The Abdus Salam
International Centre
for Theoretical Physics

2455-7

Joint ICTP-TWAS Workshop on Portable X-ray Analytical Instruments for Cultural Heritage

29 April - 3 May, 2013

Synchrotron hard X-ray imaging: applications to cultural heritage

Franco Zanini
*Elettra, Trieste
Italy*

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Franco Zanini

Elettra - Sincrotrone Trieste

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: $\Delta\lambda/\lambda \cong 2 \times 10^{-3}$

SR X-ray imaging studies in cultural heritage

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

→ archeaological findings and ancient artifacts identification

→ restauration techniques

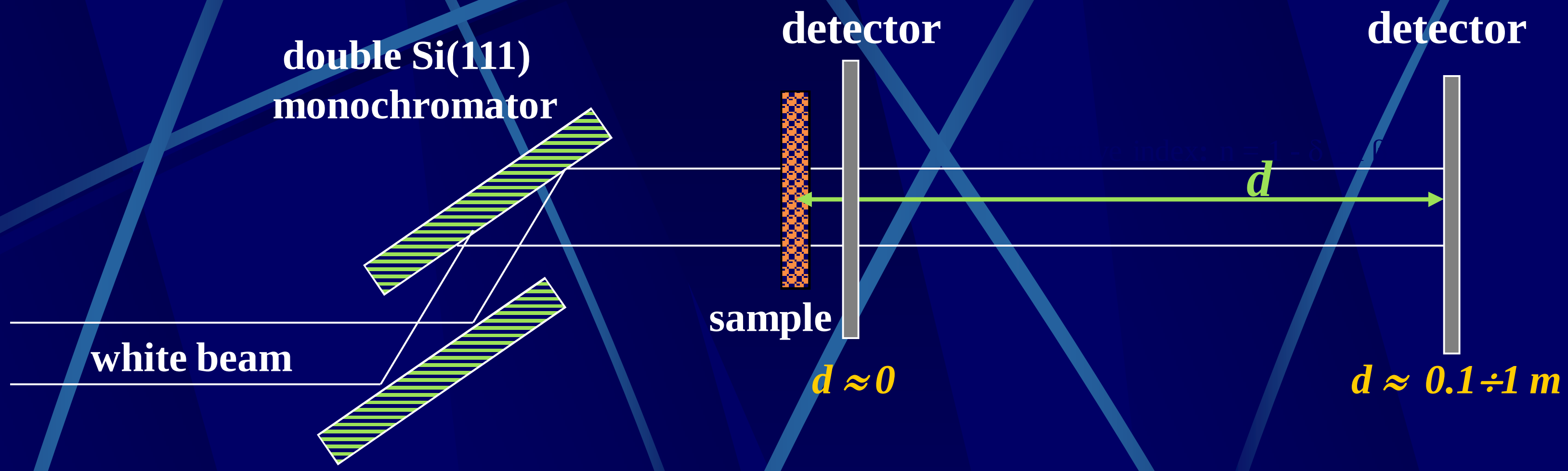
→ conservation techniques ….

The aim

to investigate the relationship between microstructural

and physical properties

Absorption and Phase Sensitive (PS) Radiography



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

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

$\mathbf{n} = 1 - \delta - i\beta$: refraction index

$\mu = 4\pi\, \beta / \lambda$: linear absorption coeff.

