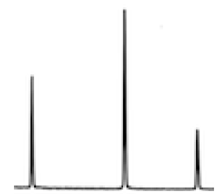
The TomoLab station at Elettra provides a state-of-the-art X-ray computed microtomography system based on a microfocus source.
Read more...

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...

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

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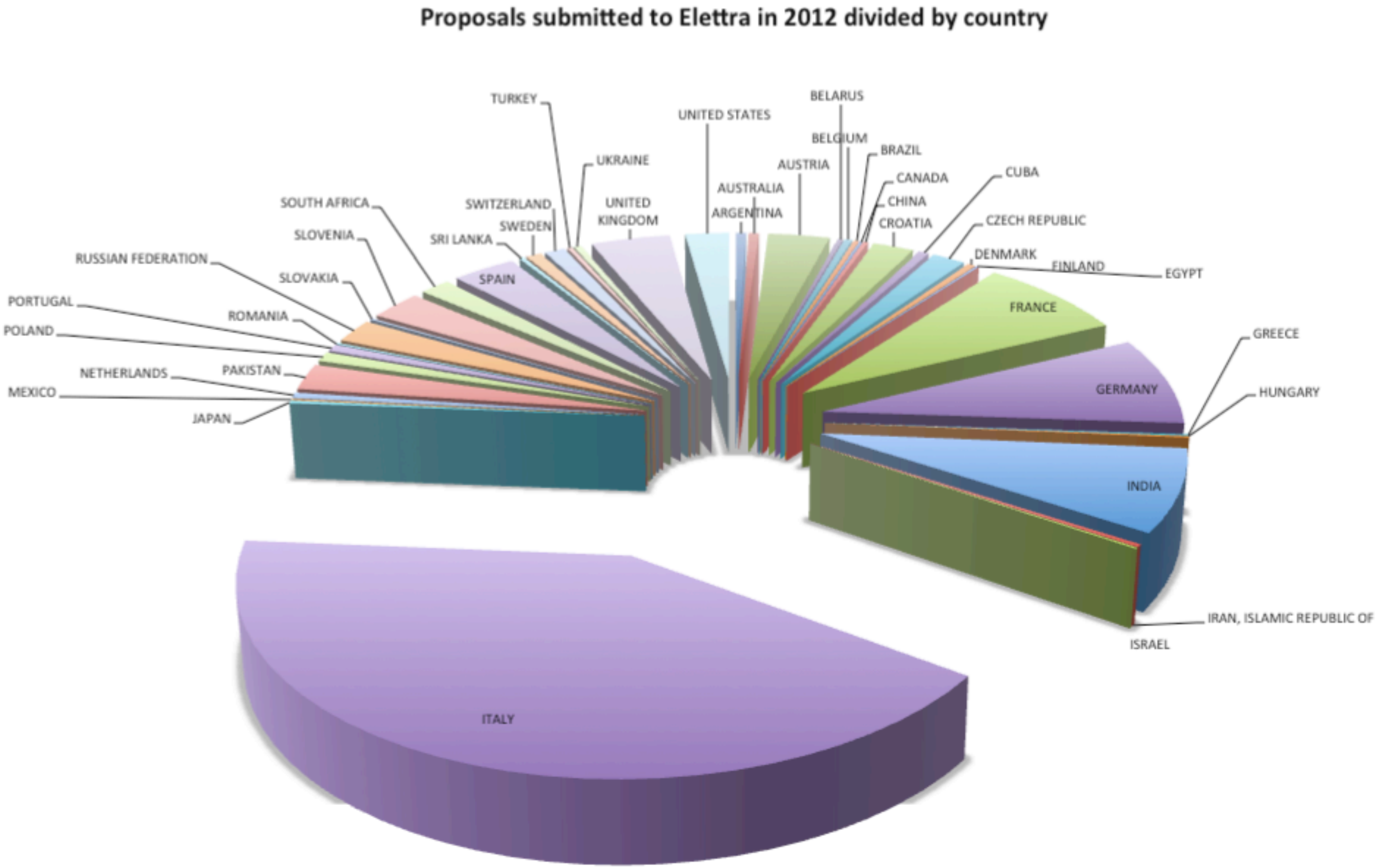
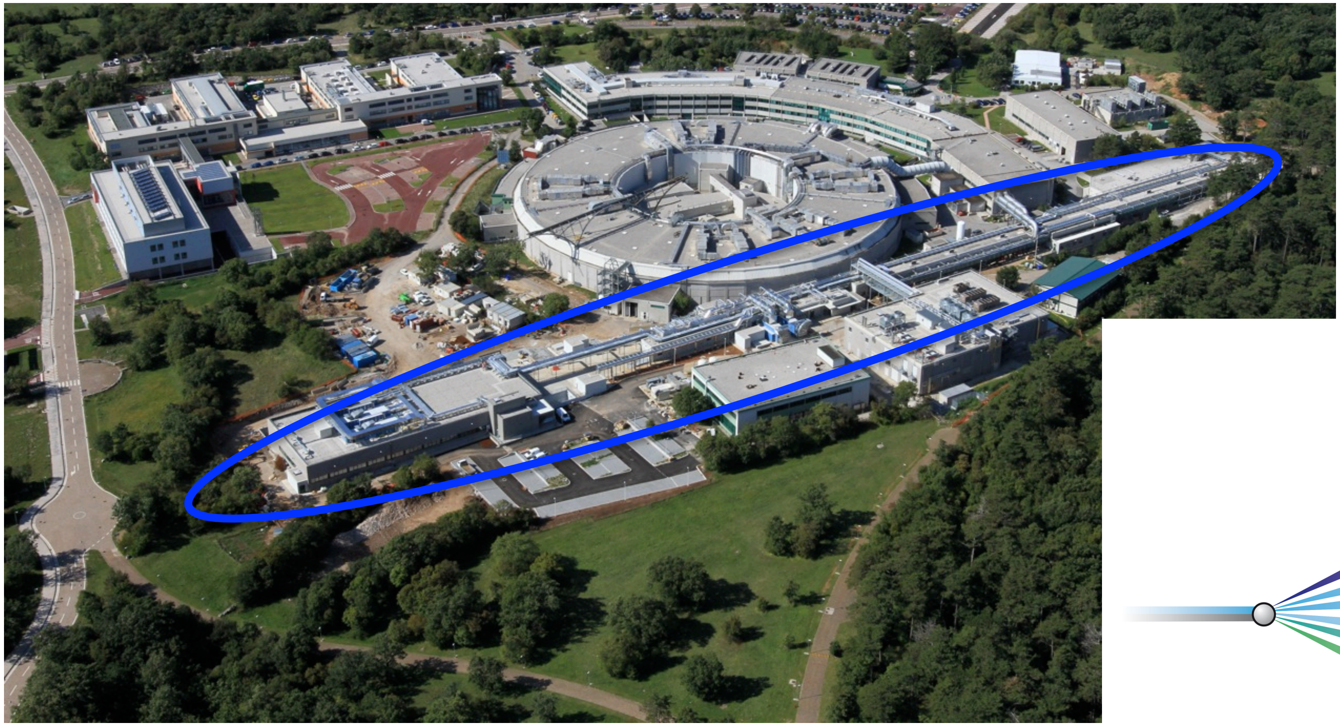
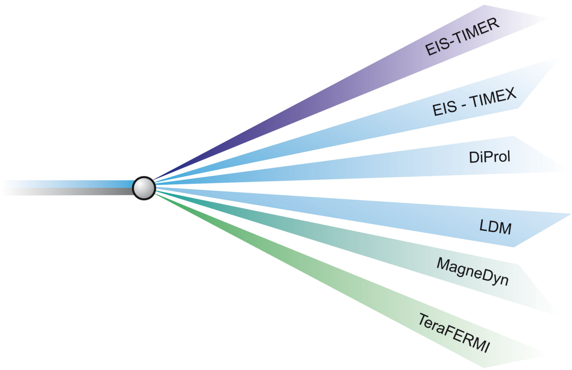


FERMI Lightsource

FERMI now in operation performing its first experiments. It integrates Elettra performances in the femtoseconds range





Our guidelines

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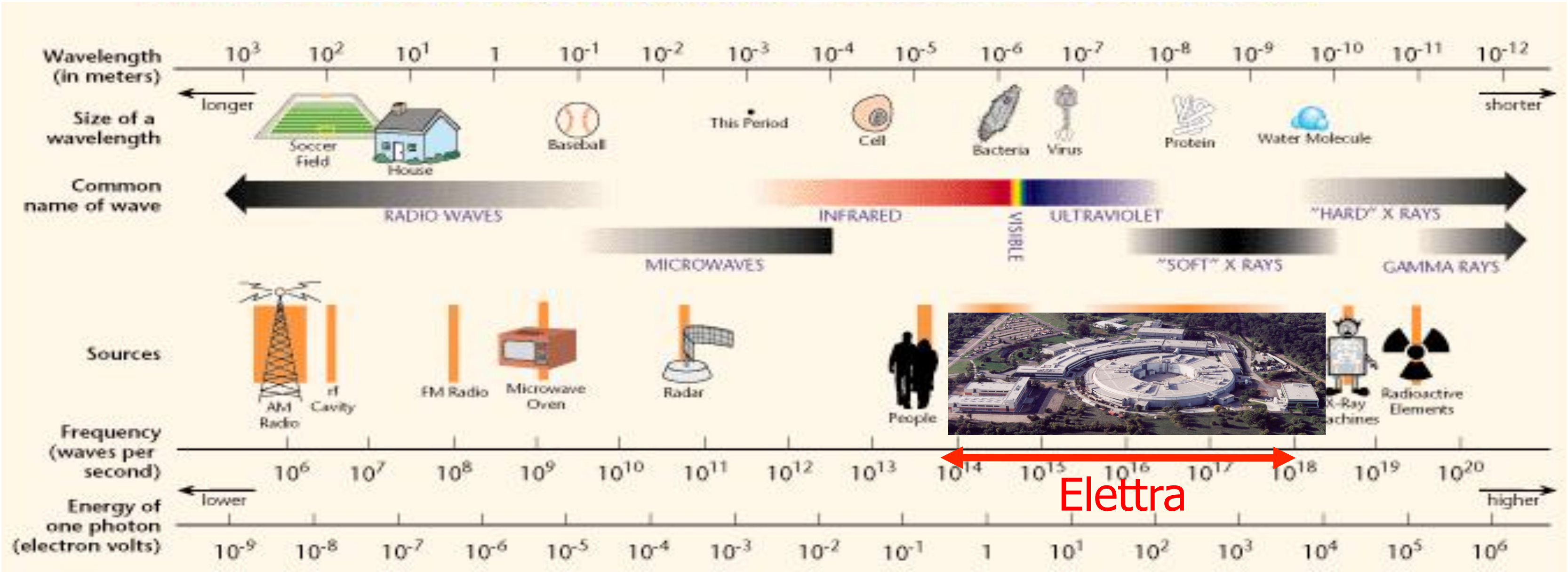
- ✓ Direct access to an extensive range of facilities and techniques
- ✓ Feasibility Study is “free of charge”
- ✓ Quotations based on time, cost and performance
- ✓ Activities structured as a Project
- ✓ Continuous collaboration, sharing of results, knowledge transfer

Synchrotron light

- Tunable: possibility to select the beam wavelength
- Intense: possibility to obtain extremely fast acquisitions
- Coherent / collimated: similar to a laser beam



Cetervm censeo, mvndvm non delendvm esse

- Samples of great historical and/or commercial value
- Monitoring of restoration and conservation protocols

Artis monvmentorvm qvi vnvm vidit nvllvm vidit, qui mille vidit vnvm 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 microdistructive
- Sinergies between conventional labs and large research infrastructures
- Easy access to thematic networks and fundings

X-ray imaging at a 3rd generation SR facility

- high energy photons and high flux

  → **heavy** and/or **bulky samples** in transmission geometry

  → **tunability** in a large energy range

  → **short** exposure times
- small angular source size and big source-to-sample distance

  → use of **natural coherence properties** of the beam

The **SYRMEP beamline**:

- Source size σ (h x v) ≅ **1100 μm x 100 μm**
- Source-to-sample distance: **D ≅ 24 m**
- Beam size at sample (h x v) ≅ **150 mm x 6 mm**
- Energy range: **8 ÷ 35 keV**, Bandwidth: $\Delta\lambda/\lambda \cong$ **2×10^{-3}**

SR X-ray imaging studies in archaeometry

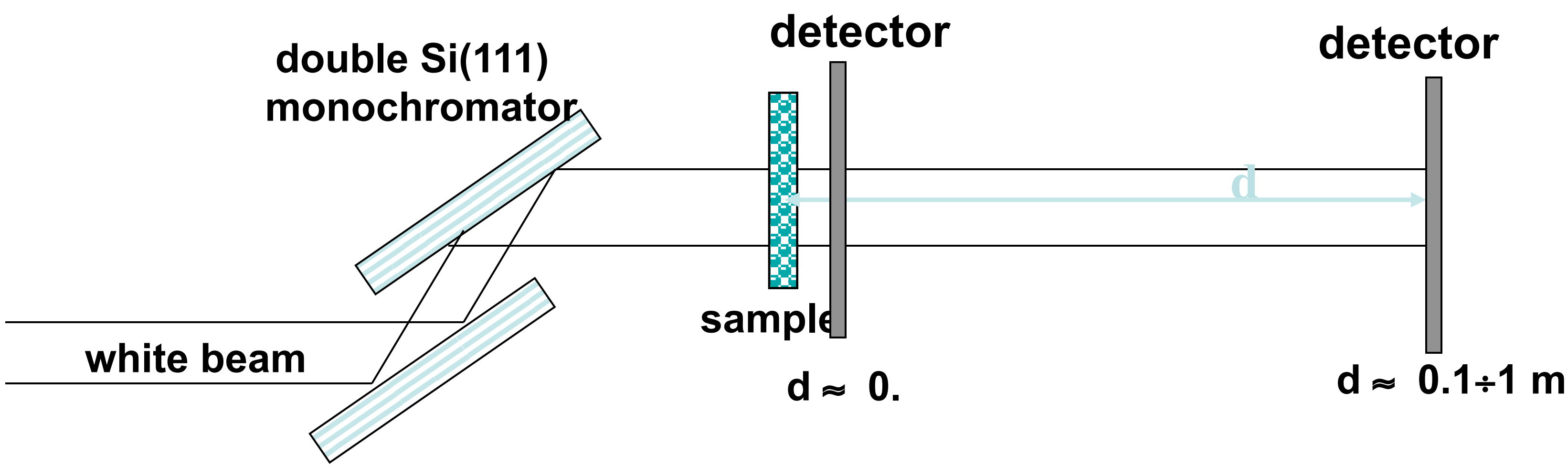
In-situ and ex-situ experiments in a large range of applications:

→ 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



$(\Delta I/I)_{abs} = e^{c\,\Delta\mu} - 1$

$\Delta\phi = 2\pi\, c\, \Delta\delta / \lambda$

n = 1 - δ - iβ : refraction index

μ = 4π β / λ : linear absorption coeff.

c : object size // to beam direction

$r \ll a \Rightarrow$ edge detection regime

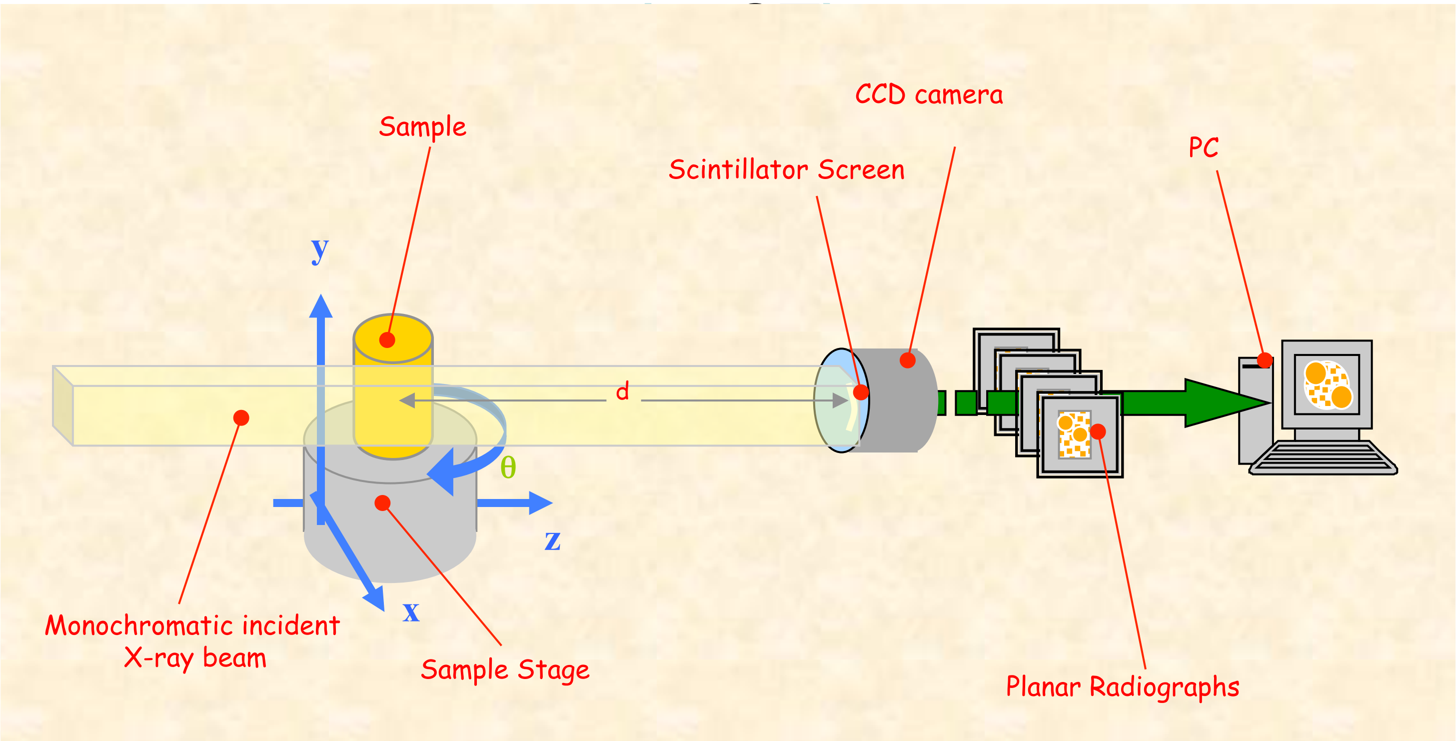
$r \cong a \Rightarrow$ holographic regime

$r \gg a \Rightarrow$ Fraunhofer diffraction

a : object size ⊥ to beam direction

$r = (\lambda\, d)^{1/2}$: first Fresnel zone radius

Absorption and PS Computed μ-Tomography



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

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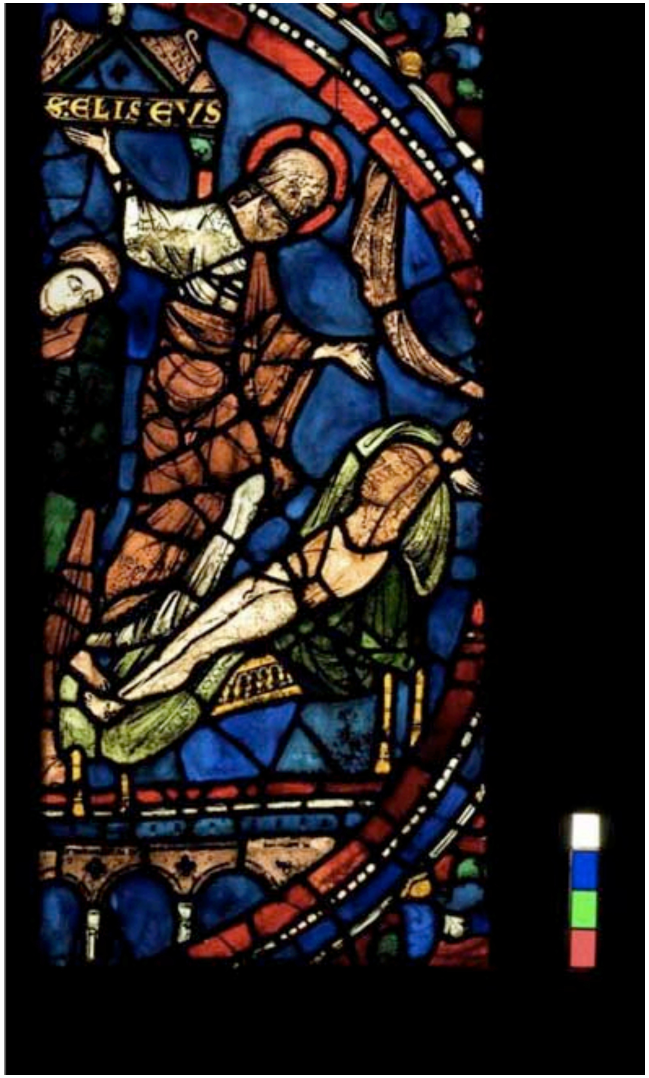
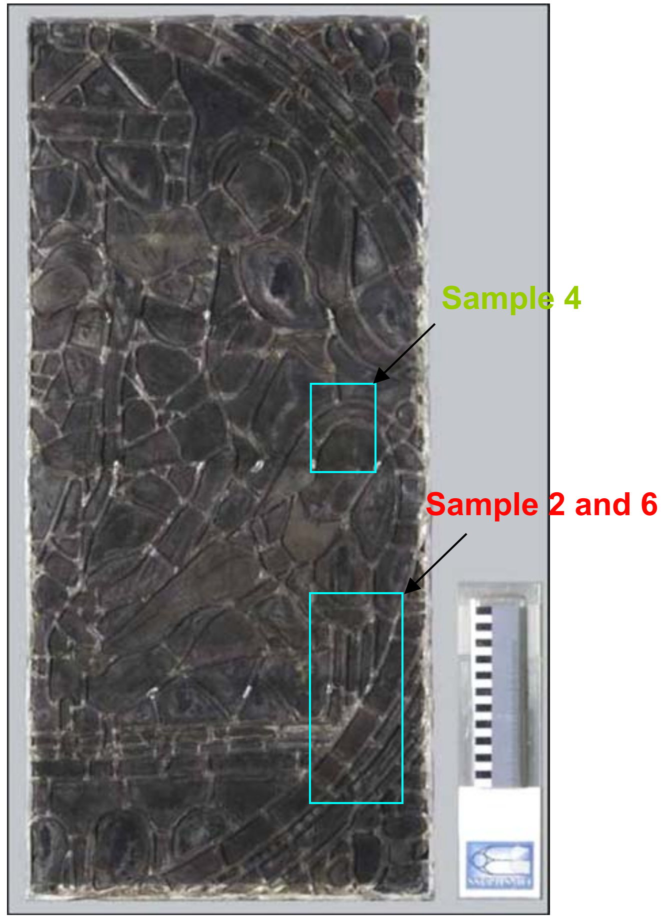
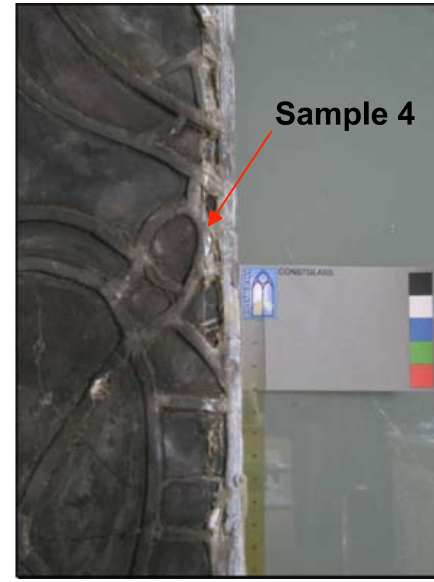
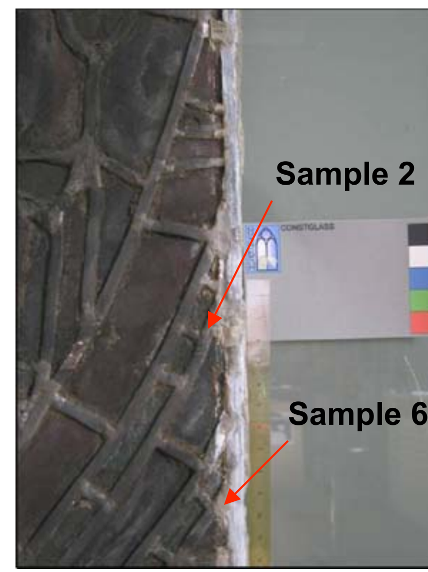
Chartres - Window 37
La Passion typologique