c : object size // to beam direction

$r \ll a \Rightarrow$ *edge detection regime*

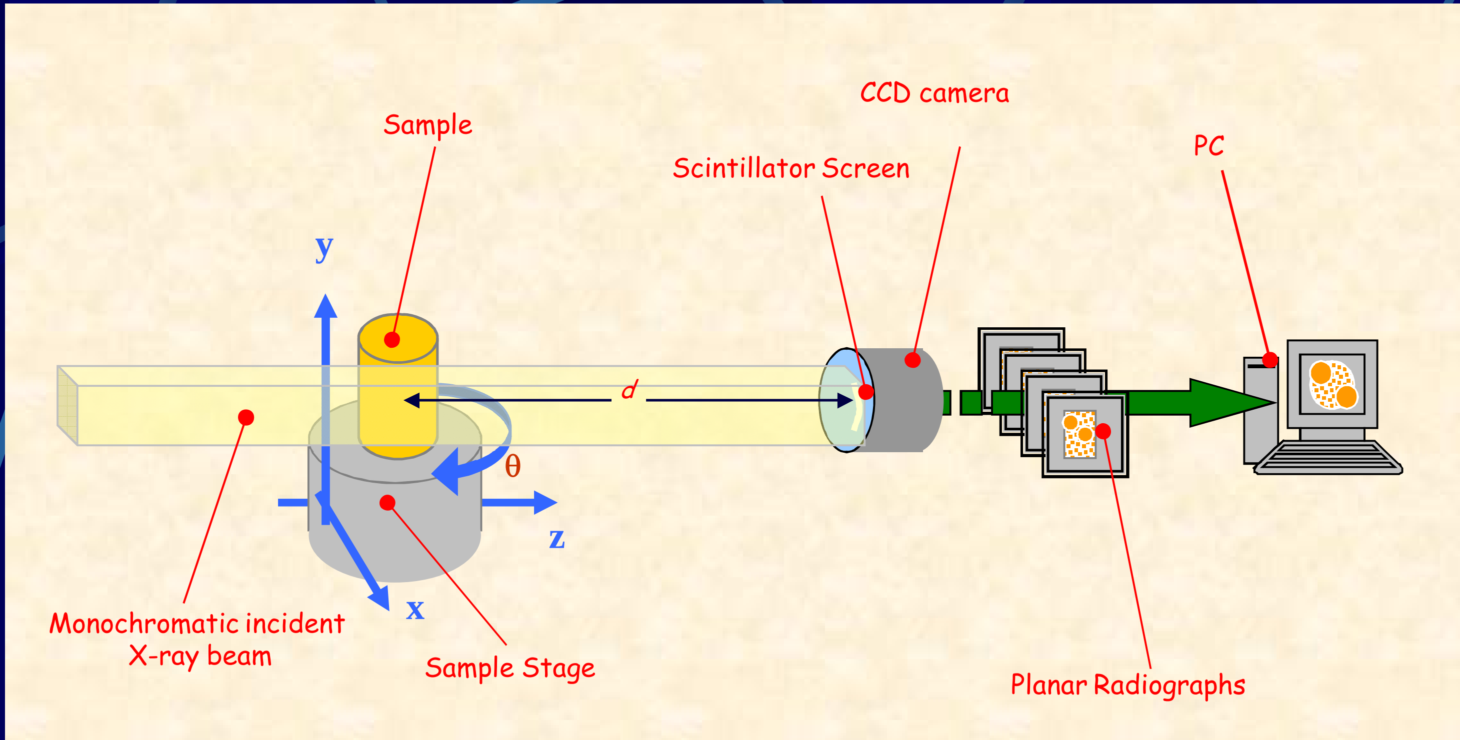
$r \cong a \Rightarrow$ *holographic regime*

$r \gg a \Rightarrow$ *Fraunhofer diffraction*

a : object size ⊥ to beam direction

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

Absorption and PS Computed μ-Tomography (μ–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...)

Waterlogged archaeological wood

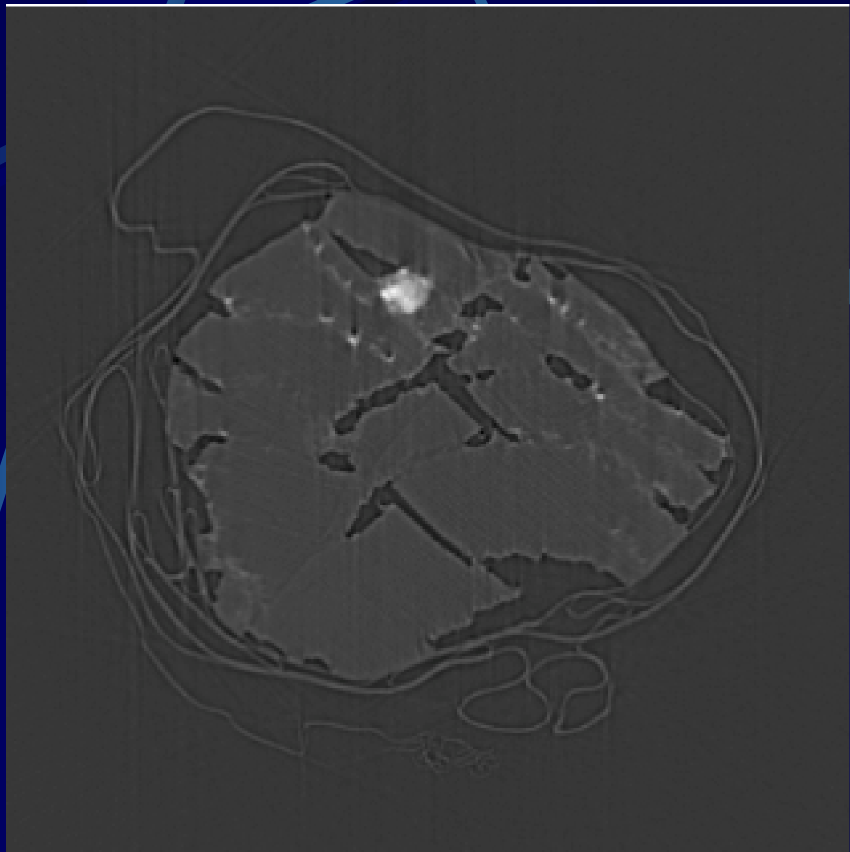
R. Auriemma[1], M. Fioravanti[2], F. Zanini[3,4], L. Mancini[4], A. Olivo[3,5], G. Tromba[4]

[1]University of Lecce, [2]University of Florence,
[3]University of Trieste, [4]Sincrotrone Trieste, [5]INFN, Sezione di Trieste

- The possibility to examine waterlogged wooden samples in their original environment, allows for a complete characterization in order to study the treatment of preservation and the restoration, both for desalinazation processes and controlled drying.