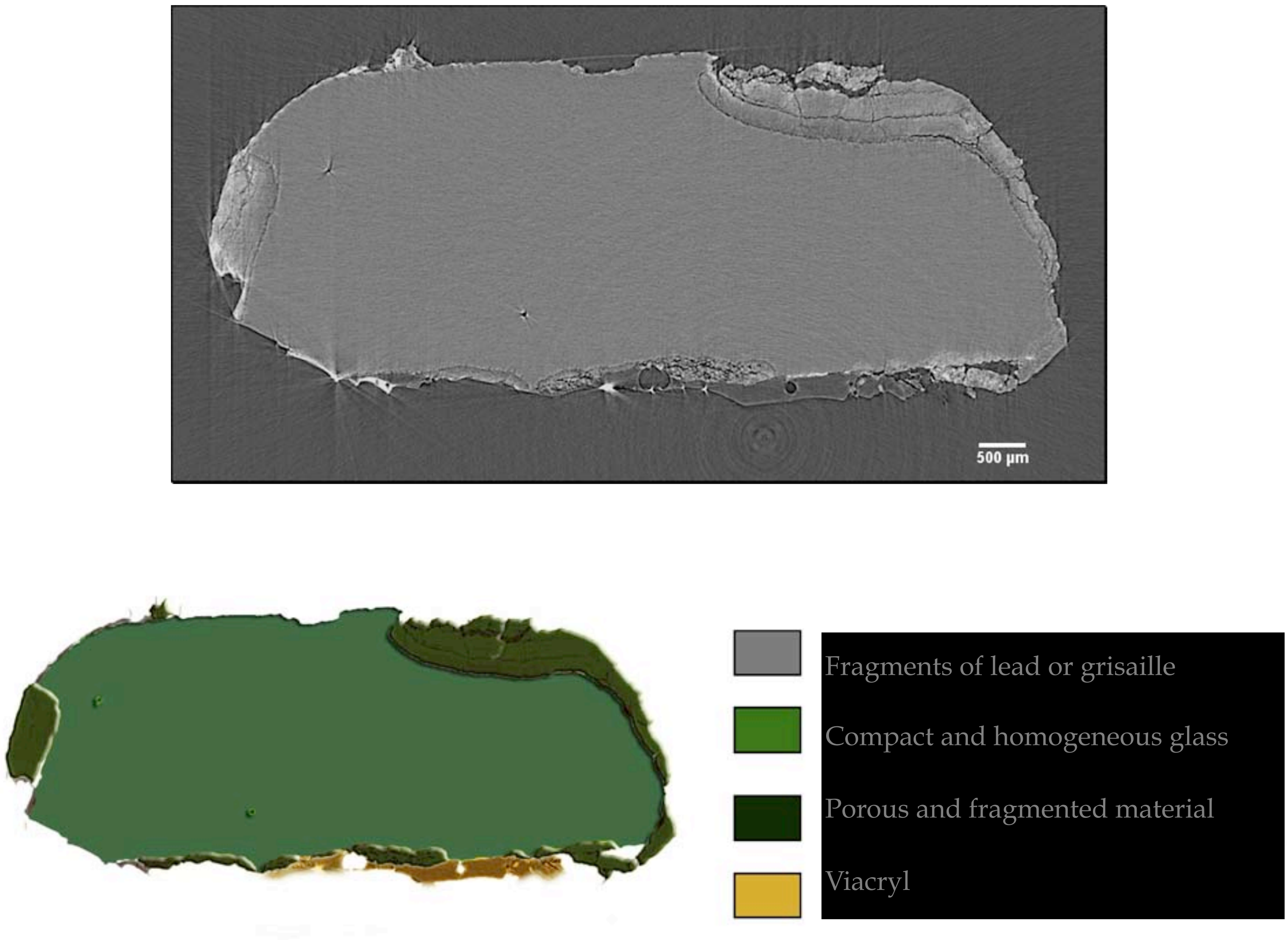




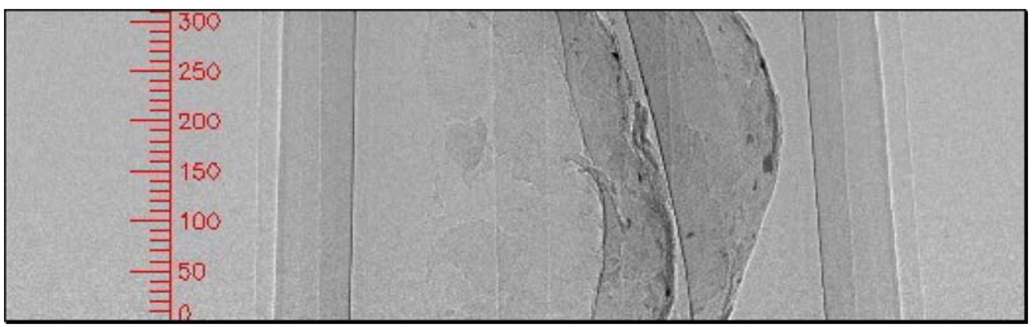




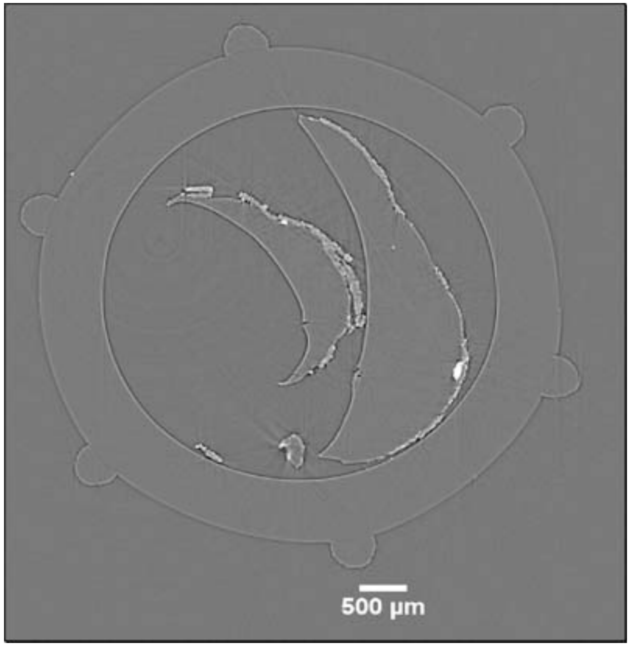
Sample 2: glass fragment with grisaille decoration and Viacryl layer on the internal face. Evident alterations on the surface sides.



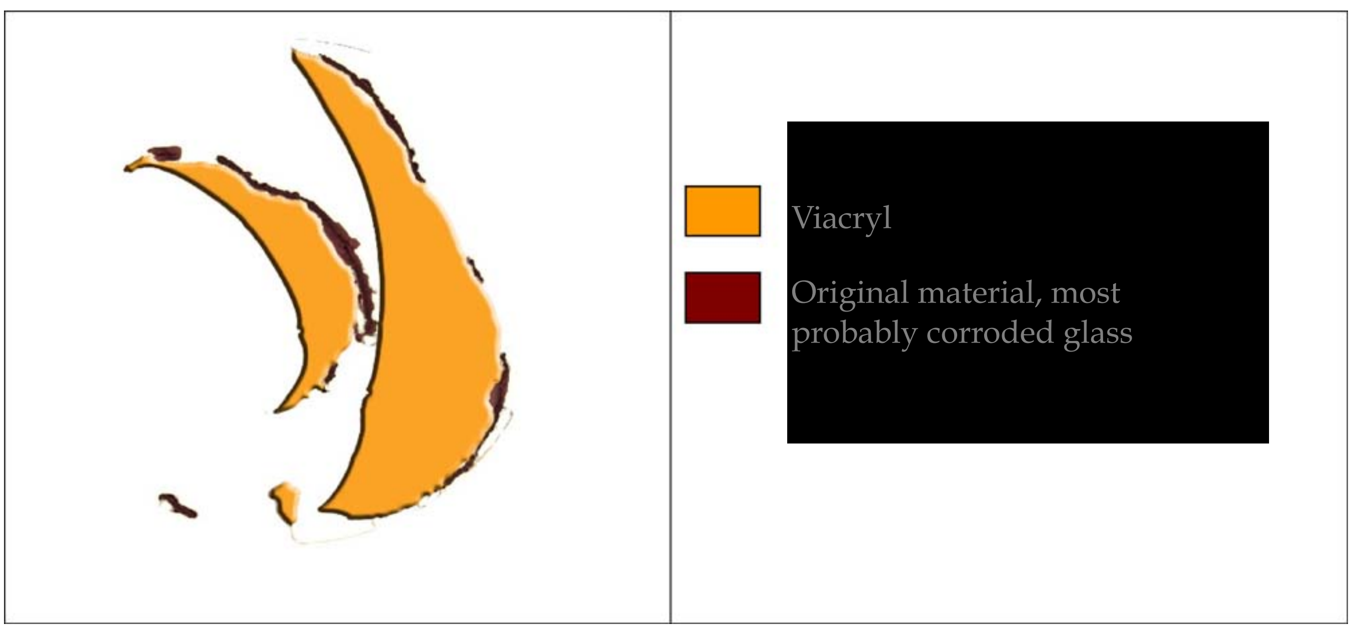
500 µm
Fragments of lead or grisaille
Compact and homogeneous glass
Porous and fragmented material
Viacryl

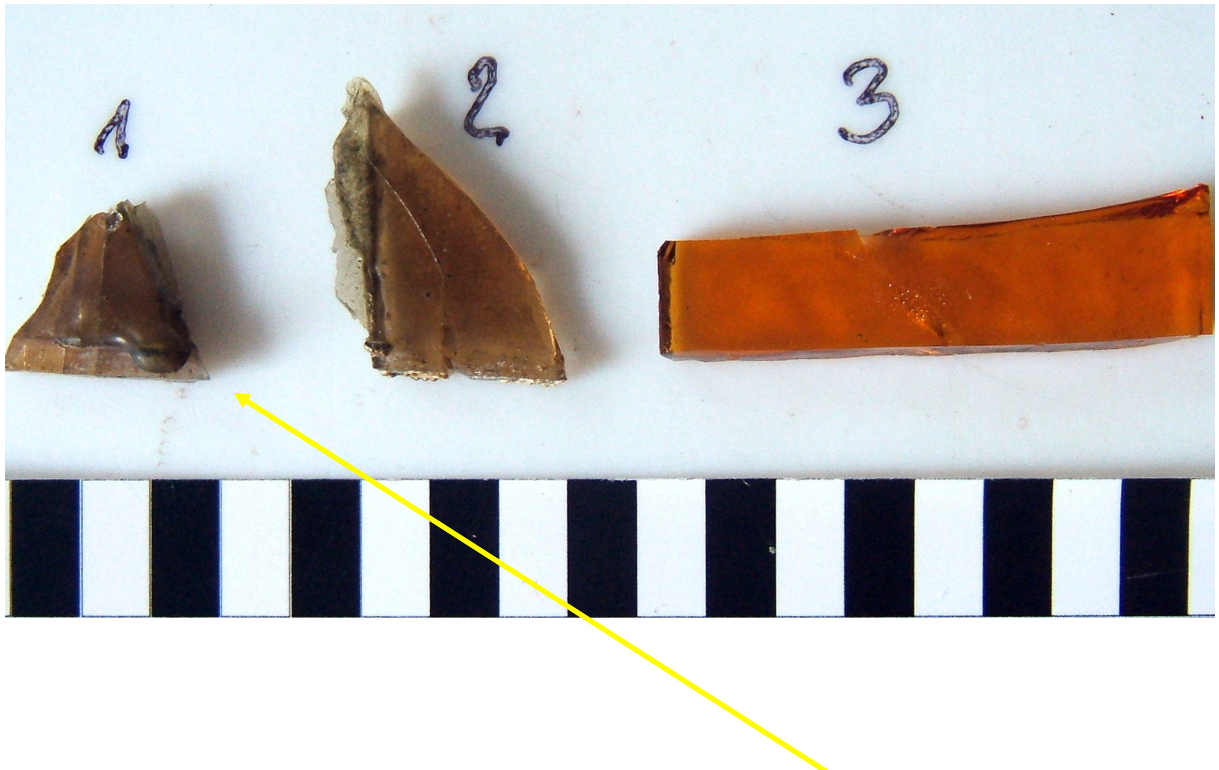


Viacryl flakes from Bourges
(window 9, panel 4)

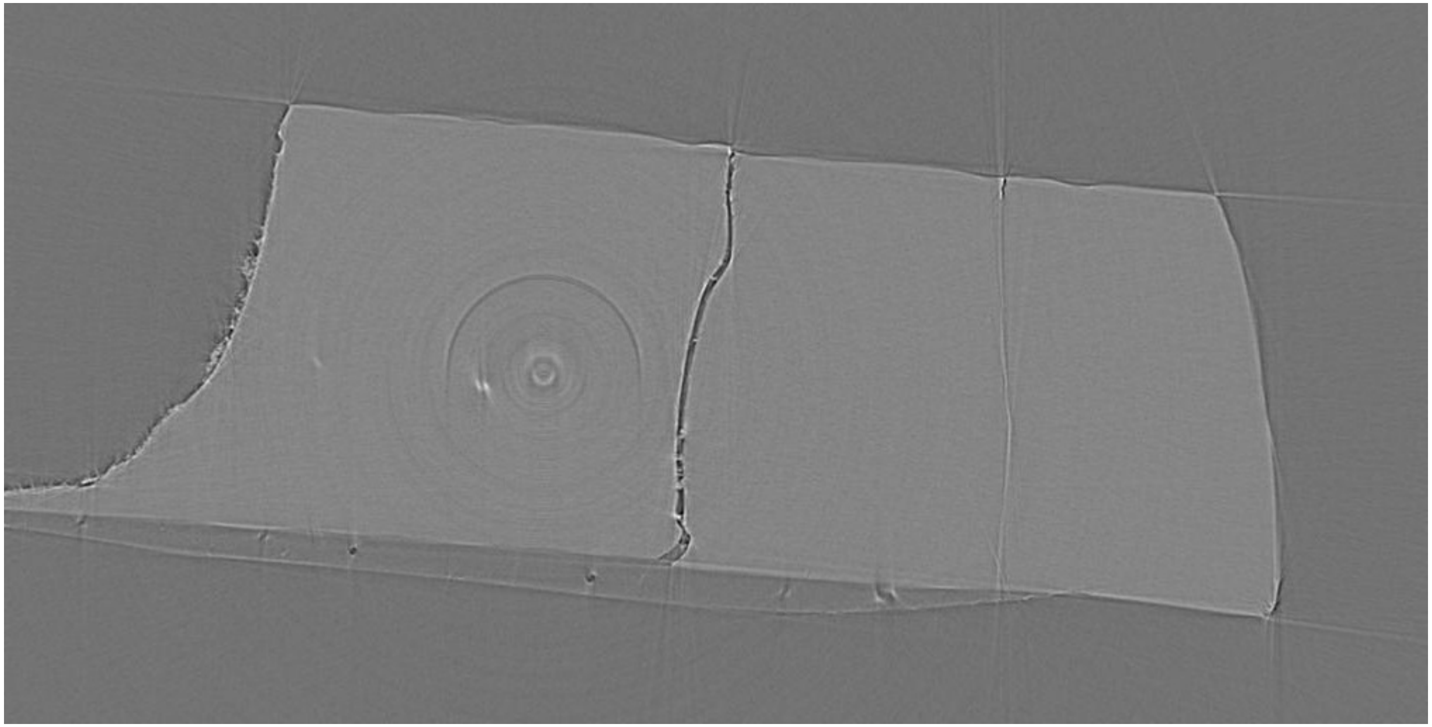


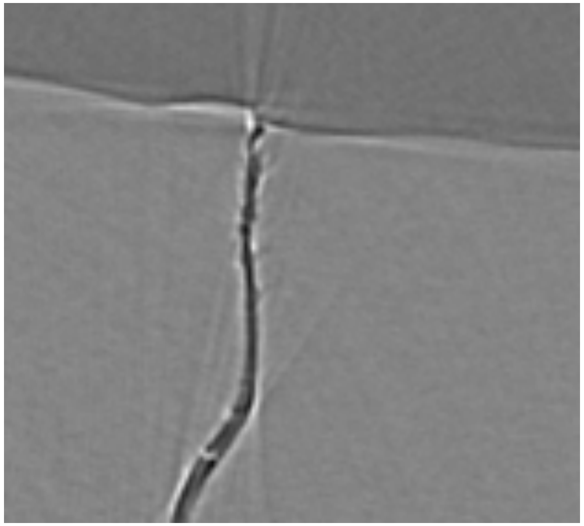
Viacryl flakes from Bourges (window 9, panel 4). It is evident that the Viacryl removes some original material from the glass panel.

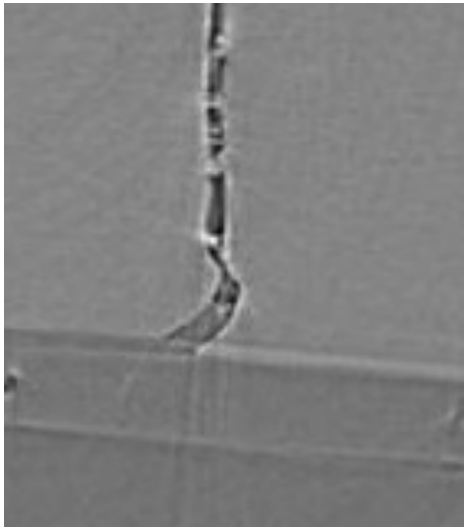


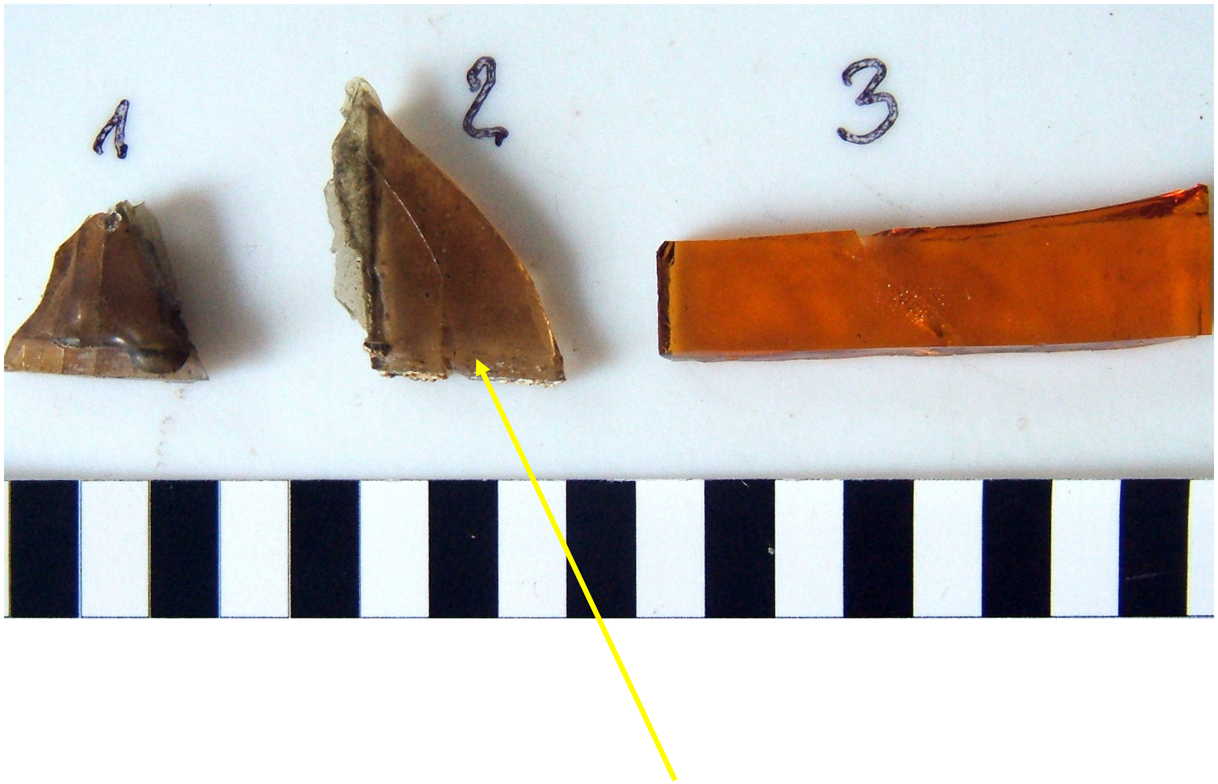


Original glass (beginning of 20th century) bonded with epoxide resin (Epidian 53) about 30 years ago from interior, without glass dismantling. Possible phenomena: thick layer of resin on the glass surface and slight penetration of resin into the break Break itself is soiled by external pollution. Bonding rather weak.