- Conventional treatments (water replacement with polyethylene glycol (PEG) or cryo-lyophilization) have well known drawbacks.

- The possibility to study the wooden structure during the treatment (and not only the final result, too often dissatisfactory) allows to evaluate its potentials and risks, and to grant the most congenial procedure regarding the kind of wood under study.



Reconstructed slice of a sample of waterlogged wood (*Abies Alba*) from the site of Murecine, near Pompei (pixel size = 14 mm, photon energy = 15 keV)

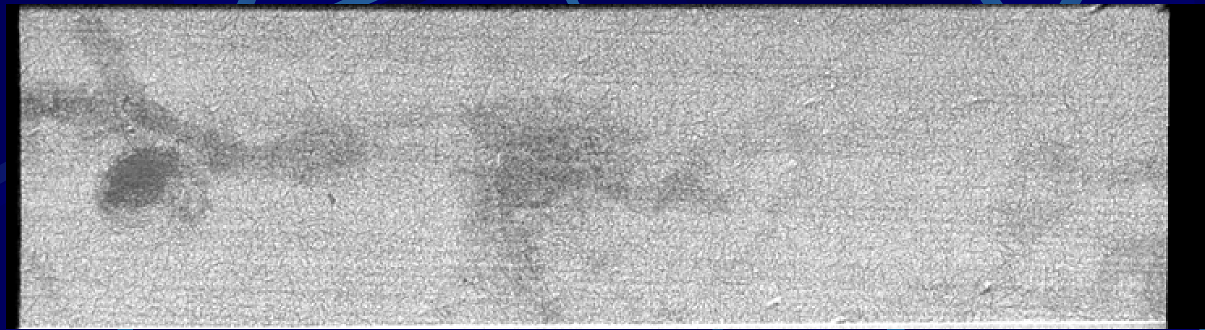
Iron gall ink on medieval papers

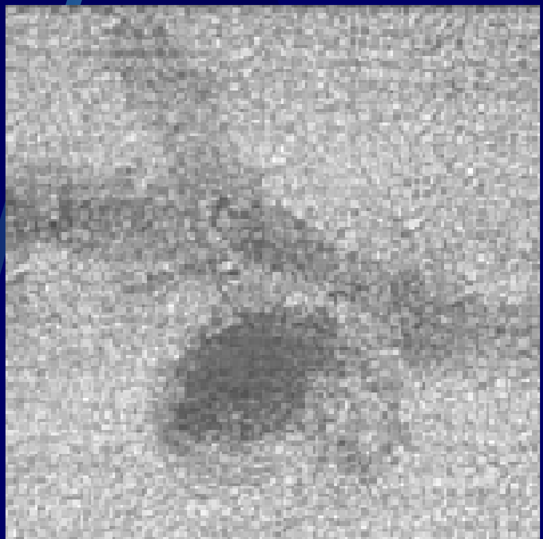
C. De Stefani[1], M. Plossi[1], A. Zappalà[1], F. Zanini[2,3], B. Kaulich[3], L. Mancini[3], M. Salome[4]

[1]University of Udine, [2]University of Trieste, [3]Sincrotrone Trieste, [4]ESRF, Grenoble

- Despite progress in our understanding of ink corrosion mechanism, the process remains very complicated involving various chemical mechanisms which influence each other directly.
- There is still confusion about the migratory behaviour of iron during natural ageing, and in particular the correlation between paper microstructure and migration paths.
- Aim of this project is the use of different synchrotron radiation techniques (X-Ray fluorescence, microEXAFS and phase-contrast imaging) in order to correlate chemical and structural information and develop safer treatments for ancient paper objects.

Iron gall ink on medieval papers

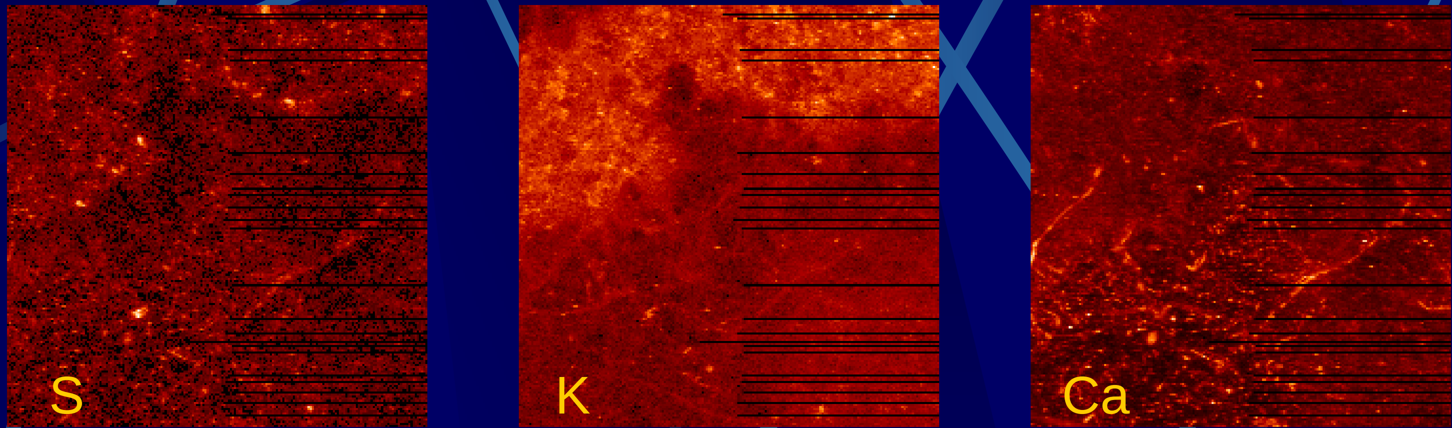


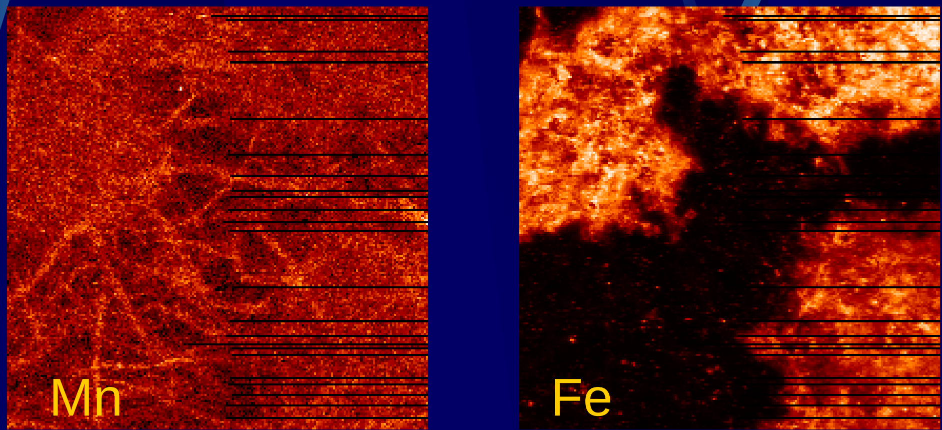


Manuscript from the Cassino Abbey, January 22nd 1626, field of view 5.5 x 20.6 mm^2, photon energy = 9 keV,

Detail of the same sample, 2.2 x 2.2 mm^2.