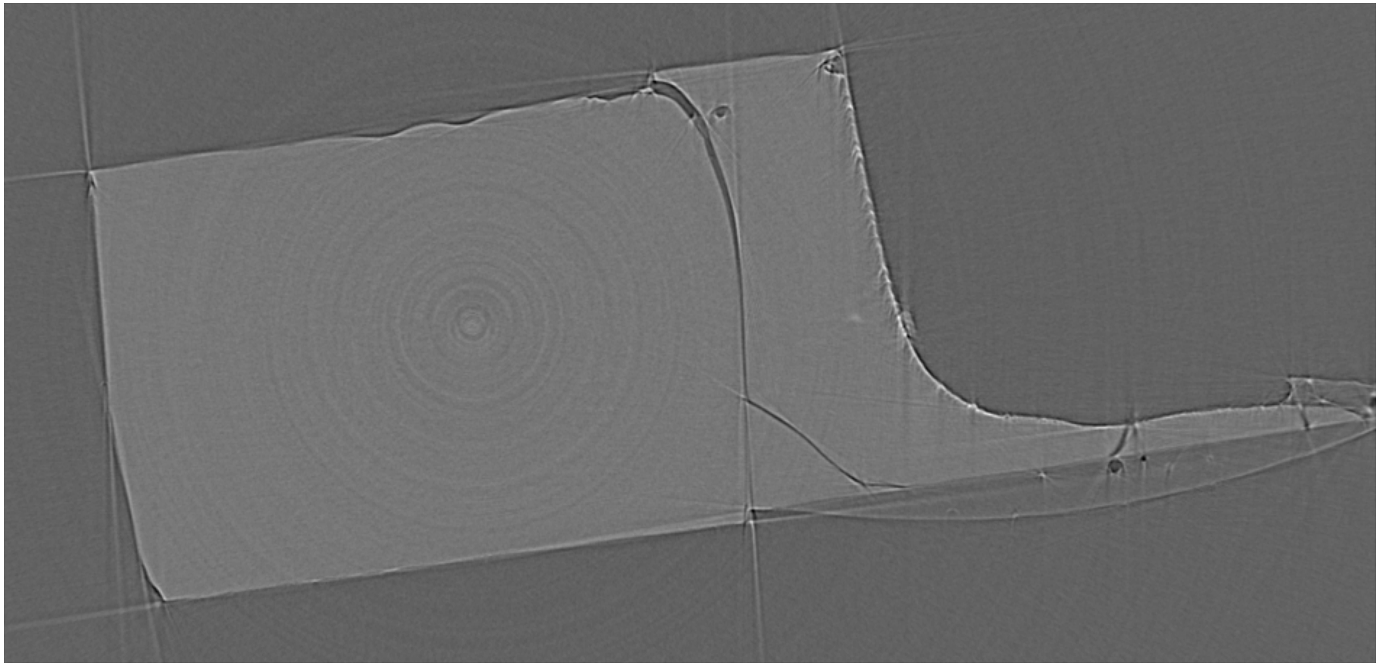


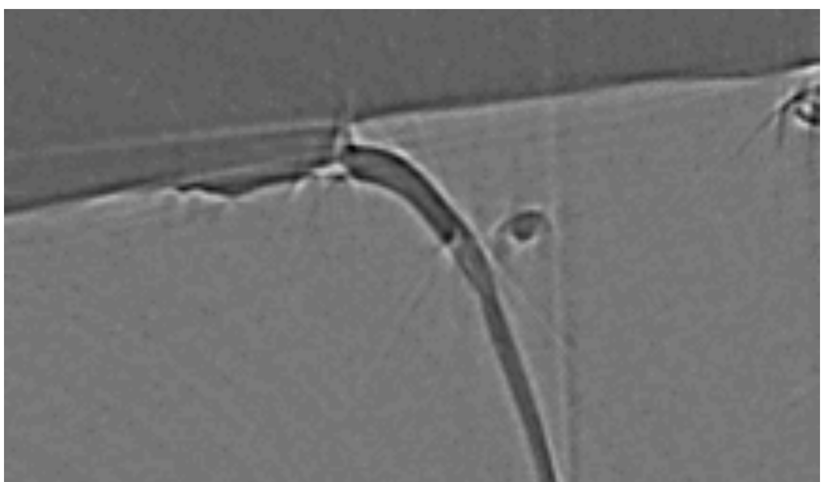


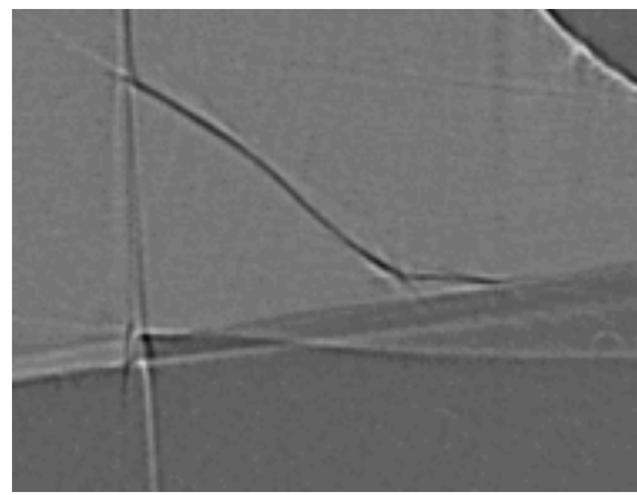


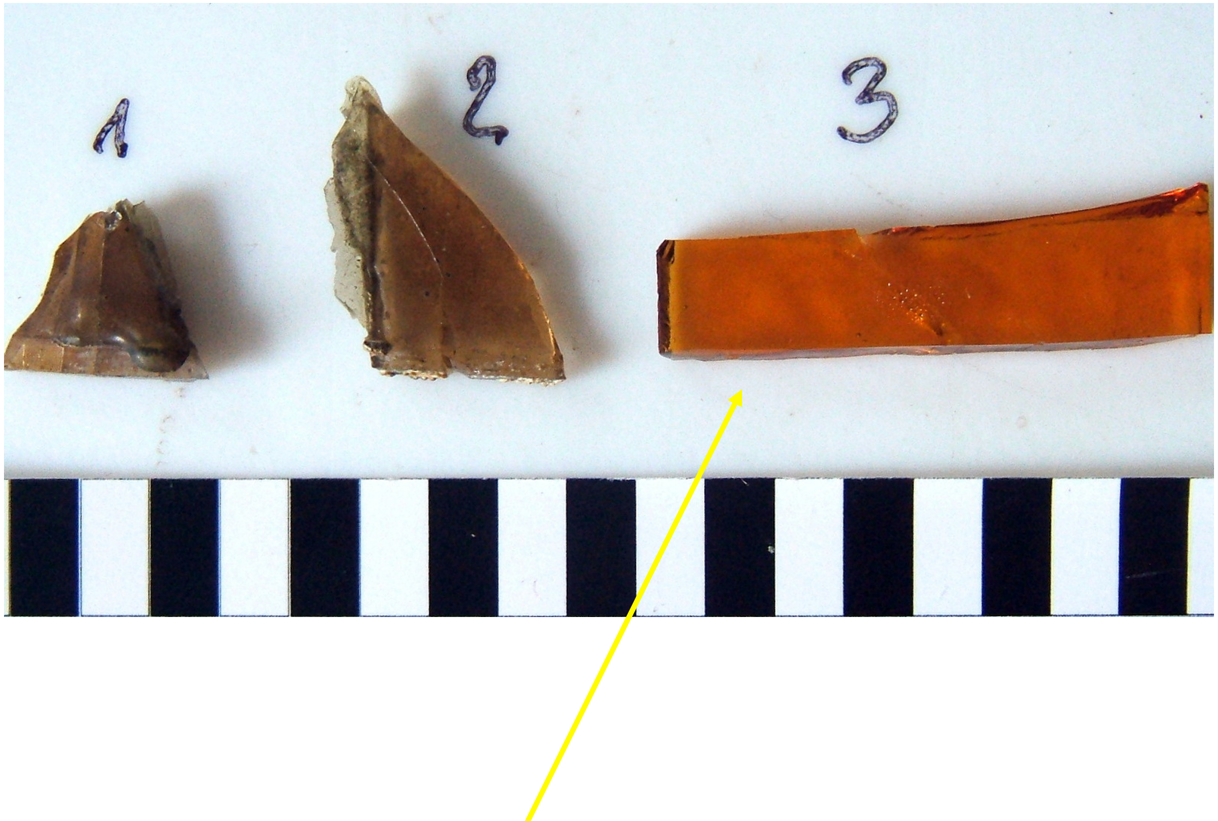


The same glass with epoxide resin on internal surface (on the left side of sample the resin only. The fresh break (center of glass) bonded with the same resin on 5th of August 2008).

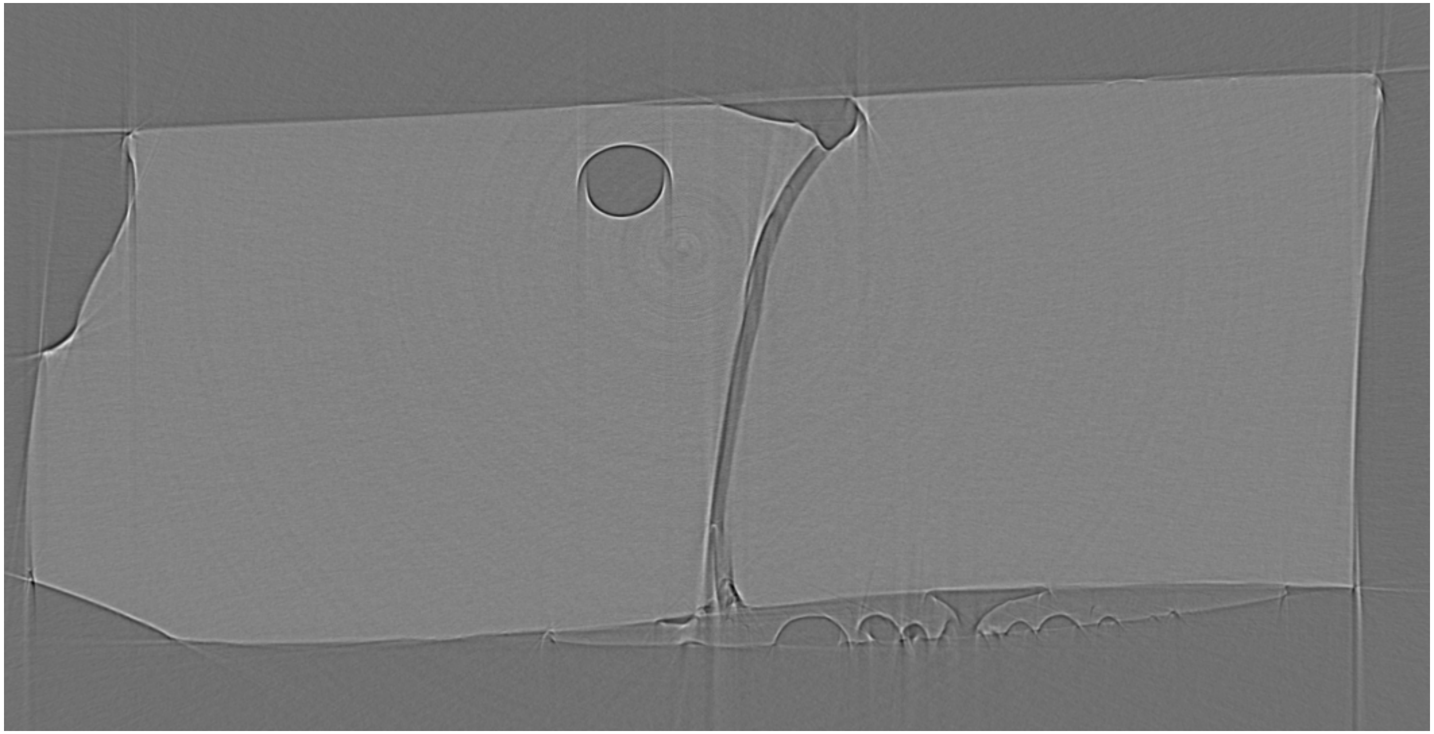
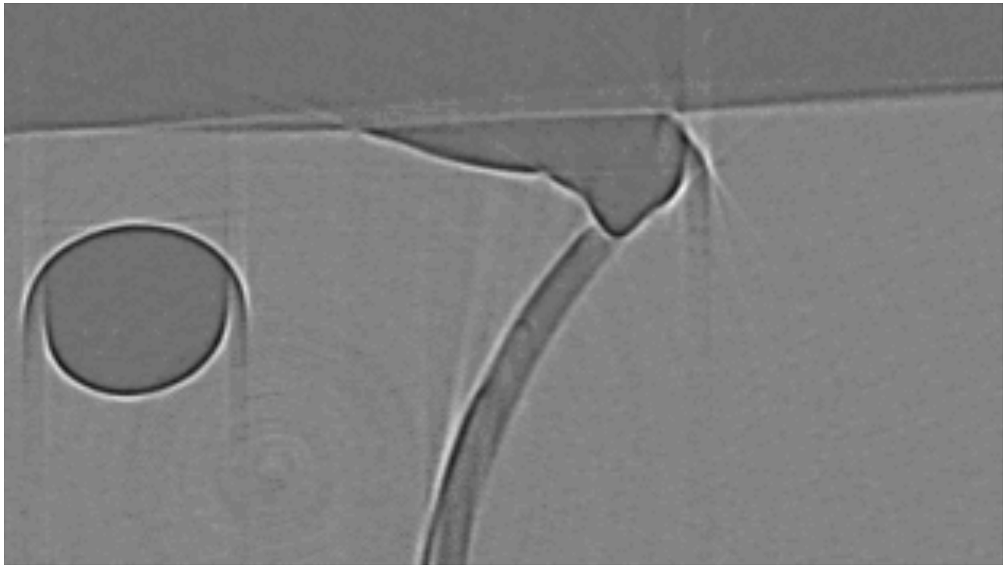
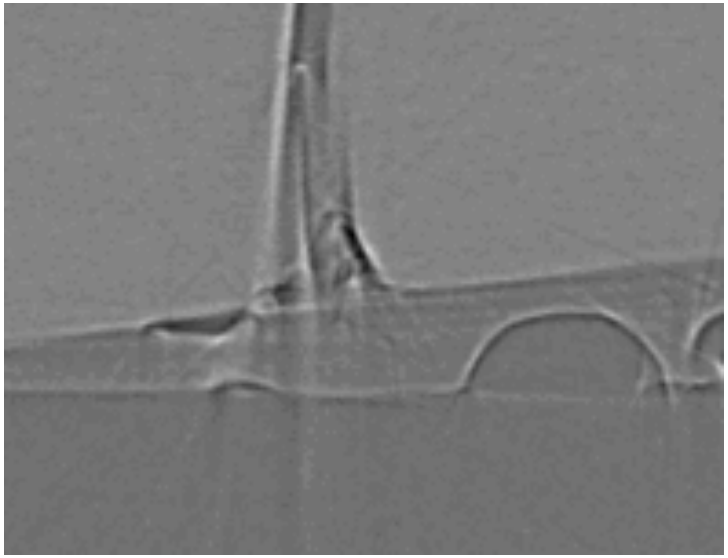








Modern broken glass bonded (with the same resin as sample 2) on August 5th, 2008.

Sample CAN 1a & 1b

Canterbury Cathedral.

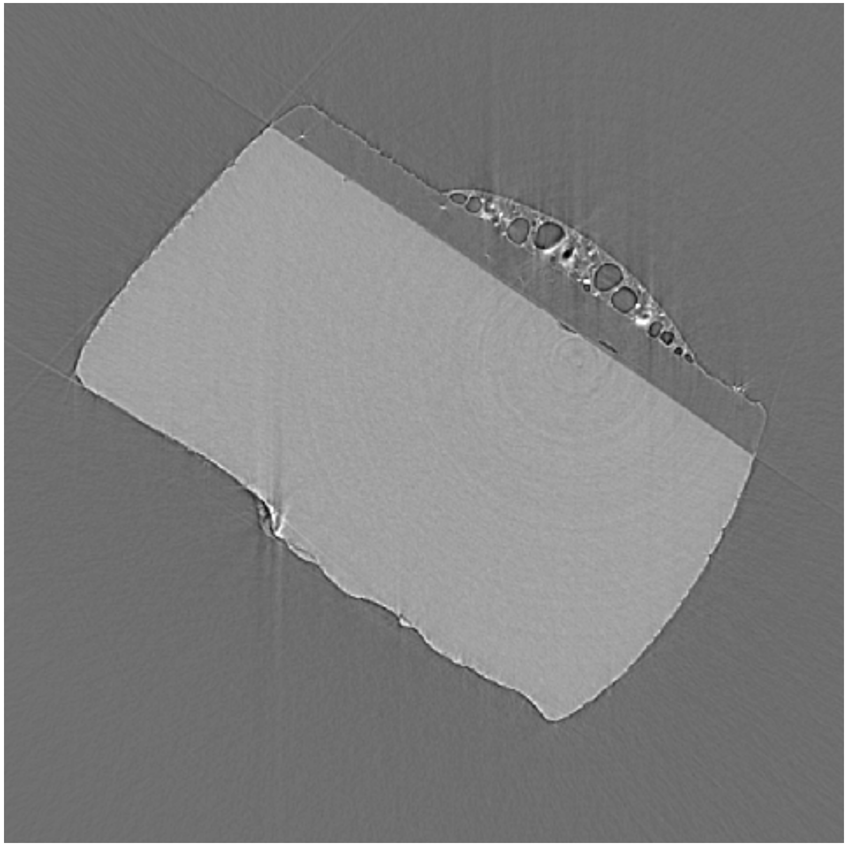
Unknown origin.

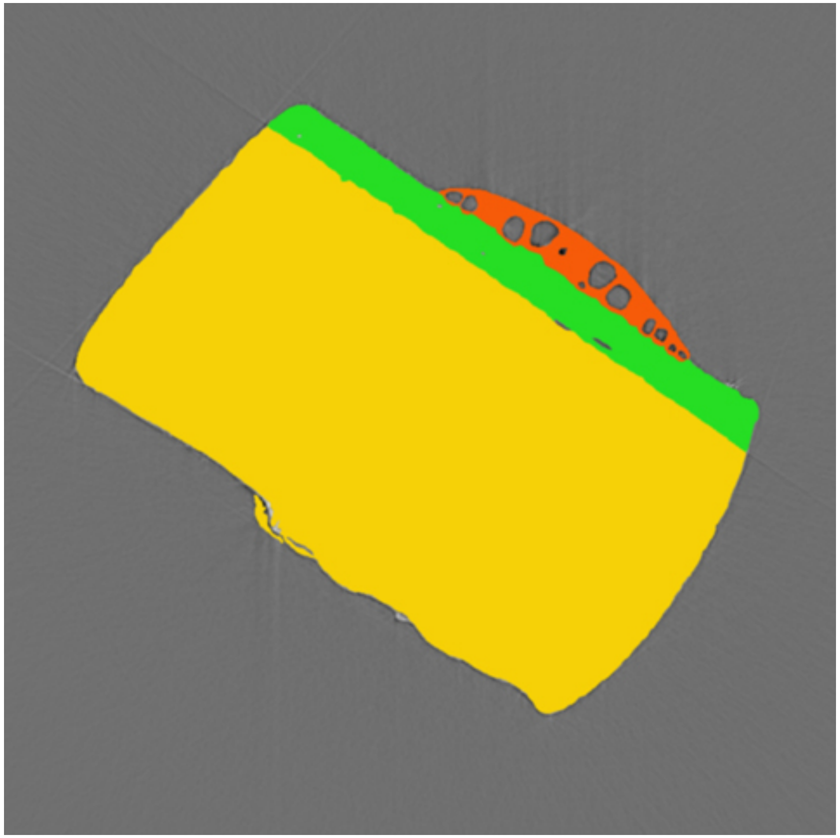
Fragment of medieval green tinted glass with slight surface corrosion.

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 centuryglass 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.

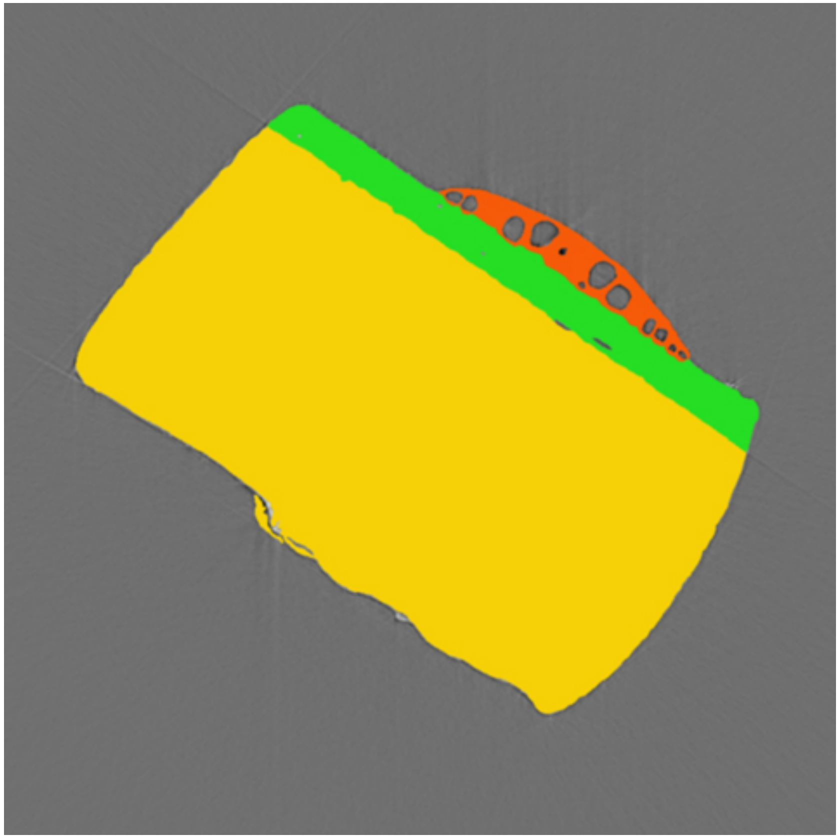
Sample CAN 1a

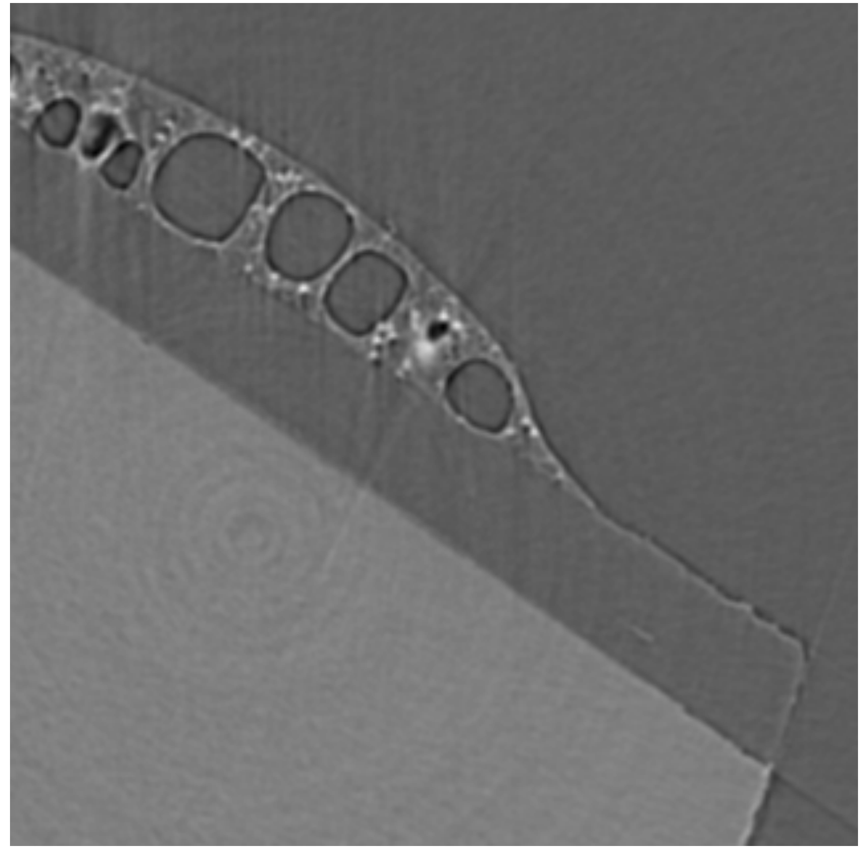




The wax was applied in 3 separate layers and left for three days. The Paraloid B72 was mixed together with raw umber pure powder pigment and this was applied with a small brush on the wax.

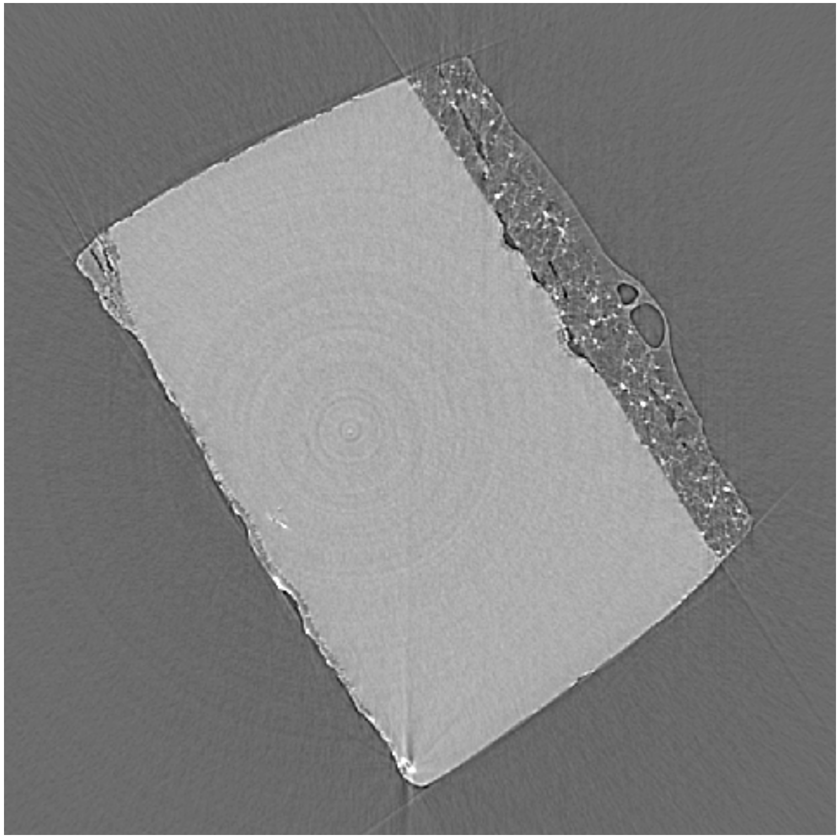
Sample CAN 1a

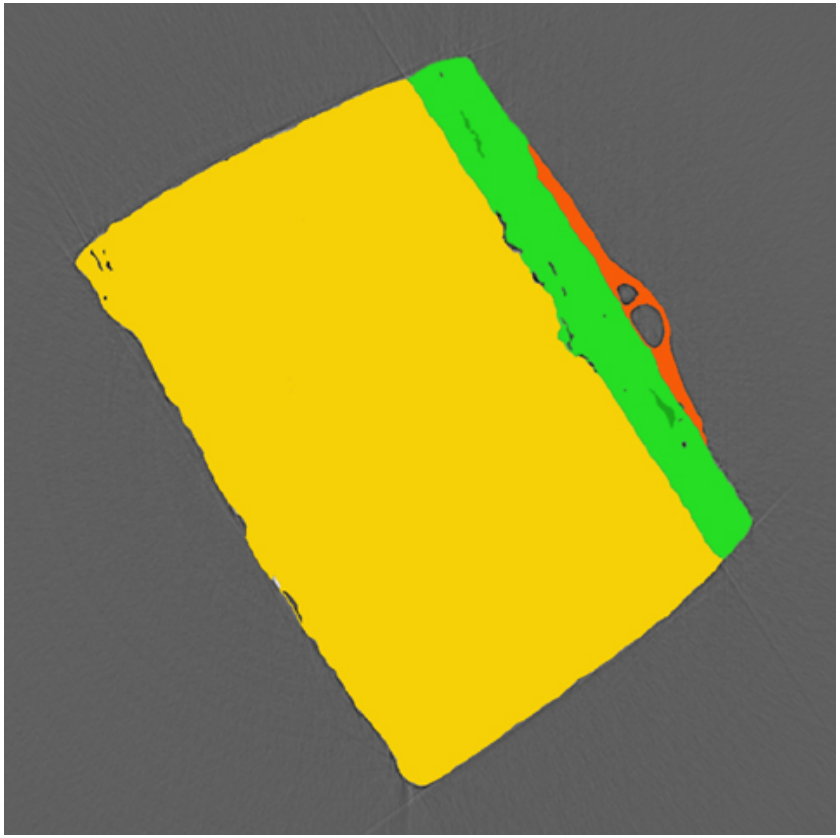




No traces of net interface between the different wax layers and between wax and Paraloid. The air bubbles in the Paraloid are due to the evaporation of the solvant.

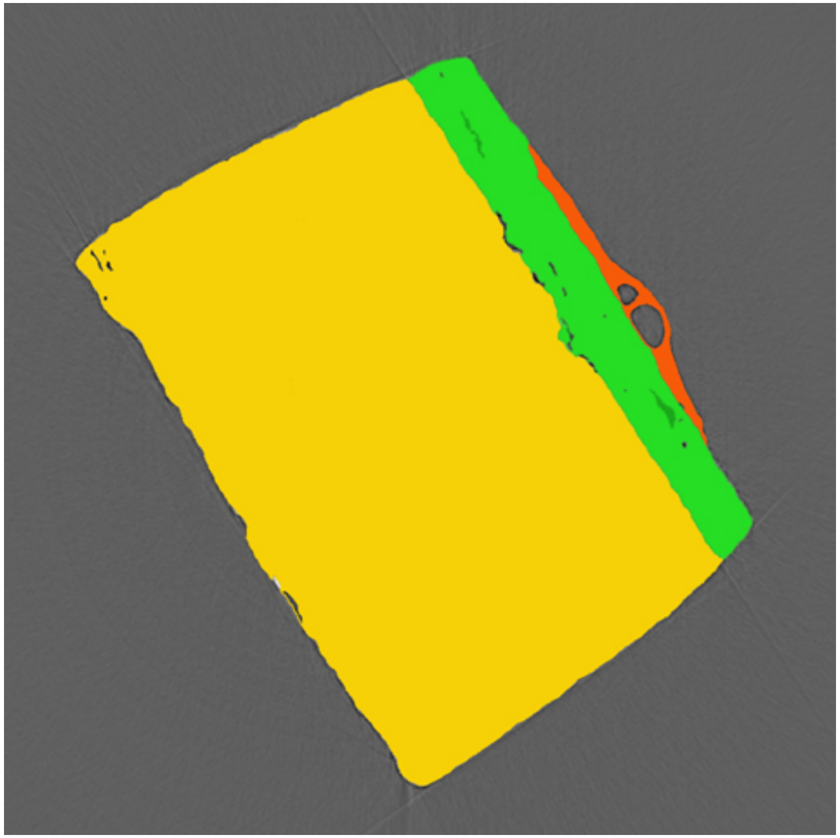
Sample CAN 1b

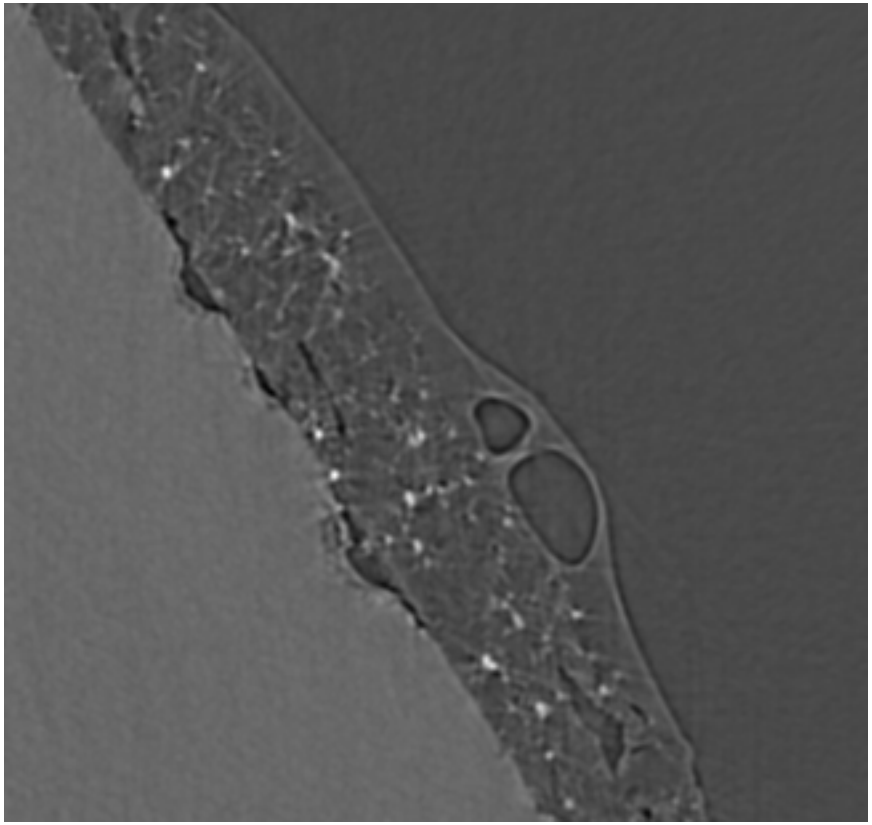




The wax was mixed together with raw umber pure powder pigment and applied in 3 separate layers and left for thre days. The Paraloid B72 was was applied with a small brush on the wax.

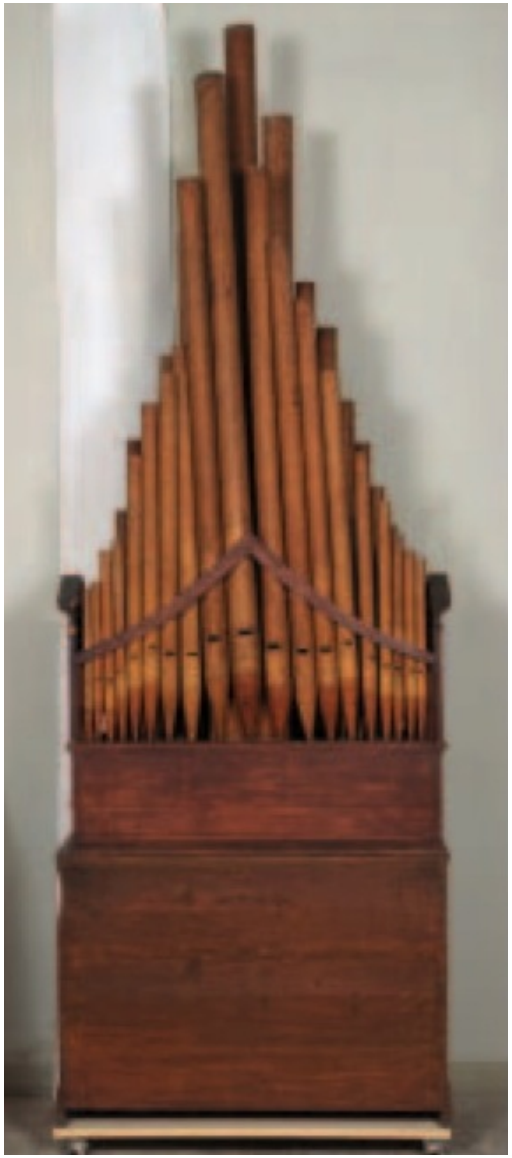
Sample CAN 1b





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



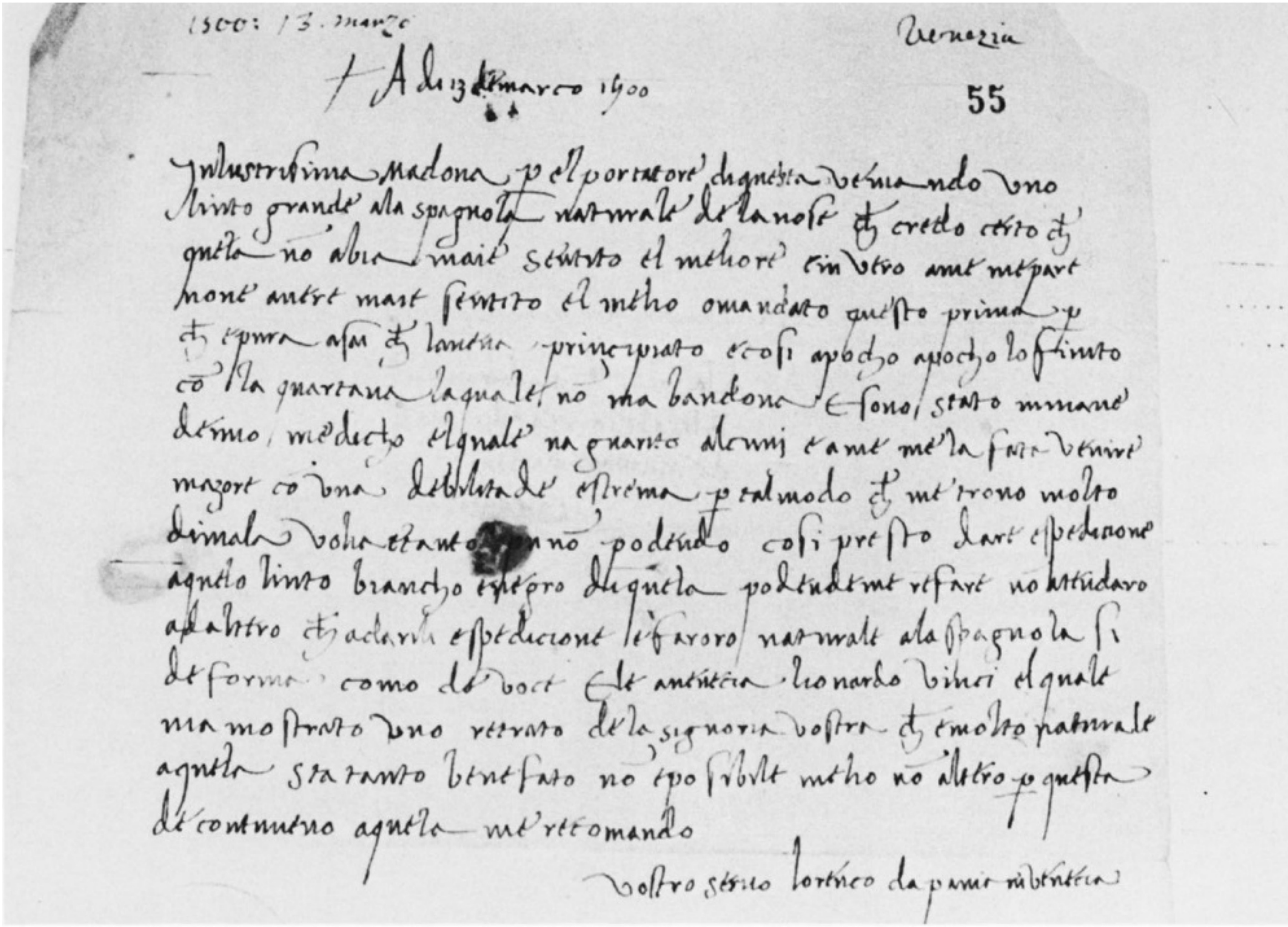
Organ by Lorenzo Gusnasco (1494)

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



1500: 13. marzo — Venezia

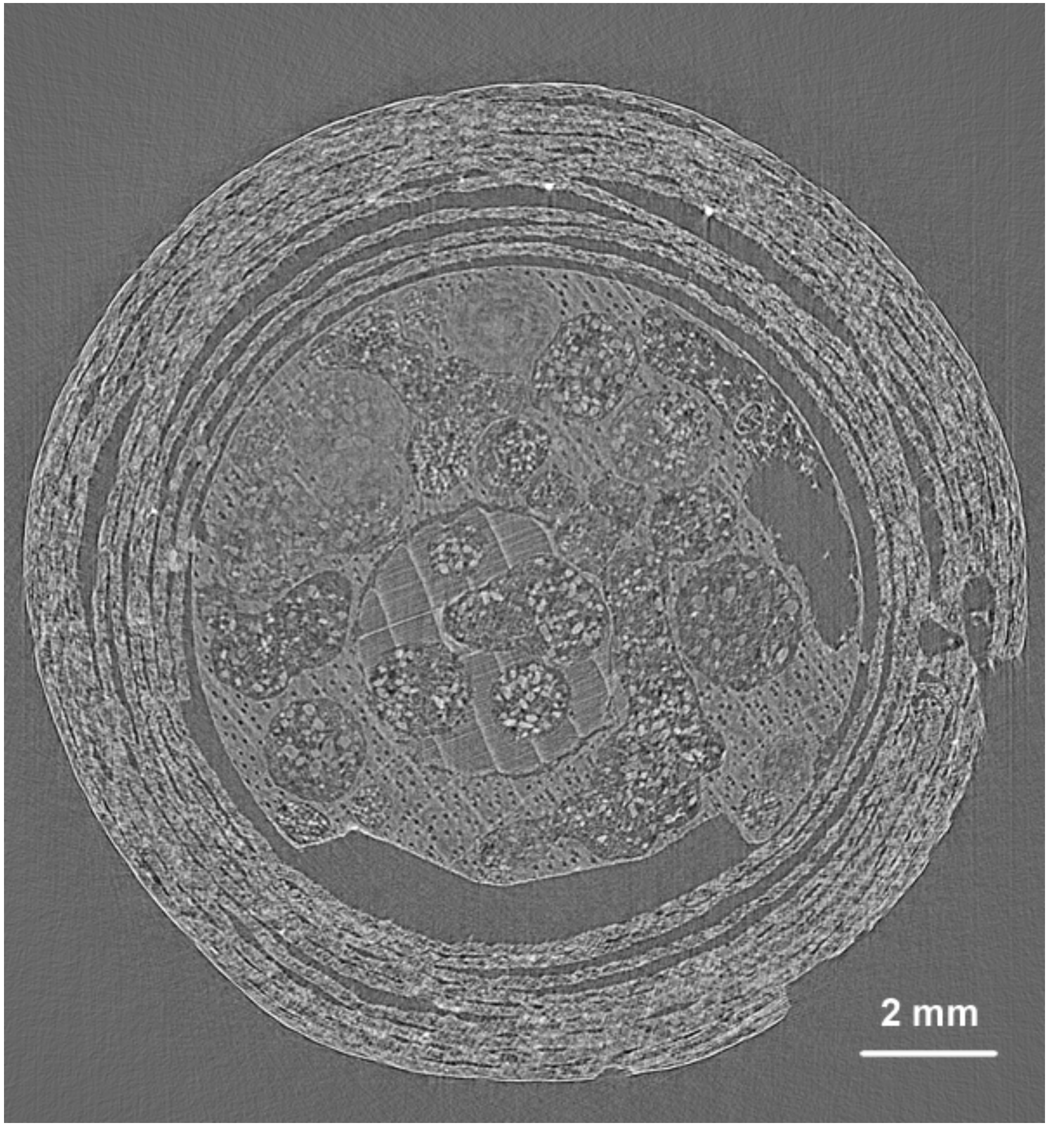
+ Adi 13 de marzo 1500 — 55

Illustrissima Madona p el portatore di questa ve mando uno
liuto grande a la spagnola naturale de la noce che credo certo che
quella no abia mai sentito el meliore in vero a me me pare
non e avere mai sentito el melio o mandato questo prima per
che e stato asai che l'ancora principiato e cosi a pocho a pocho lo finito
co la quartana la quale no ma bandona e sono stato in mane
de uno medicho el quale na guarito alcuni e a me me la fata venire
magiore co una debilitade estrema per tal modo che me trovo molto
di mala voglia e tanto [illegible] non podendo cosi presto dare expedicione
a quello liuto biancho e per questo di quella [illegible] me refare no atendaro
ad altro che a darli expedicione lo faro naturale a la spagnola si
de forma como de voce. E le a Venetia Lionardo Vinci el quale
ma mostrato uno retrato de la signoria vostra che e molto naturale
a quella sta tanto bene fato no e posibile melio no altro per questa
de continuo a quella me recomando