Iron gall ink on medieval papers

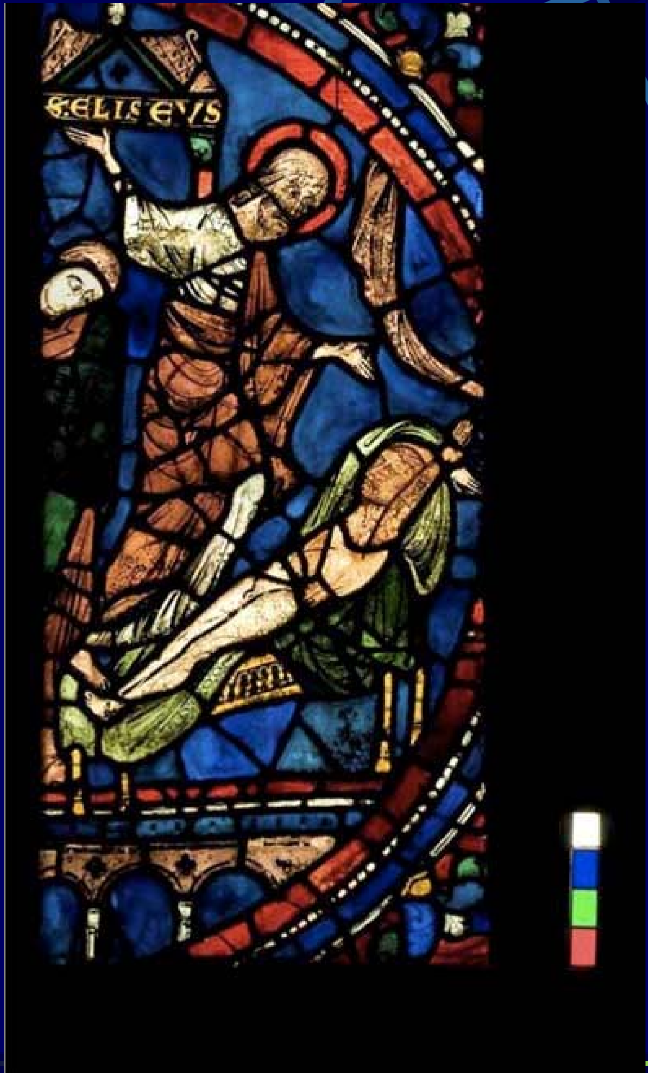


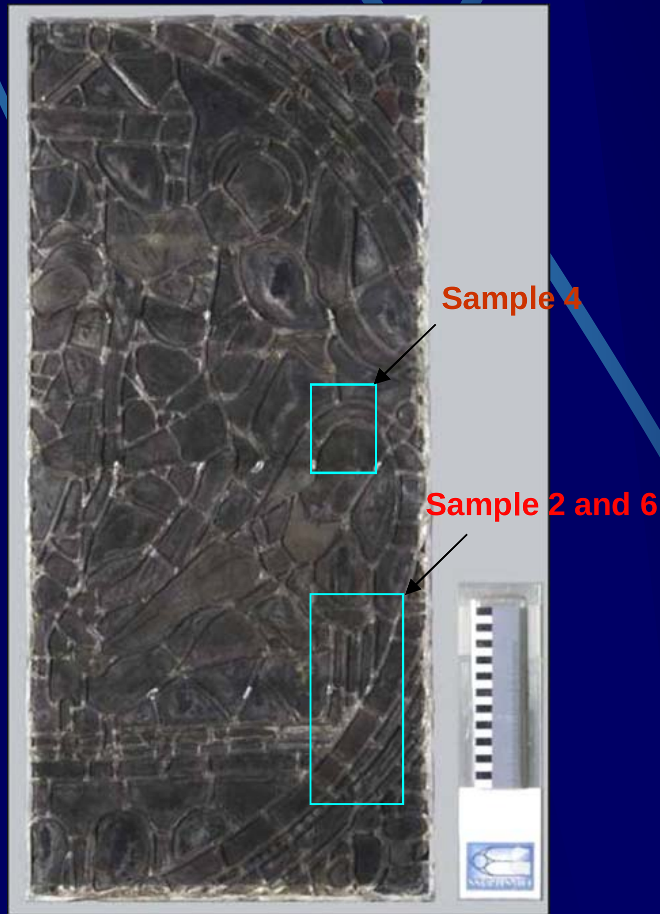


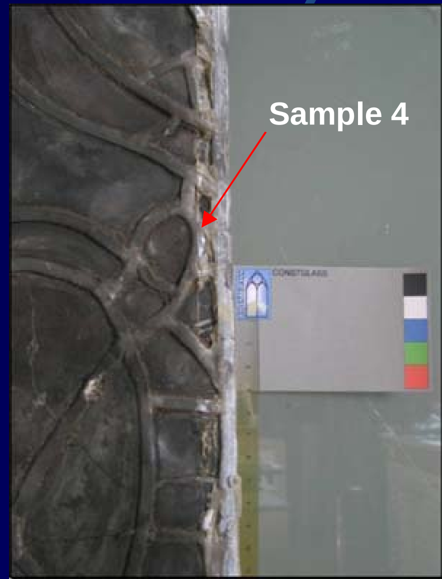
1mm x 1mm
5 microns steps
300 ms/point
in air
7.2 keV

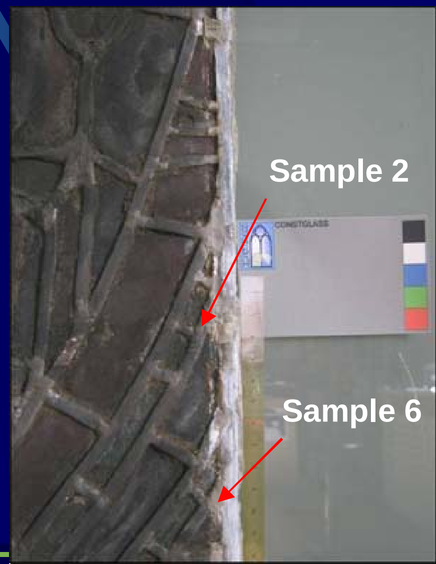
ESRF, ID 21

Chartres - Window 37
La Passion typologique

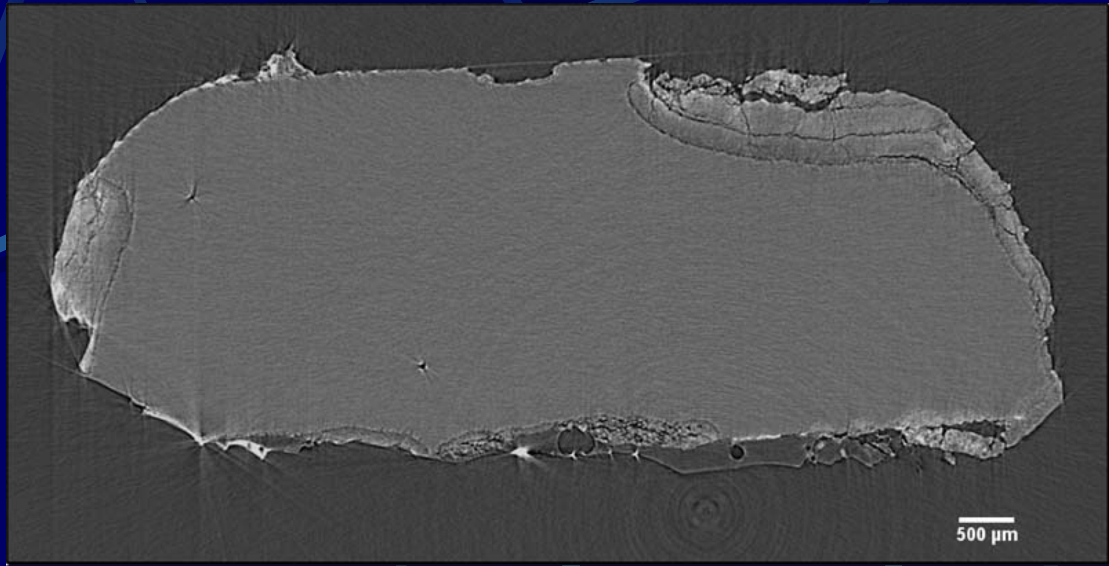


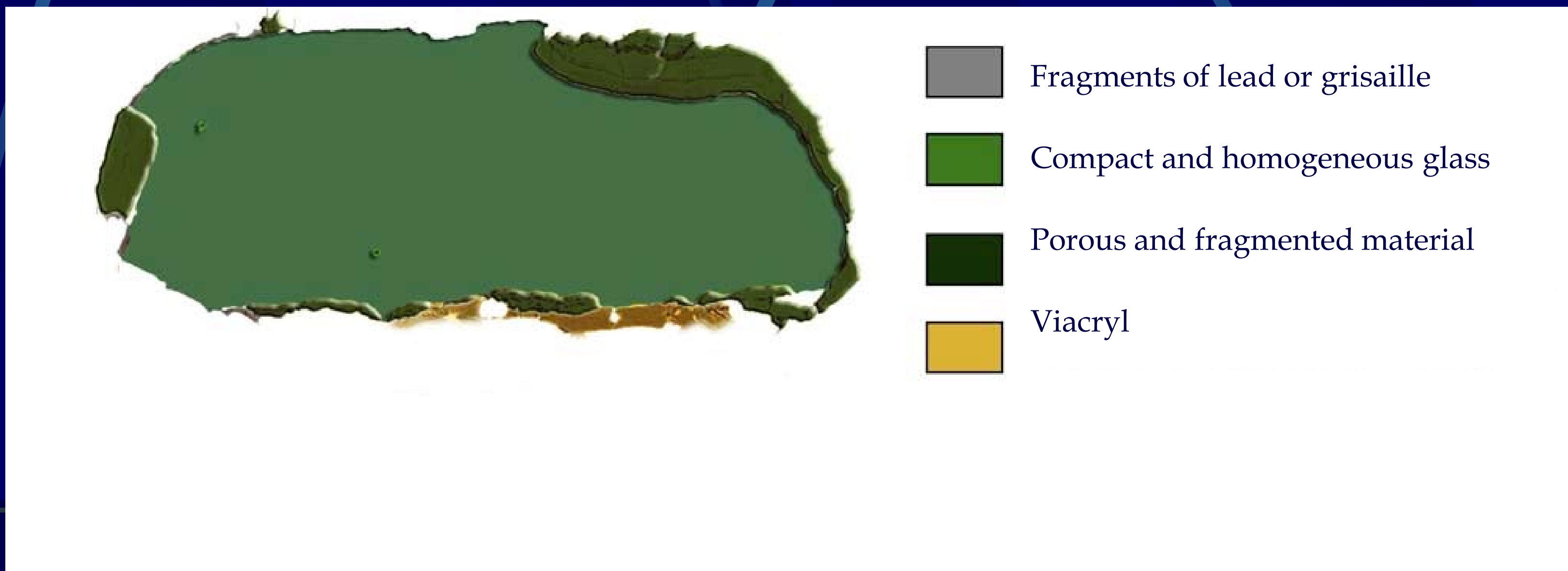


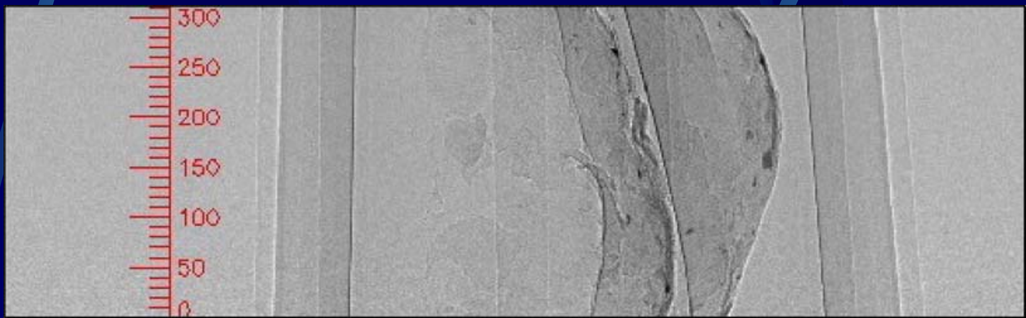




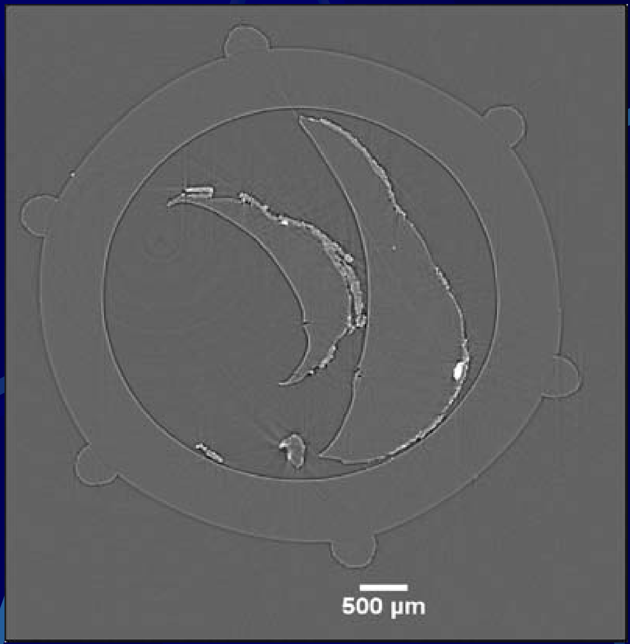
Sample 2: glass fragment with grisaille decoration and