vostro servo Lorencho da pavia in Venetia

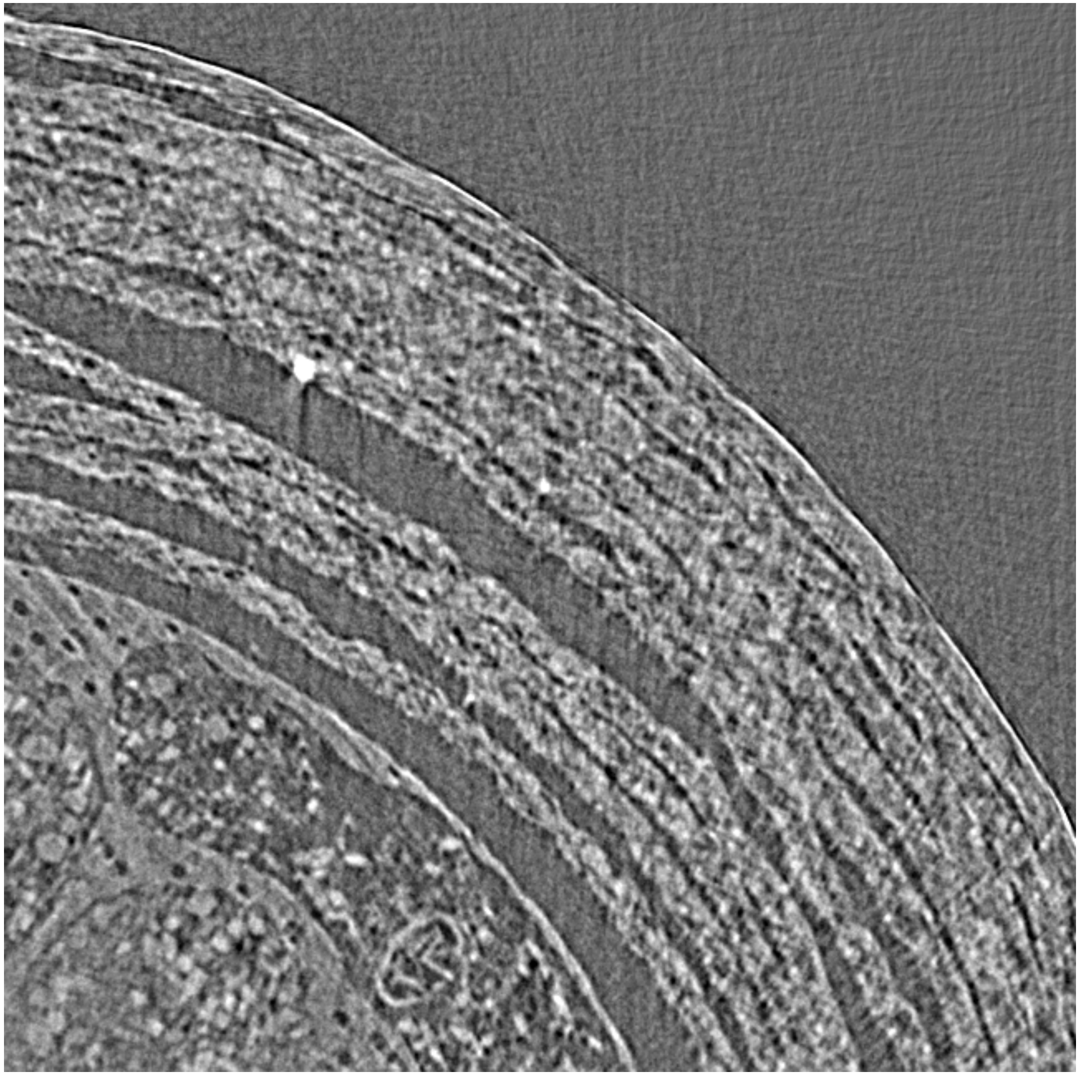
Correspondence between Lorenzo Gusnasco and Isabella d'Este

The organ by Lorenzo da Pavia



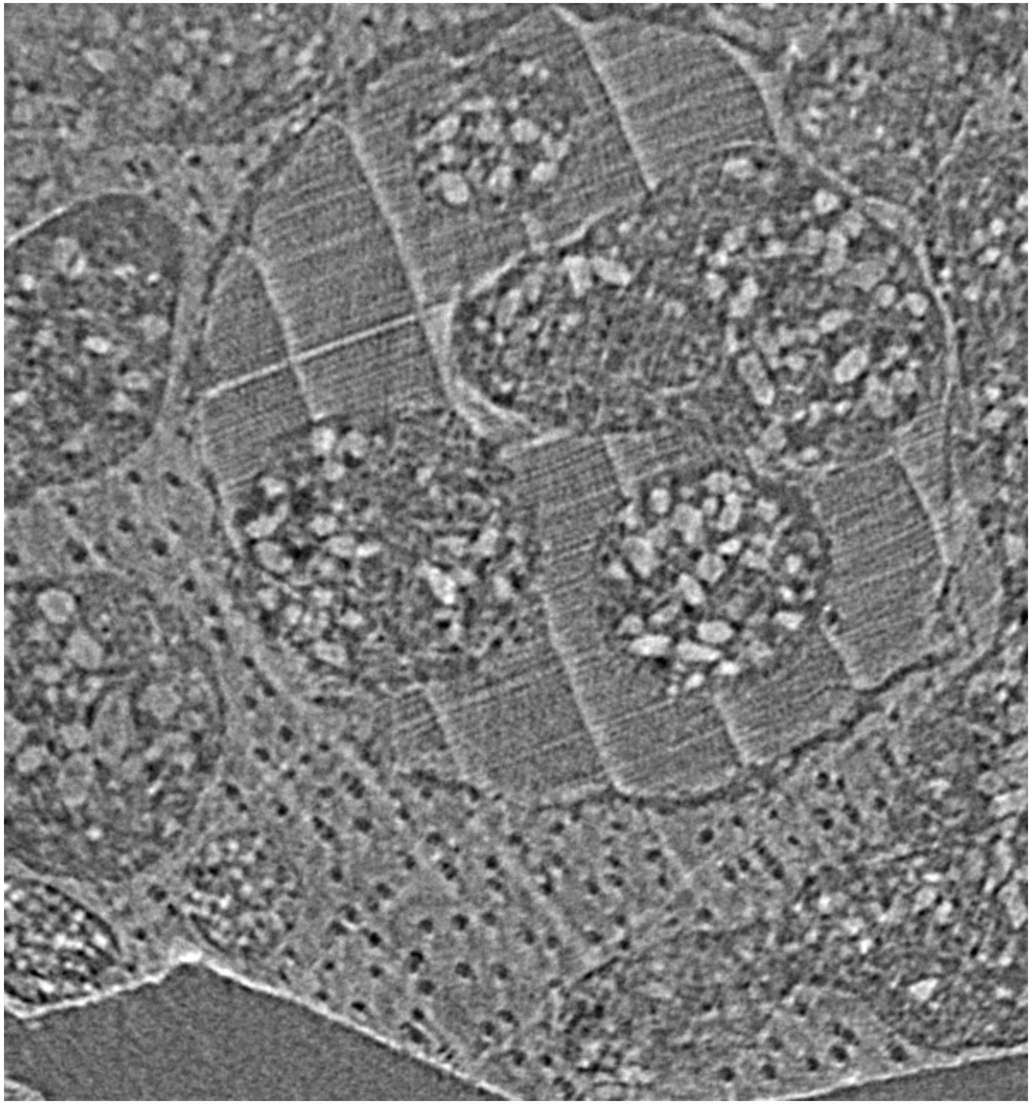
Slice at the wood foot position

The organ by Lorenzo da Pavia



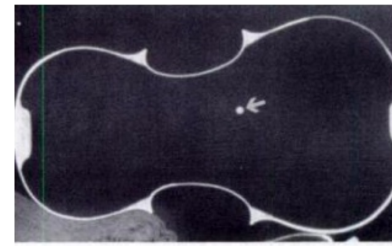
- Ten 0.25 mm layers
- Good quality of the external layer
- Evident degradation of the layers adhesion

The organ by Lorenzo da Pavia

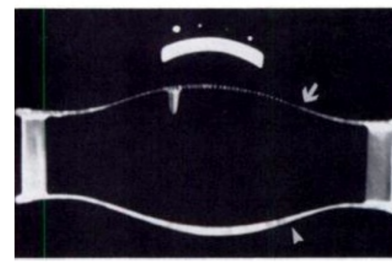


- Possibility of wood species characterization
- Presence of larvae

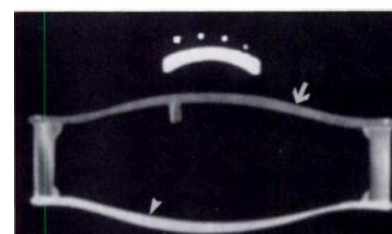
Computed tomography (CT) is a unique tool for characterization of bowed stringed instruments.
Sirr and Waddle are the authors of the first works where clinical CT has been applied to the study of violins. Internal damage or repair invisible at visual inspection were detected in historical instruments.



3.



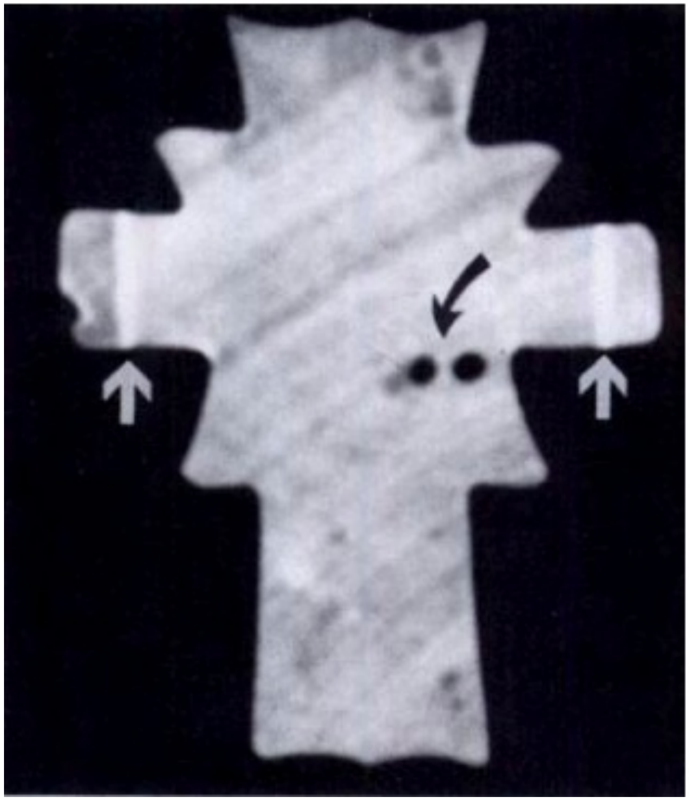
4a.



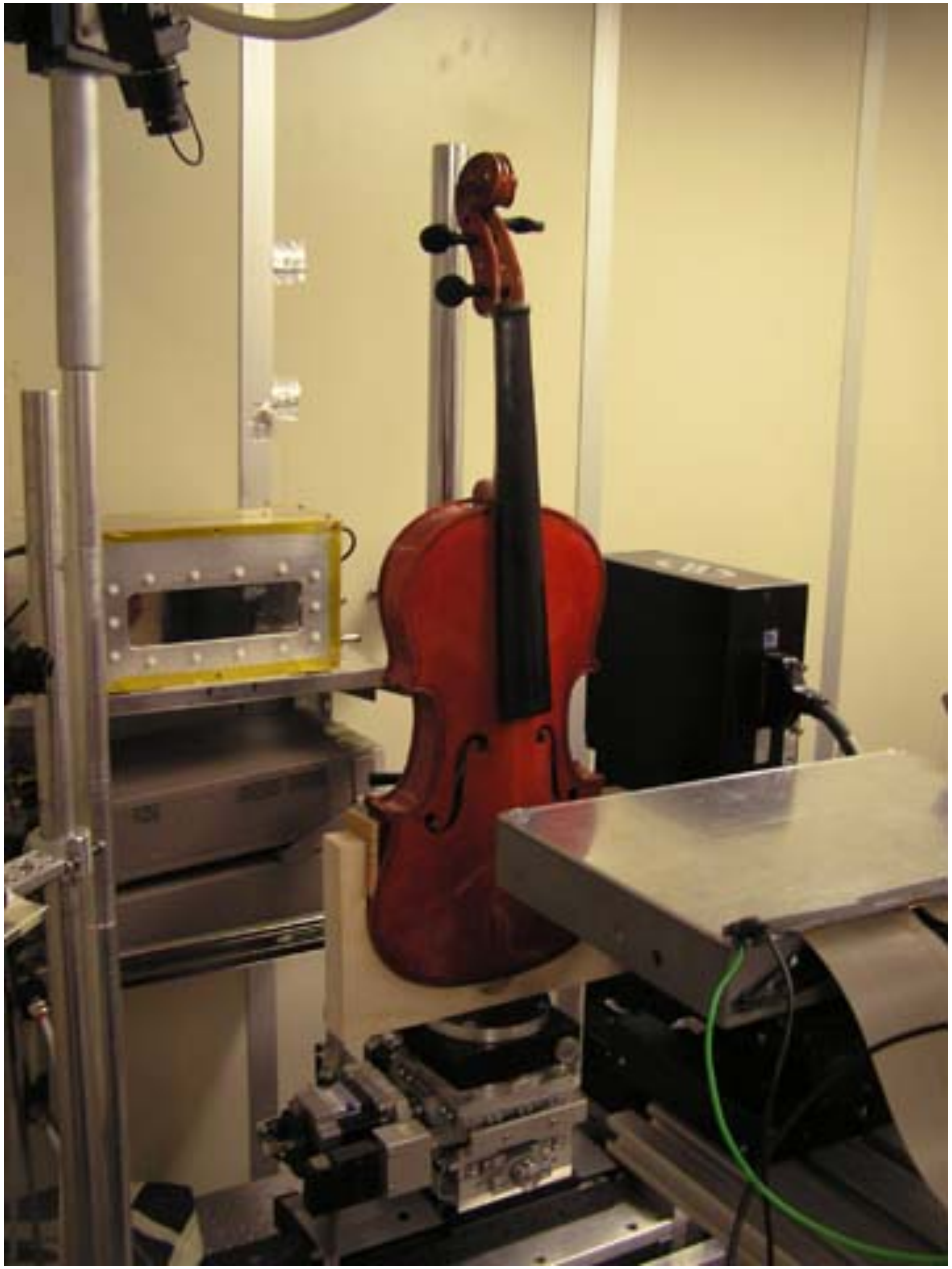
4b.

S.A. Sirr, J.R. Waddle, Radiology, 1997, **203**, 801

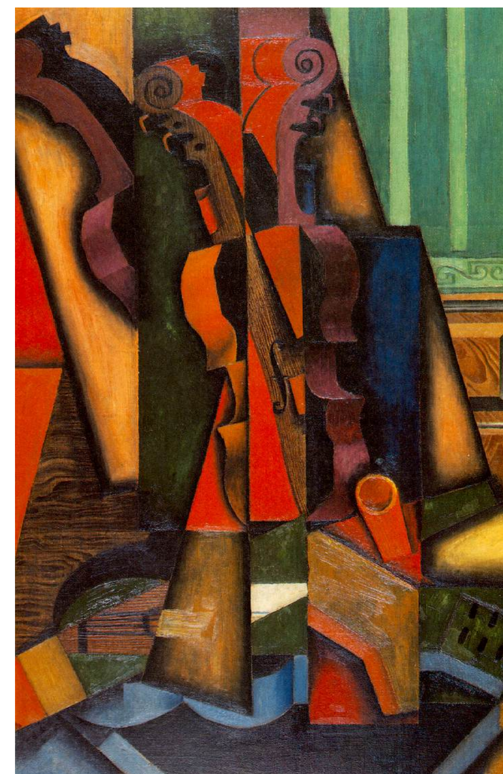
The main limitation in the application of clinical CT to the structural analysis of bowed instruments, however, is the **limited spatial resolution** of commercial instruments, ($0.4 \times 0.6 \times 0.6\ mm^3$). Every defect with lateral dimensions smaller than this value **cannot be detected** with state-of-the-art hospital instruments.

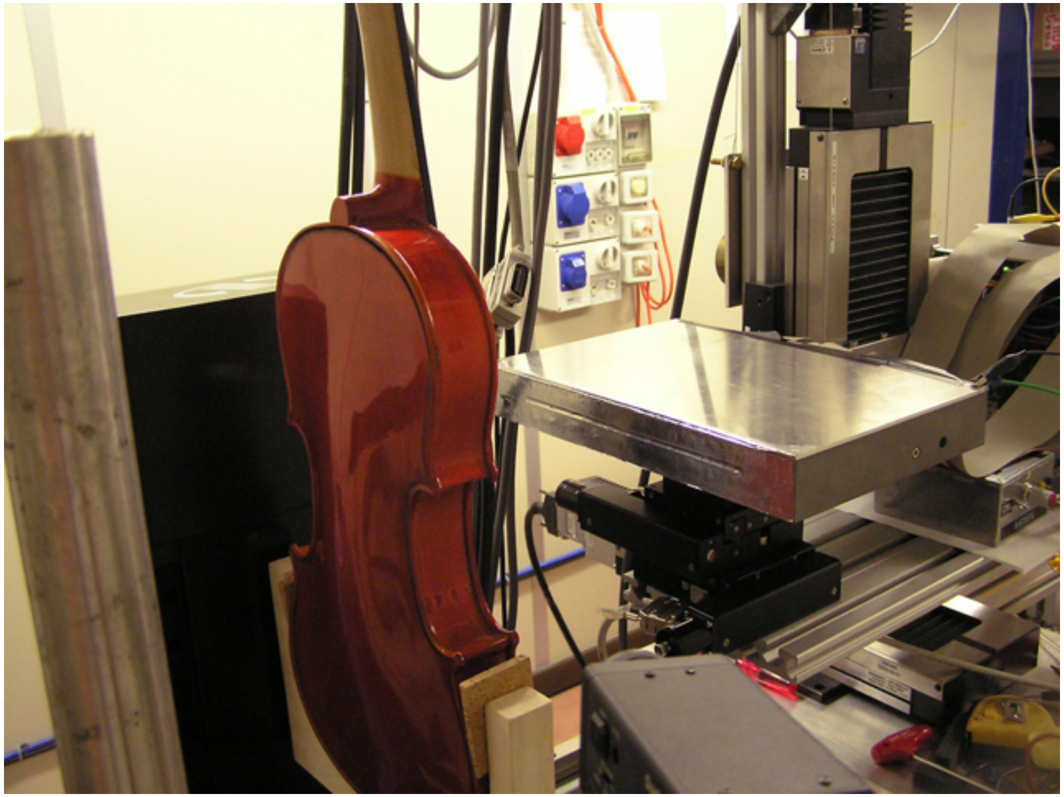


S.A. Sirr, J.R. Waddle, Radiology, 1997, **203**, 801



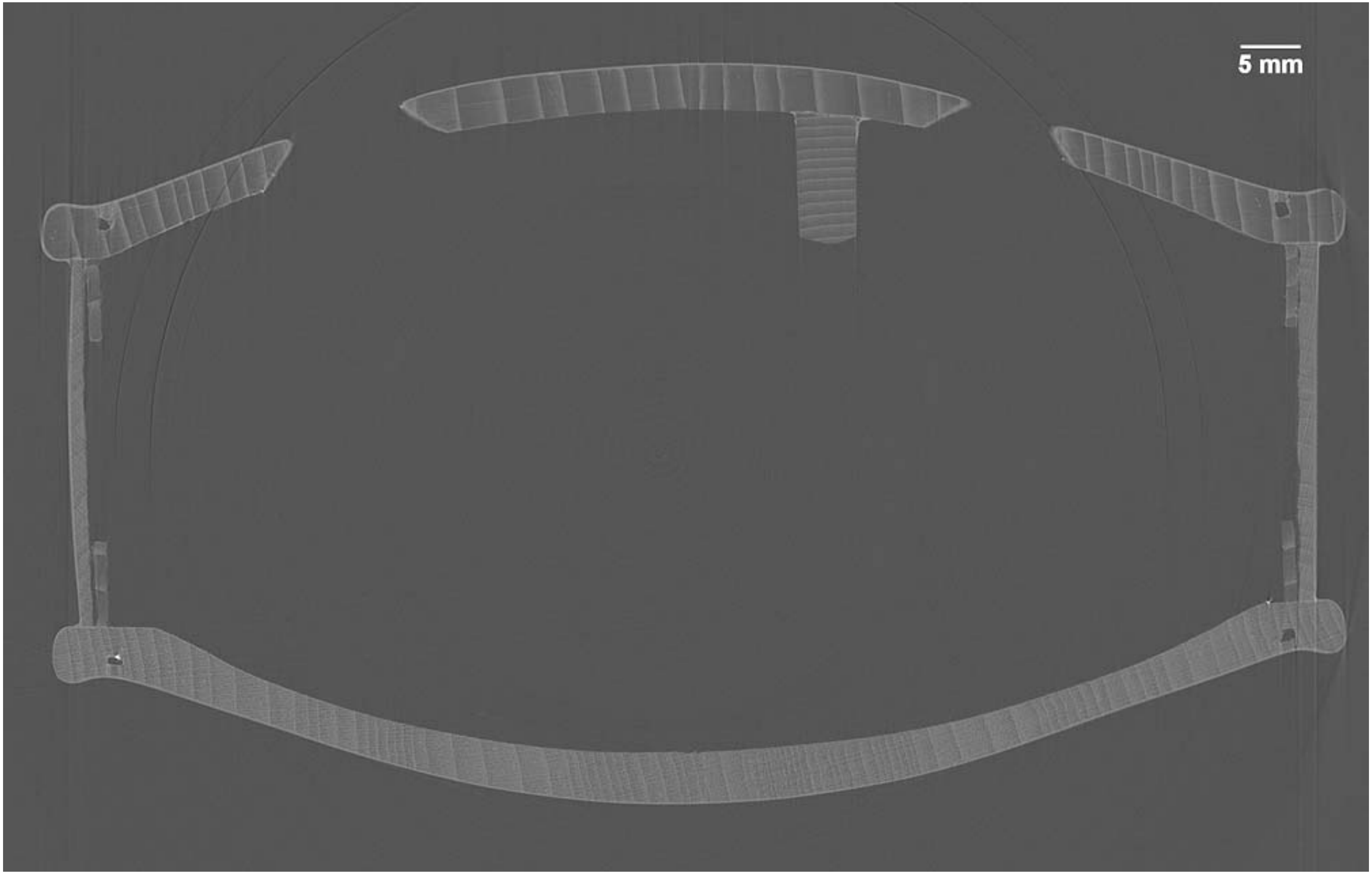
The main limitation in the use of synchrotron radiation is related to the reduced dimensions of the samples under investigation. The development of new X-ray detectors designed for the particular characteristics allows the researchers to overcome this kind of problems.