Viacryl layer on the internal face. Evident alterations on the surface sides.


500 µm

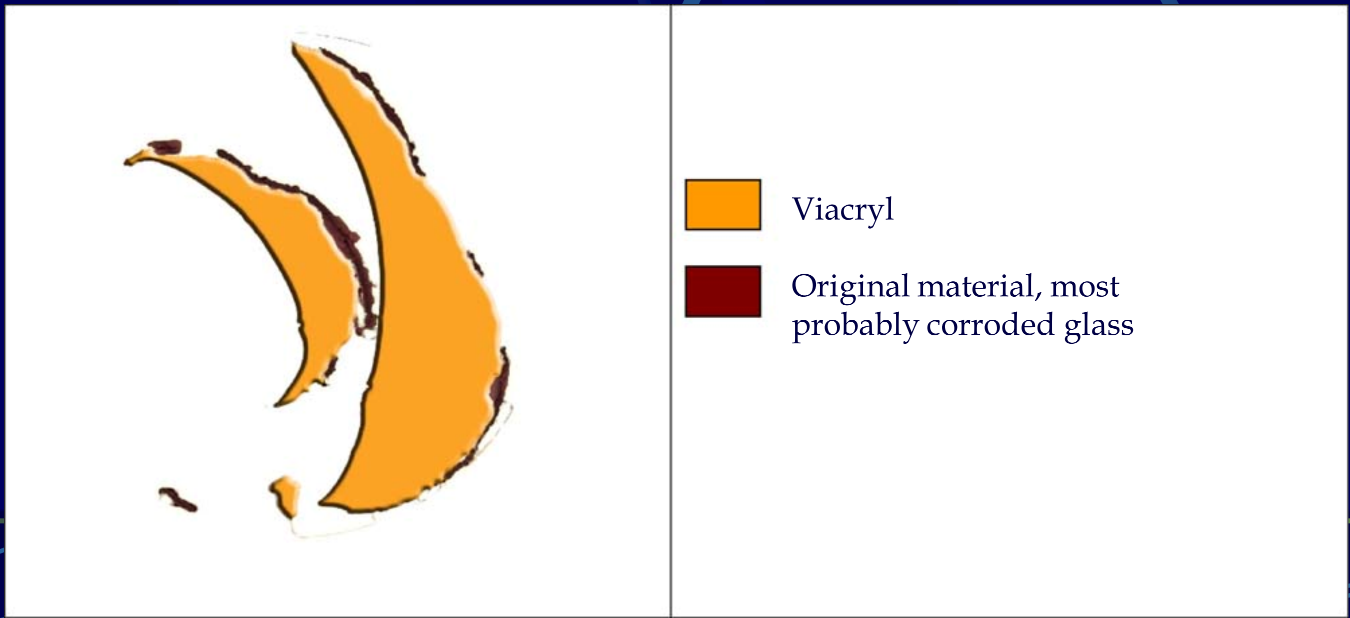

Fragments of lead or grisaille
Compact and homogeneous glass
Porous and fragmented material
Viacryl

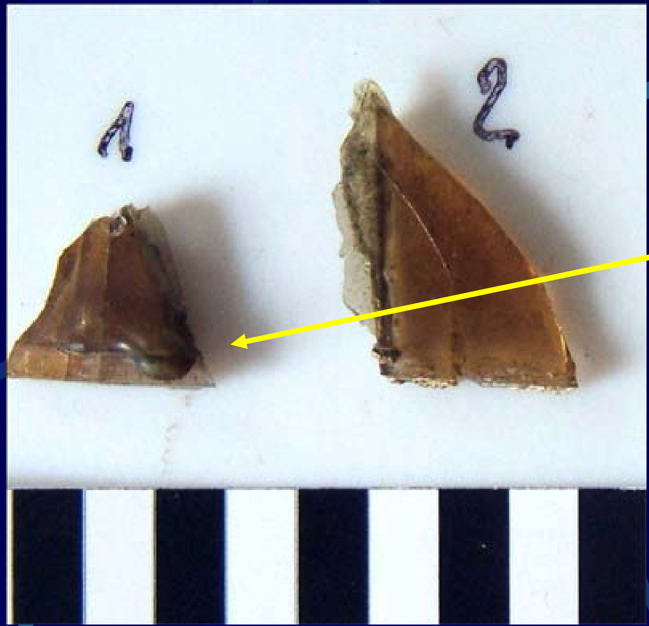


Viacryl flakes from Bourges
(window 9, panel 4)