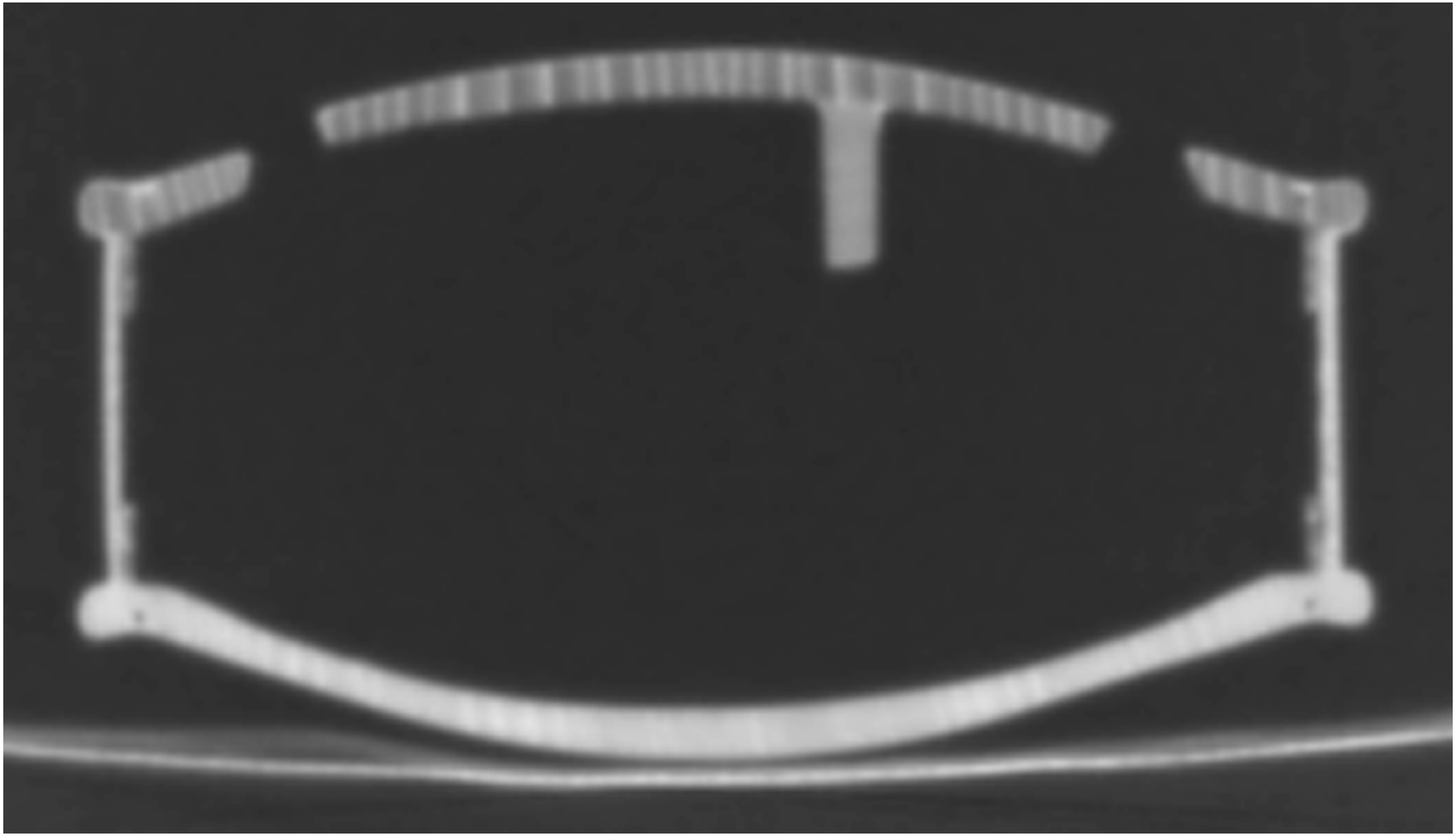


PICASSO (Phase Imaging for Clinical Application with Silicon detector and Synchrotron radiation) has been developed by the Istituto Nazionale di Fisica Nucleare (INFN). It is a silicon microstrip detector in “edge-on” configuration.

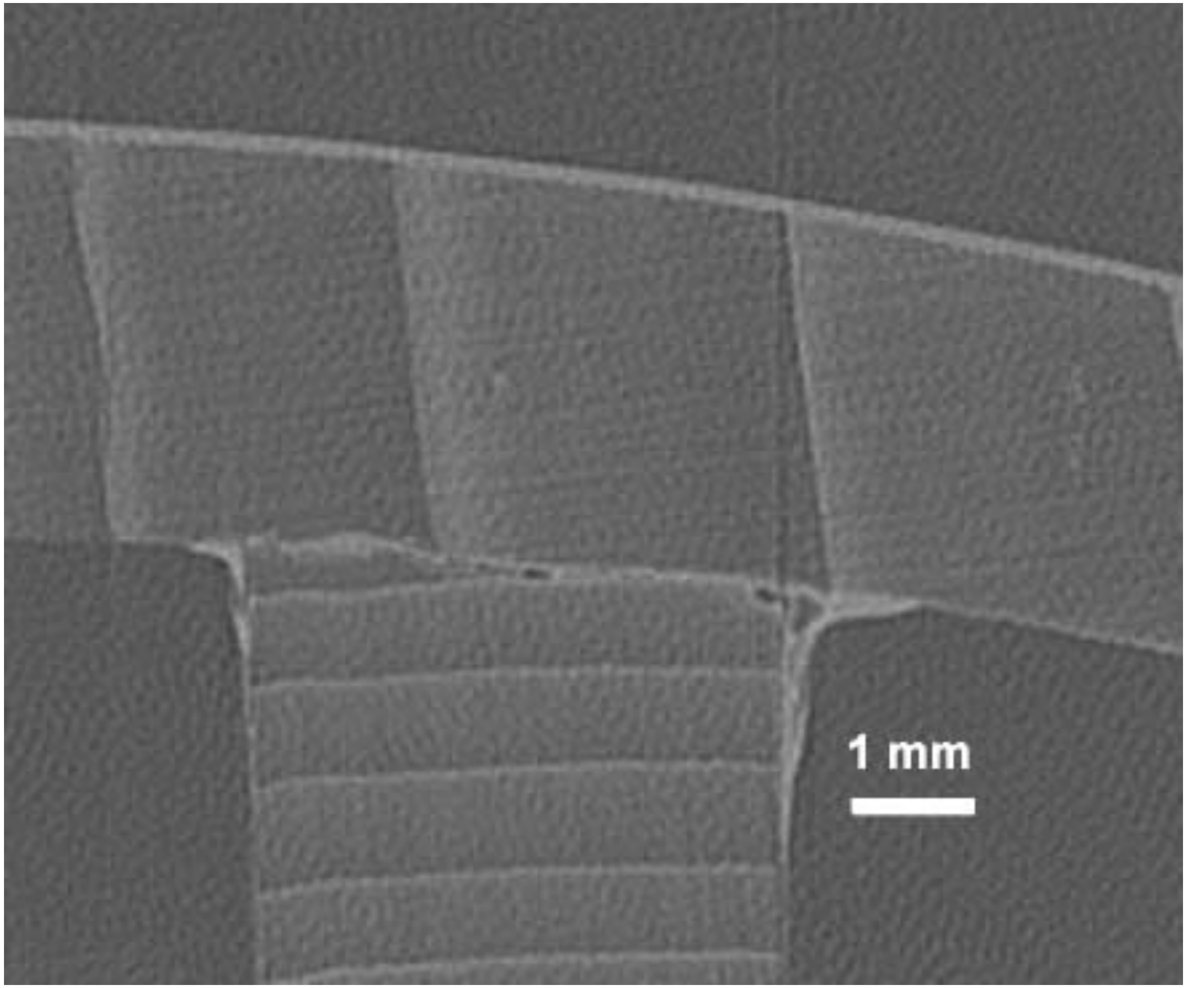
The aperture of each pixel is determined by the strip pitch (0.05 mm, H) and the sensor thickness (0.3 mm, V). The detector is operated in single-photon counting and it is read out by a high-rate electronics based on the Mythen-II application-specific integrated circuit (ASIC). Each pixel is wire-bonded 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)



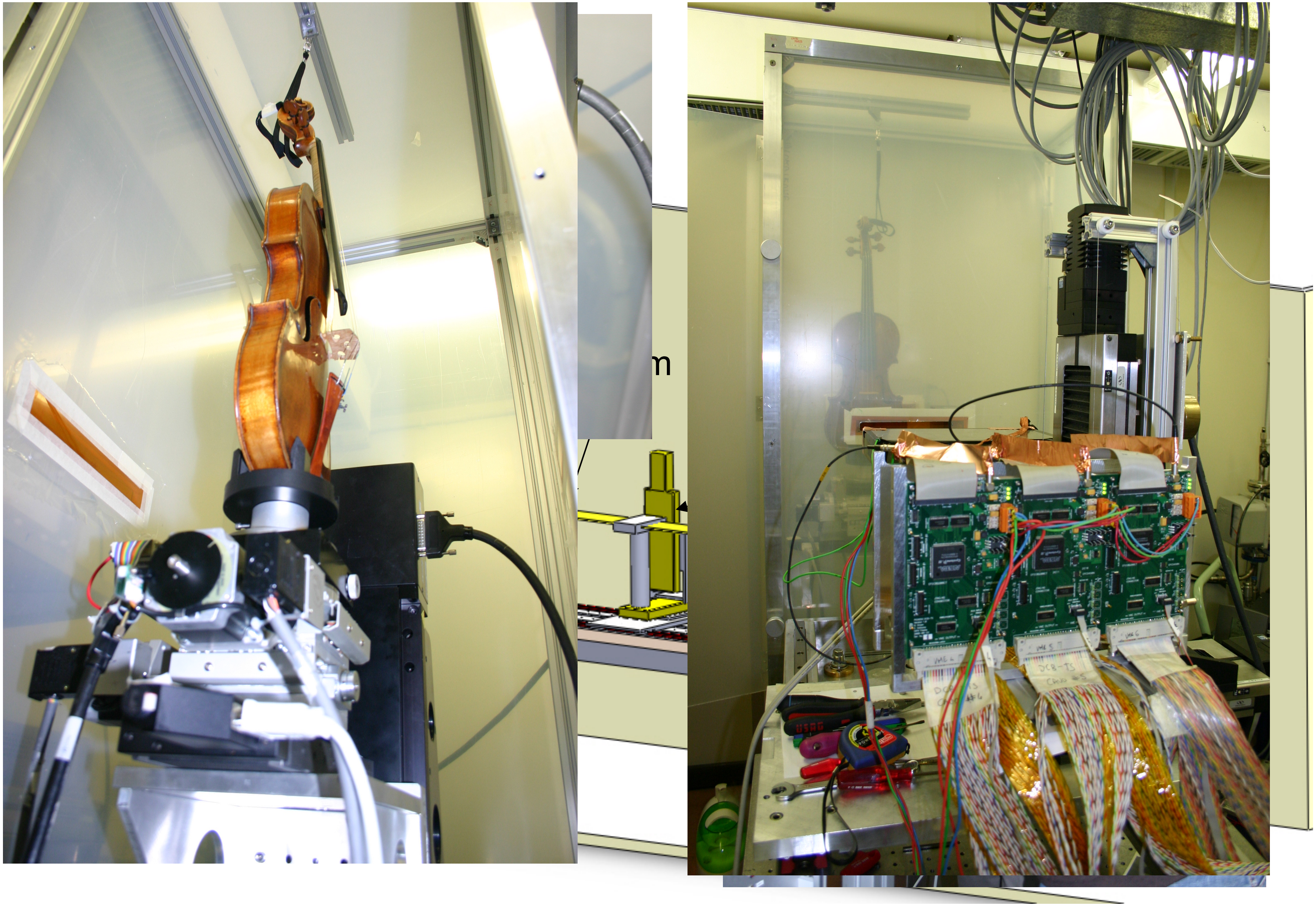
Transaxial CT taken with a state-of-the-art clinical instrument
(Toshiba Aquilion, helical scan 120 kVp, 512x512 matrix, 0.5 mm slice thickness,
0.5 s exposure time, 0.485/0.485 pixel spacing, Torax protocol)



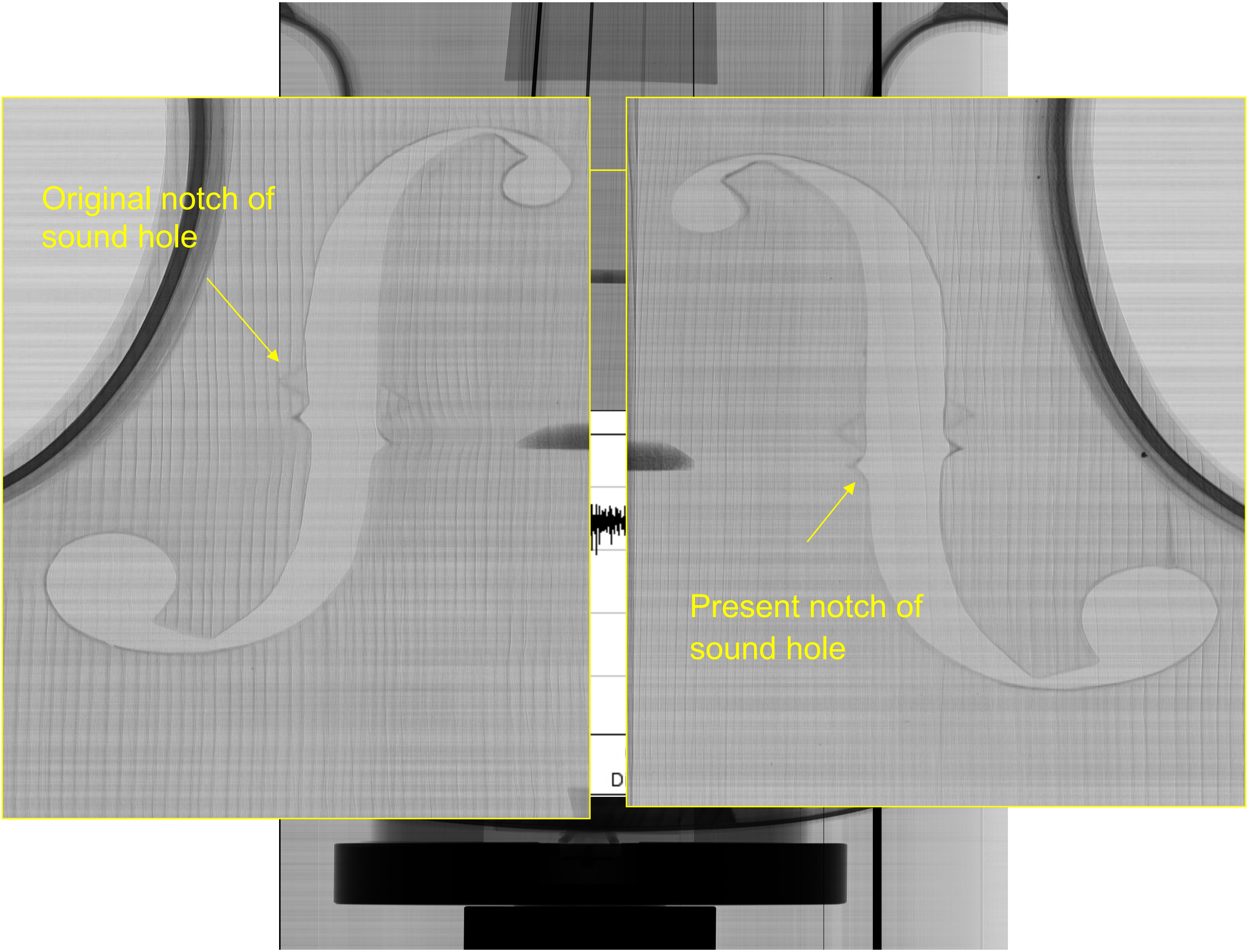
Detail showing the bass bar and the glue used to attach it to the front plate.

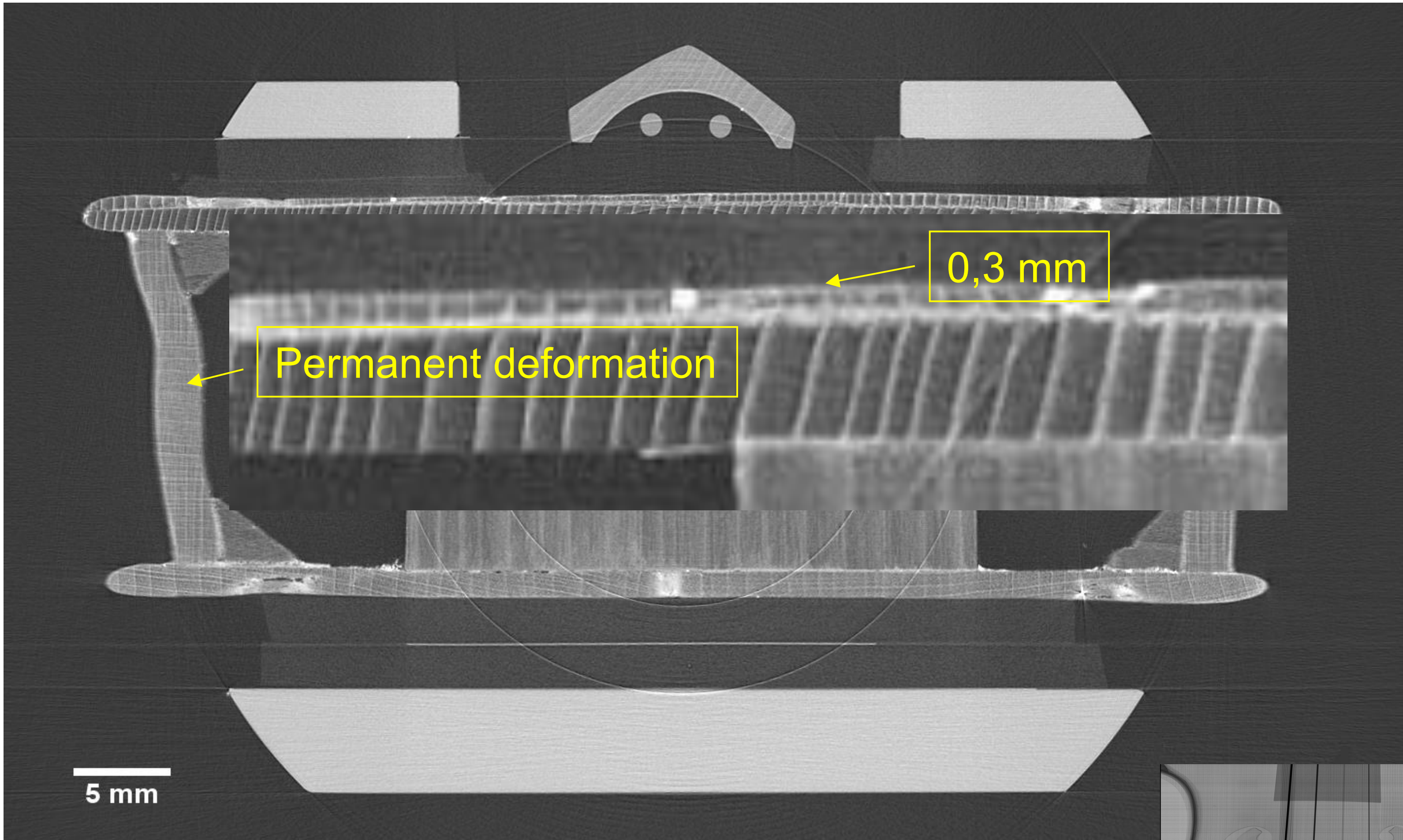
Peter Herresthal and his Giovanni Battista Guadagnini (1753)

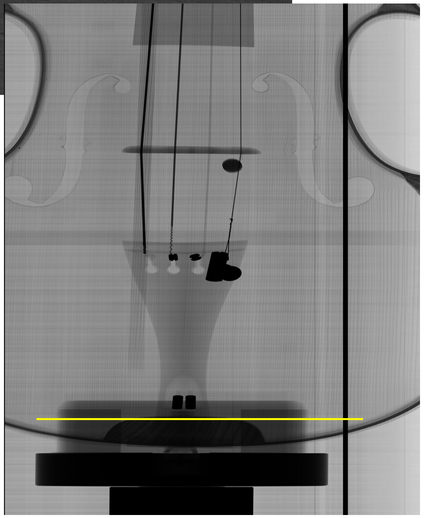
The experimental hutch



The planar image



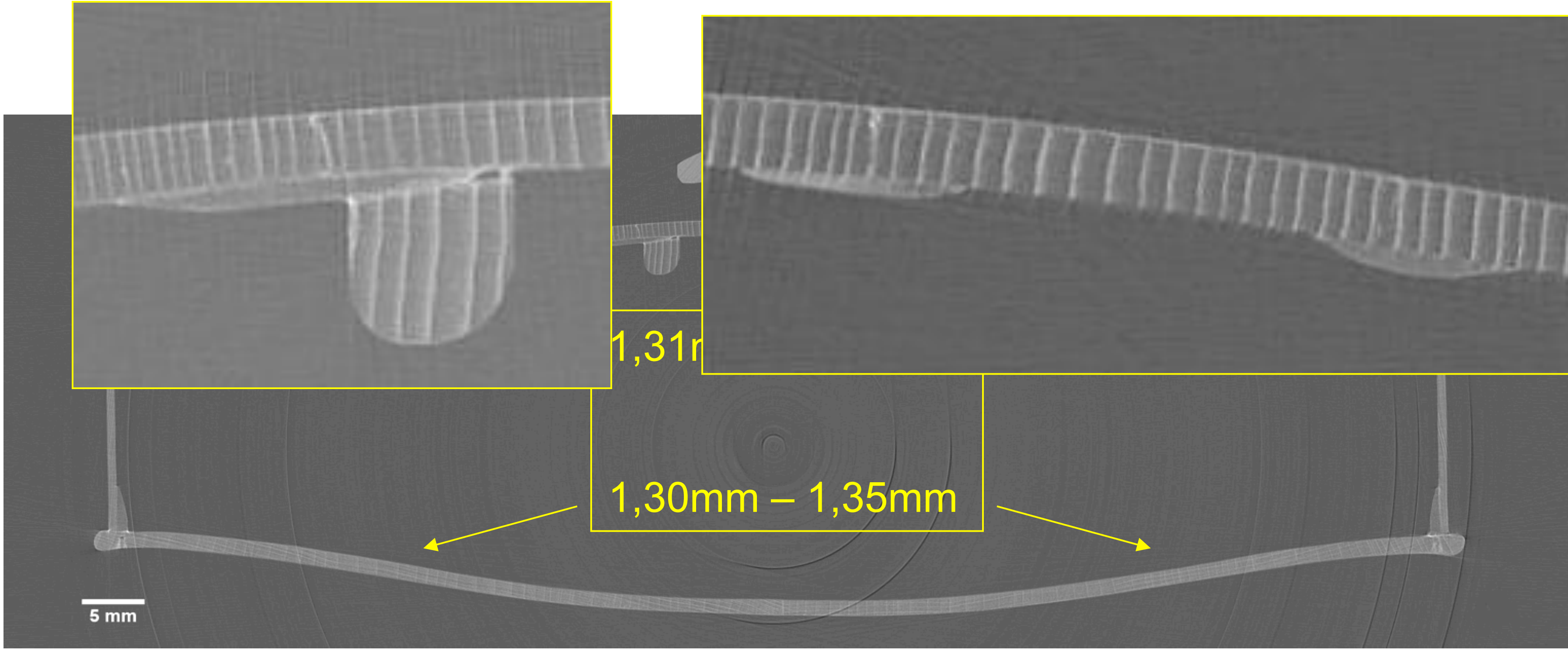


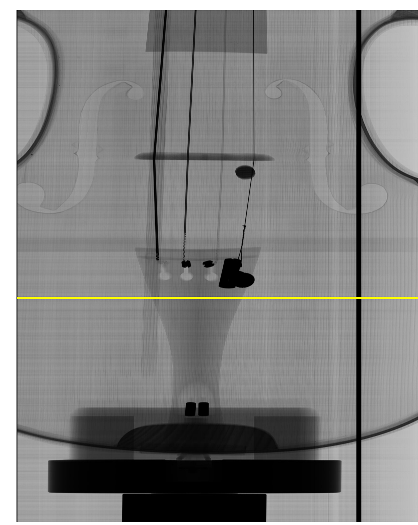


- The original top plate is very thin, expecially on the left side;

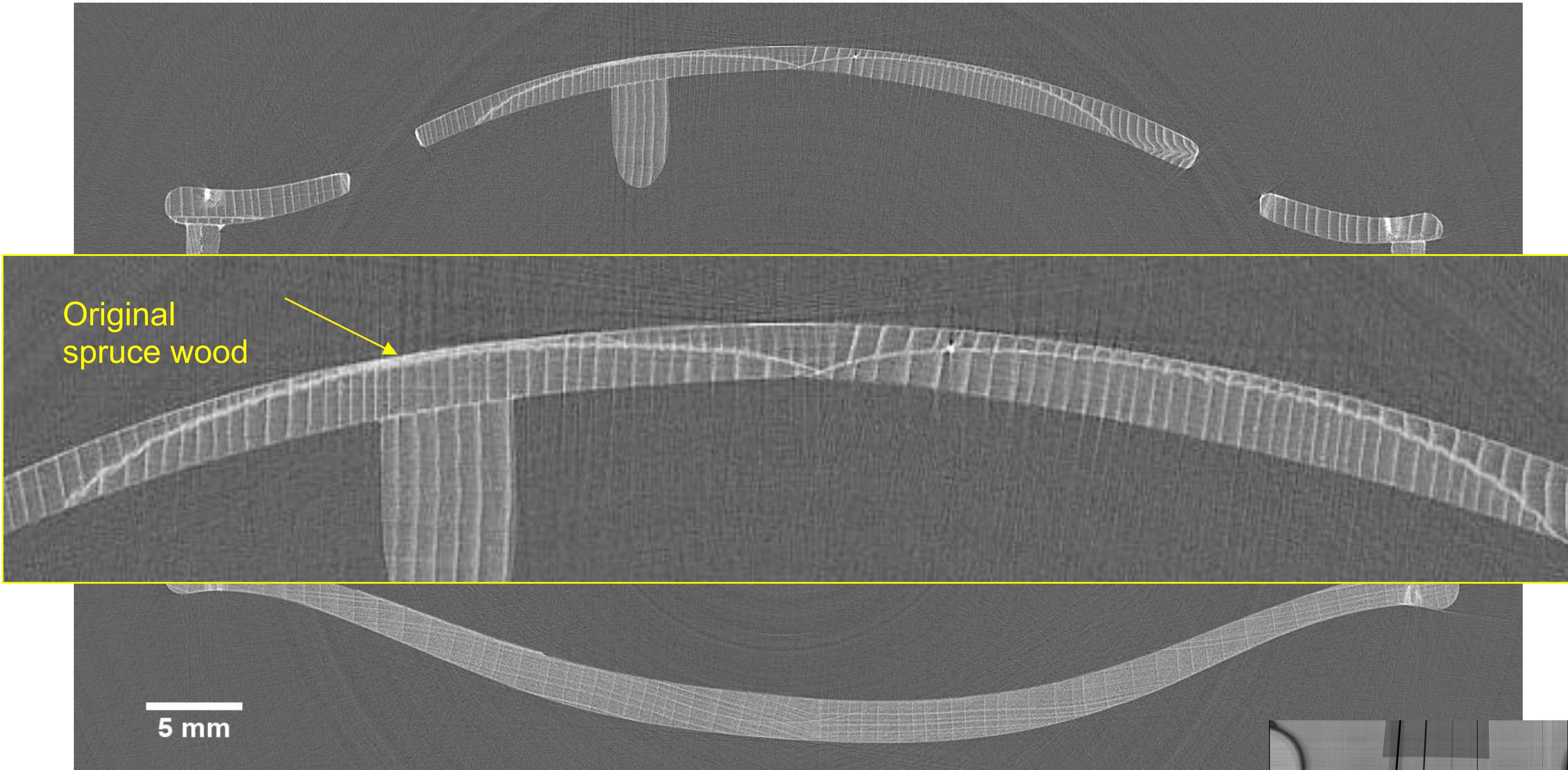
-The grain of the patch below the top plate is not straight (probably obtained by cutting and not by splitting);

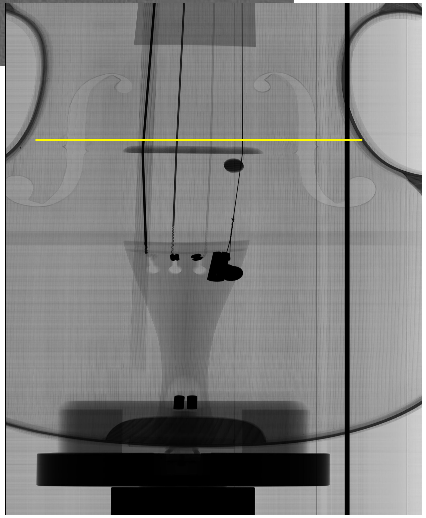
- Warped shape of left rib due to the chin rest position.



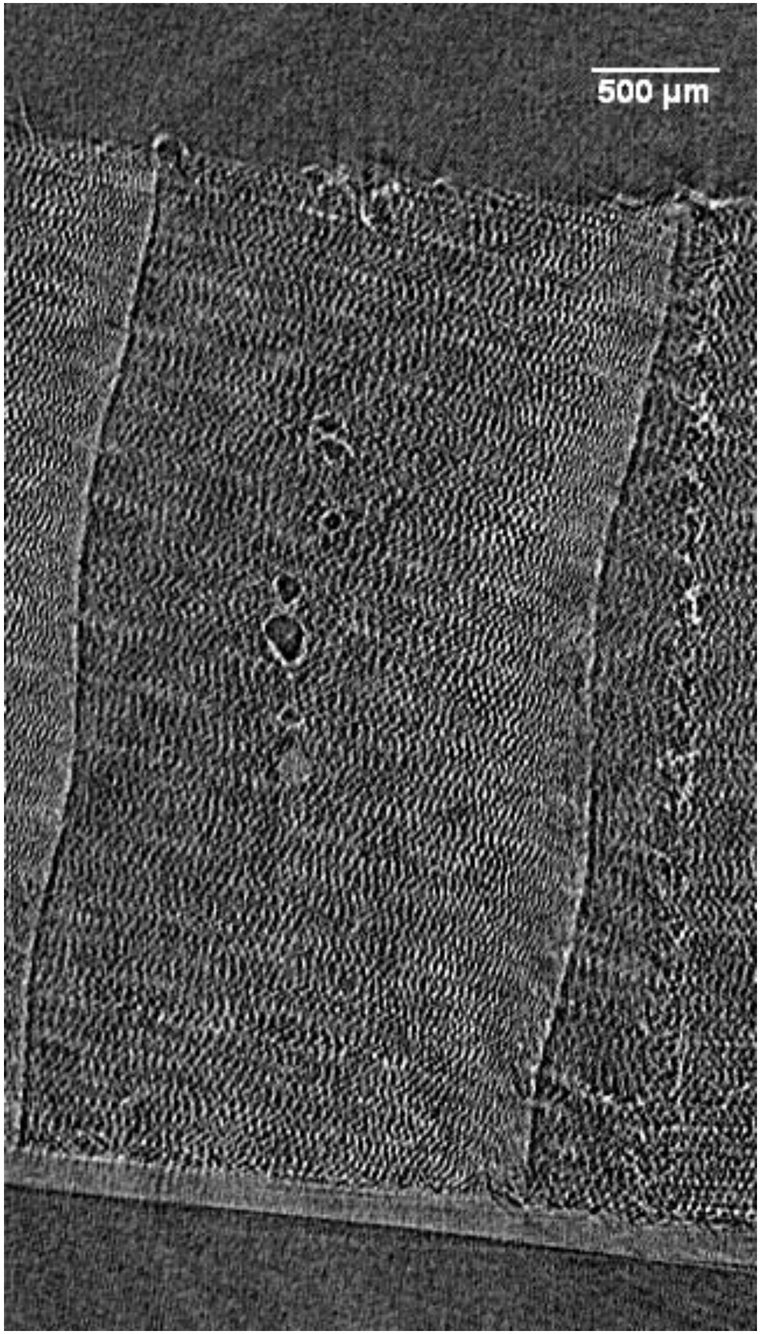


- Thickness of top and back plate
- Bass bar is glued on a patch;
- Two patches on the right part of top plate.





- 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*

archaeometry

Archaeometry ••, •• (2011) ••–••

doi: 10.1111/j.1475-4754.2011.00630.x

DID NEANDERTHALS PLAY MUSIC? X-RAY COMPUTED MICRO-TOMOGRAPHY OF THE DIVJE BABE 'FLUTE'*

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Archaeological evidence for wind musical instruments made by modern humans has been well established from the Upper Palaeolithic in Europe. Musical instruments evidently made by Neanderthals have not been found so far. The most controversial object is a juvenile cave bear femur with two complete holes, found in 1995 in the Middle Palaeolithic layers of the Cave Divje babe I, Slovenia. The bone was interpreted as a possible Neanderthal 'flute', but some scholars have firmly rejected this hypothesis on the basis of taphonomic observations, suggesting a carnivore origin for the holes. Here, we show the results of X-ray computed micro-tomography (mCT) performed on the Divje babe I 'flute'. Our analyses demonstrate that there were originally four holes, possibly made with pointed stones and bone tools. Most surface modifications near the holes, previously interpreted as effects of carnivore gnawing, are post-depositional marks. Furthermore, a thin layer has been removed around one of the complete holes, producing a flat surface, possibly to facilitate perforation. The new data show that a Neanderthal manufacture of the object cannot be ruled out.

KEYWORDS: MICRO-COMPUTED TOMOGRAPHY, DIVJE BABE I 'FLUTE', NEANDERTHAL

INTRODUCTION

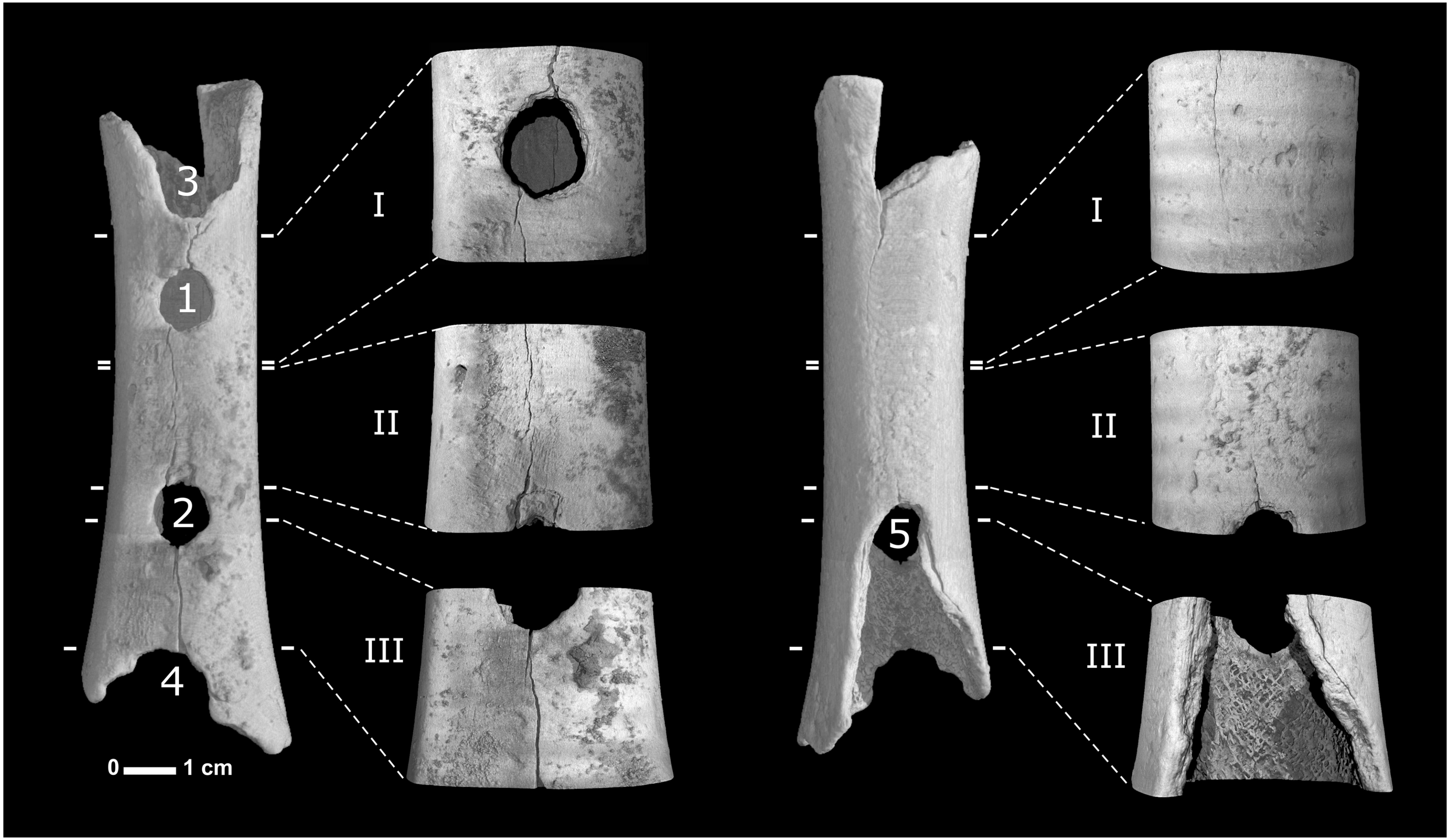
Music is a characteristic expression of modern human culture (Jerison 2000), but there are no anatomical or other biological barriers (Frayer and Nicolay 2000) to its presence in the Neanderthal cultural package, which included the symbolic use of marine shells and mineral pigments (Zilhão *et al*. 2010) and, possibly, language (Krause *et al*. 2007).

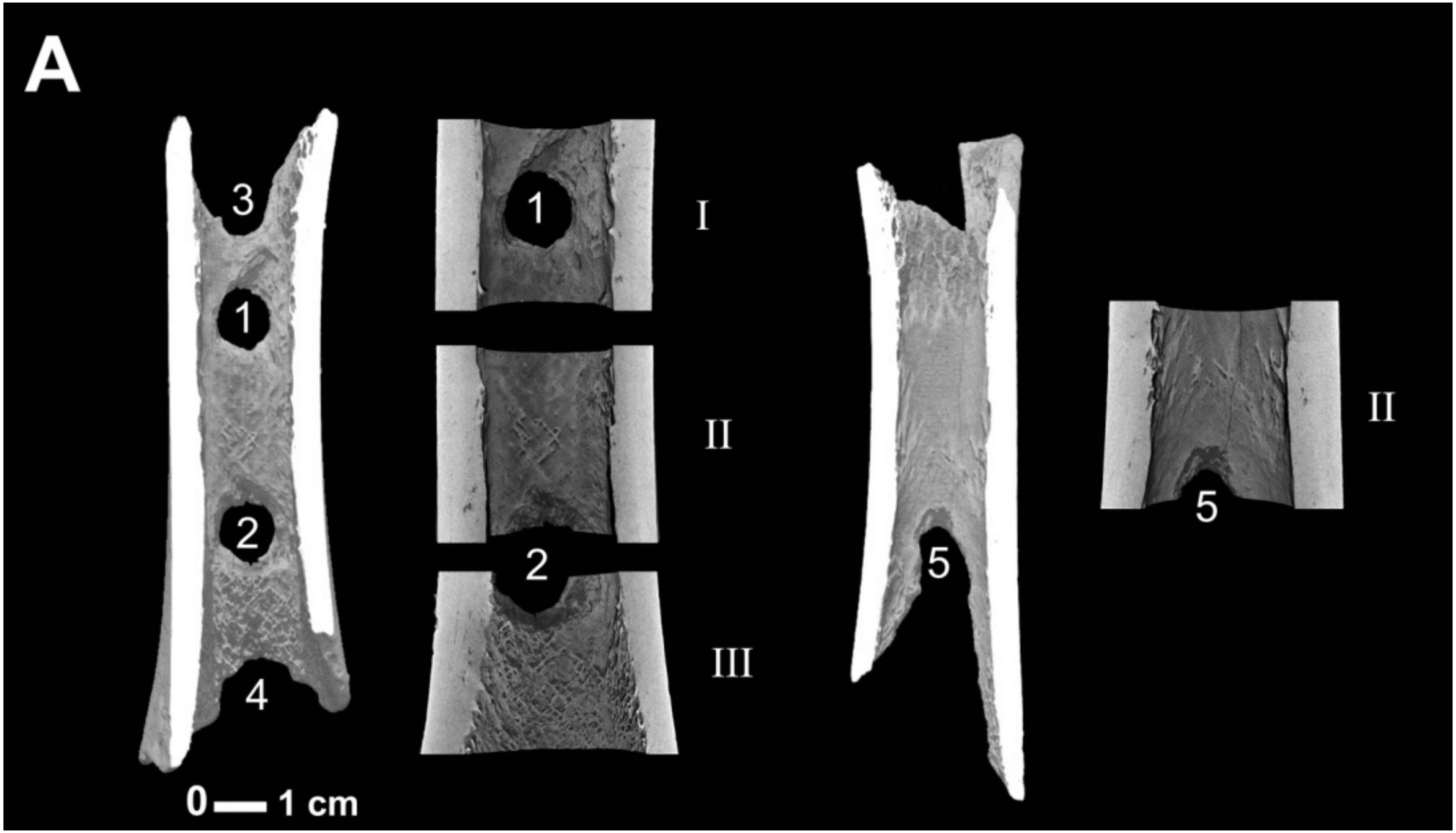
The oldest musical instruments are bone and ivory flutes with two to five finger holes. More than 120 specimens have been found throughout Europe in Upper Palaeolithic sites linked to modern humans. The most famous are from the Isturitz Cave in France and some German caves, including Vogelherd, Geissenklösterle and Hohle Fels (Conard *et al*. 2009). Flutes from these three German caves are Aurignacian and are considered to be the oldest Upper Palaeolithic musical instruments in Europe (about 36 ka). Similar finds from the Gravettian and the Magdalenian are mostly made on bird bones, but there are other examples fashioned from mammal bones (e.g., Pas du Miroir, France; Grubgraben, Austria) (Einwögerer and Käfer 1998; Atema 2004).

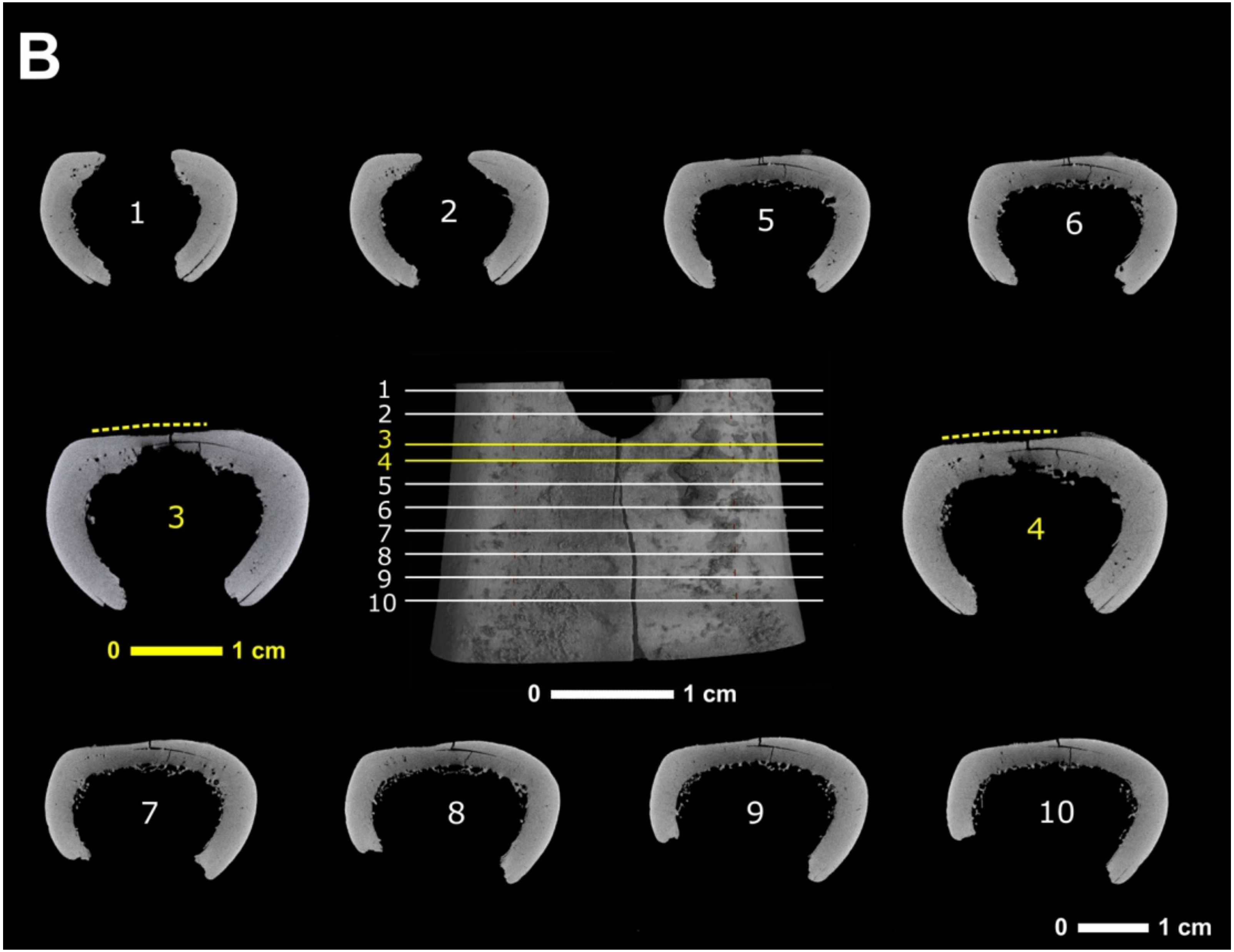
Putative musical instruments from cave sites in Eastern Europe, ranging from the Mousterian to the Aurignacian (Horusitzky 2003), consist of perforated limb bones of cave bears. Other

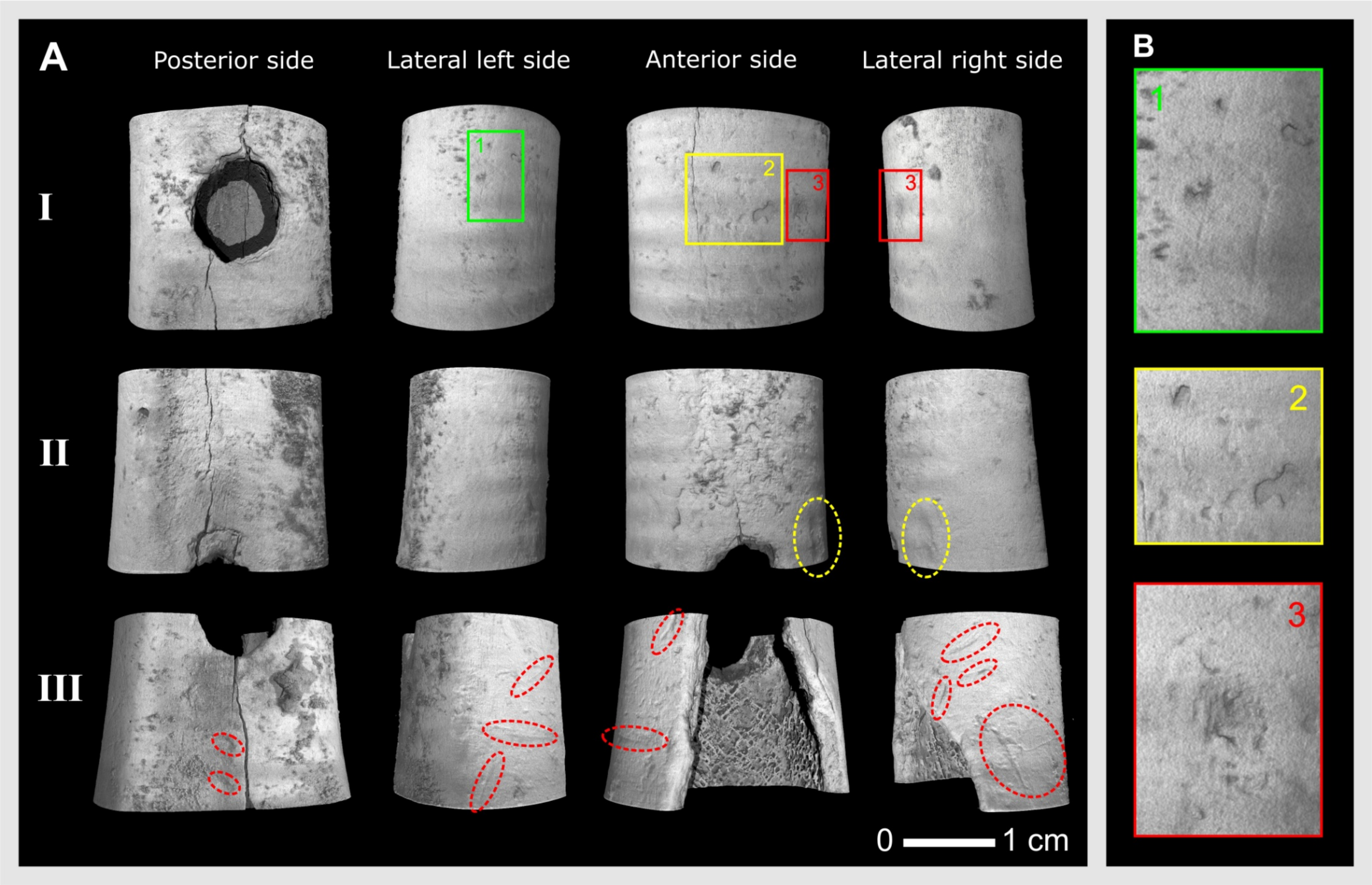
*Received 5 November 2010; accepted 23 June 2011

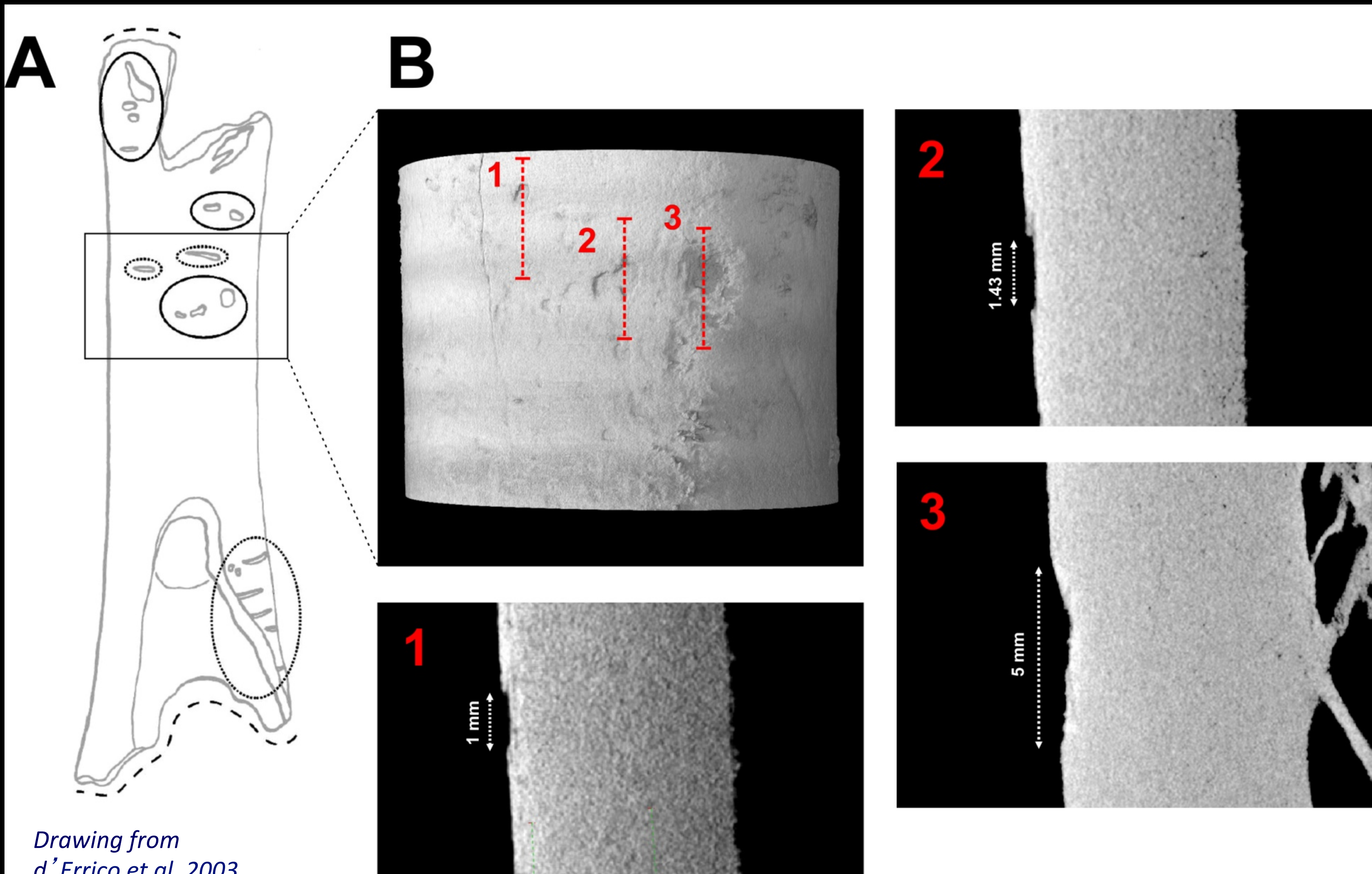
In collaboration with National Museum of Slovenia, Ljubljana


3
1
2
4
I
II
III
0 1 cm
5
I
II
III


A
3
1
2
4
0 1 cm
1
I
II
2
III
5
II
5


B
1
2
5
6
1
2
3
4
5
6
7
8
9
10
3
4
0 1 cm
0 1 cm
7
8
9
10
0 1 cm


A
Posterior side
Lateral left side
Anterior side
Lateral right side
I
II
III
1
2
3
3
0
1 cm
B
1
2
3



Drawing from d'Errico et al. 2003

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".

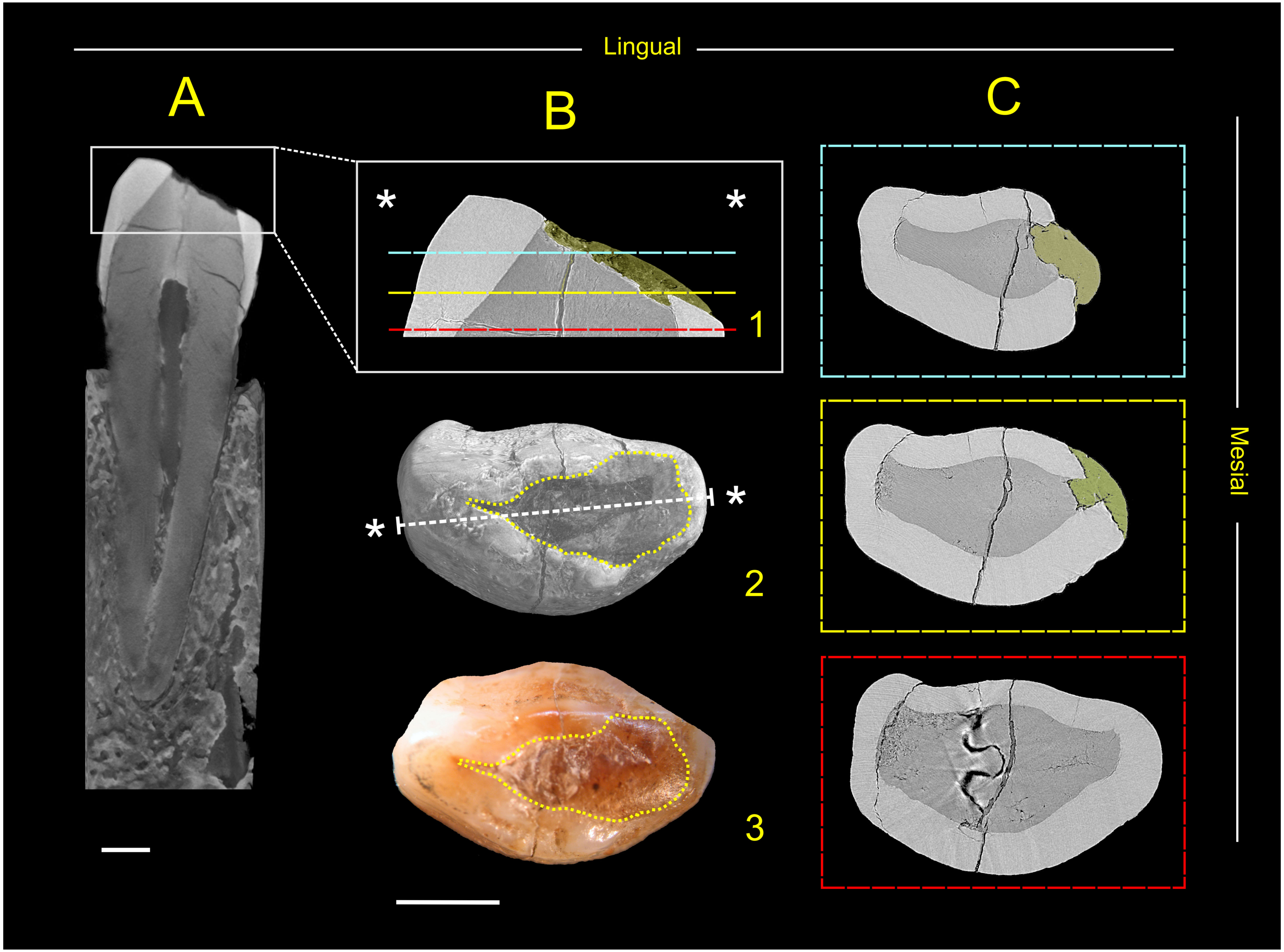
The Lonche *mandible*

Upper Pleistocene, about 6500 years old, Slovenia

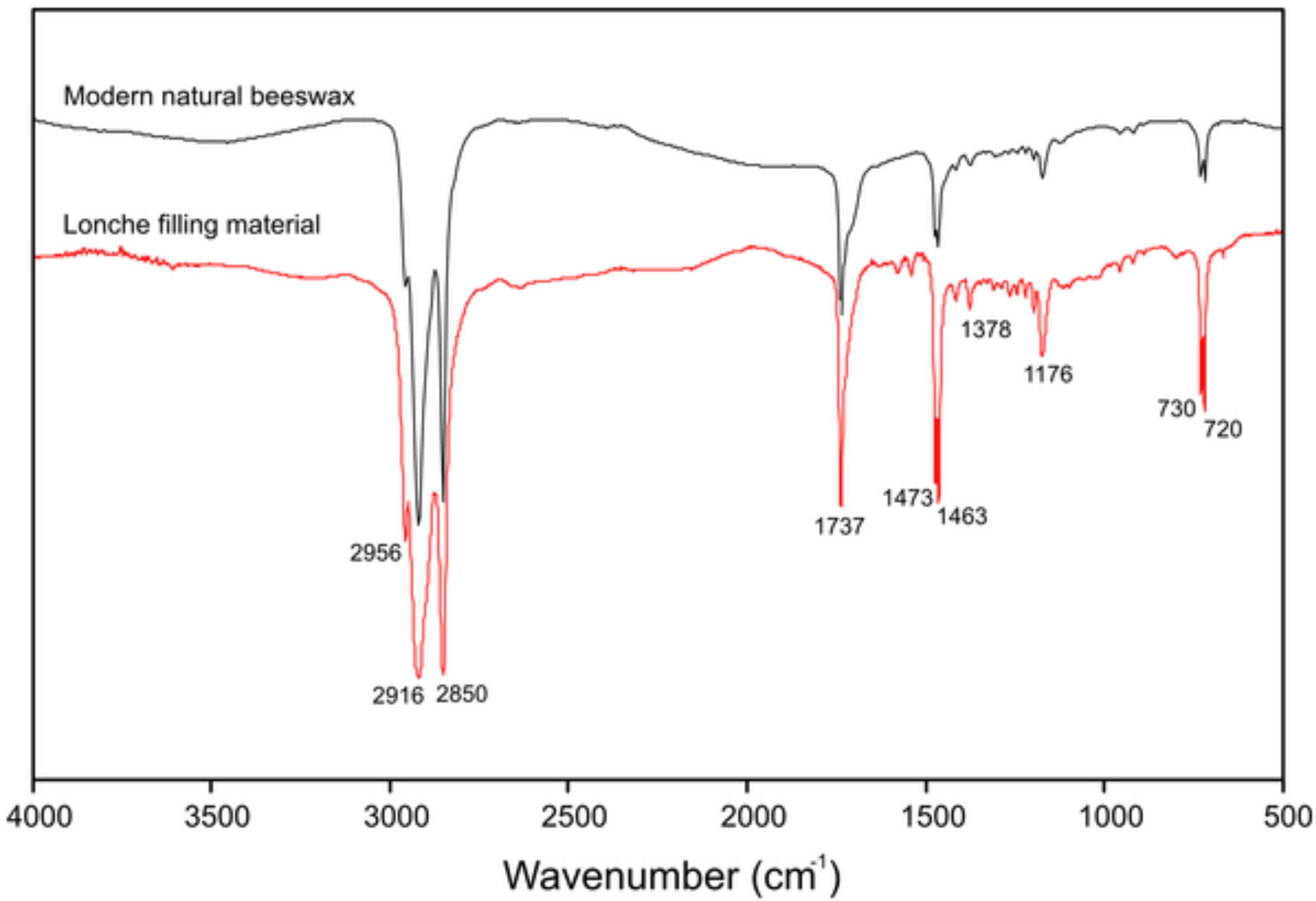
Left canine shows presence of beeswax Inside a vertical crack

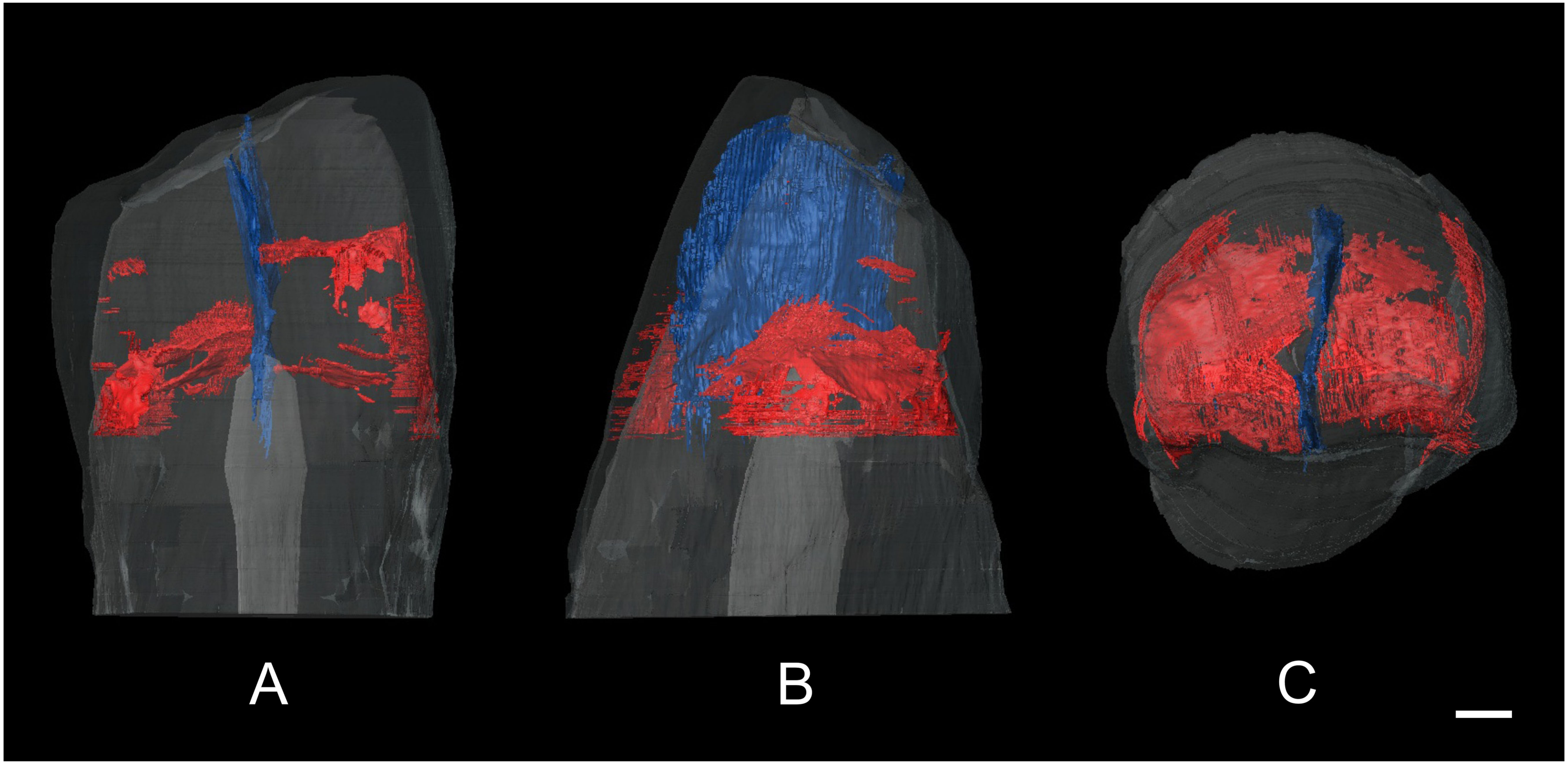
Earliest known evidence of therapeutic dental filling

In collaboration with the Natural History Museum of Trieste



Lingual
A
B
C
*
*
1
*
*
2
3
Mesial


Modern natural beeswax
Lonche filling material
2956
2916
2850
1737
1473
1463
1378
1176
730
720
4000
3500
3000
2500
2000
1500
1000
500
Wavenumber (cm-1)


A
B
C

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