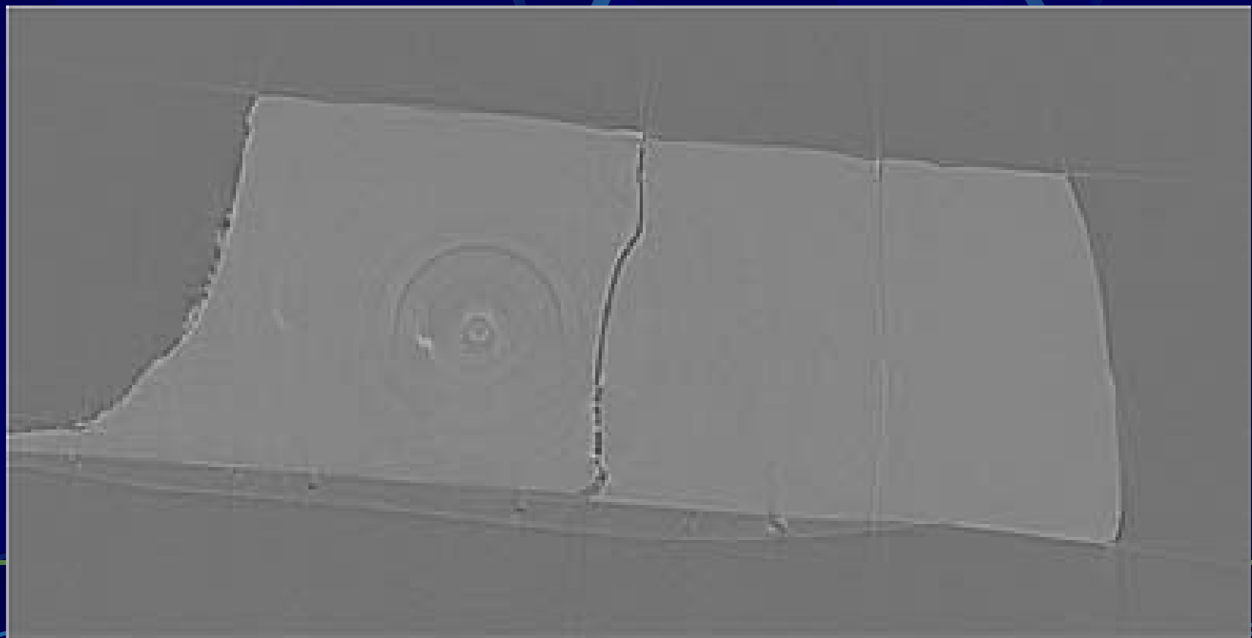


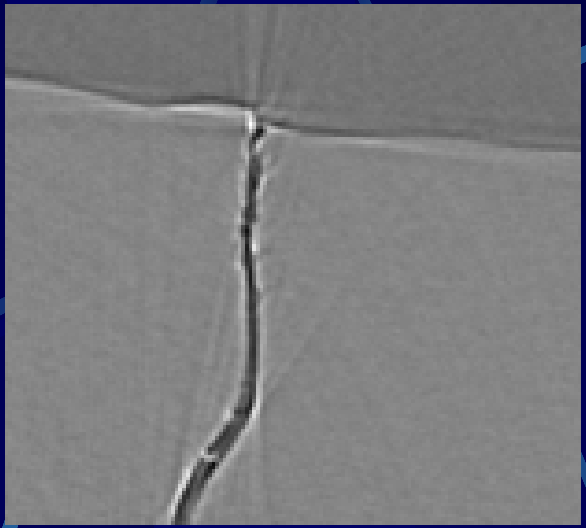
Viacryl flakes from Bourges (window 9, panel 4). It is evident that the Viacryl removes some original material from the glass panel.

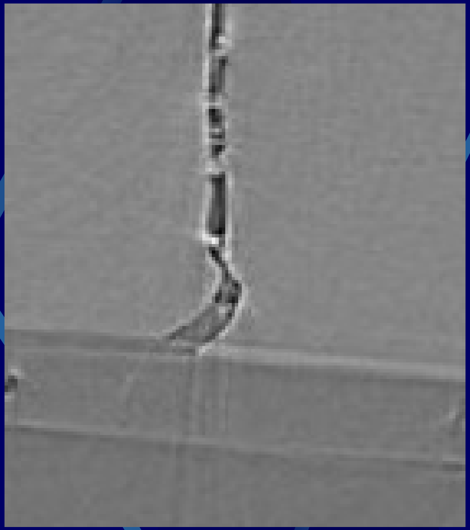




Original glass (beginning of 20th century) bonded with epoxide resin (Epidian 53) about 30 years ago from interior, without glass dismantling.

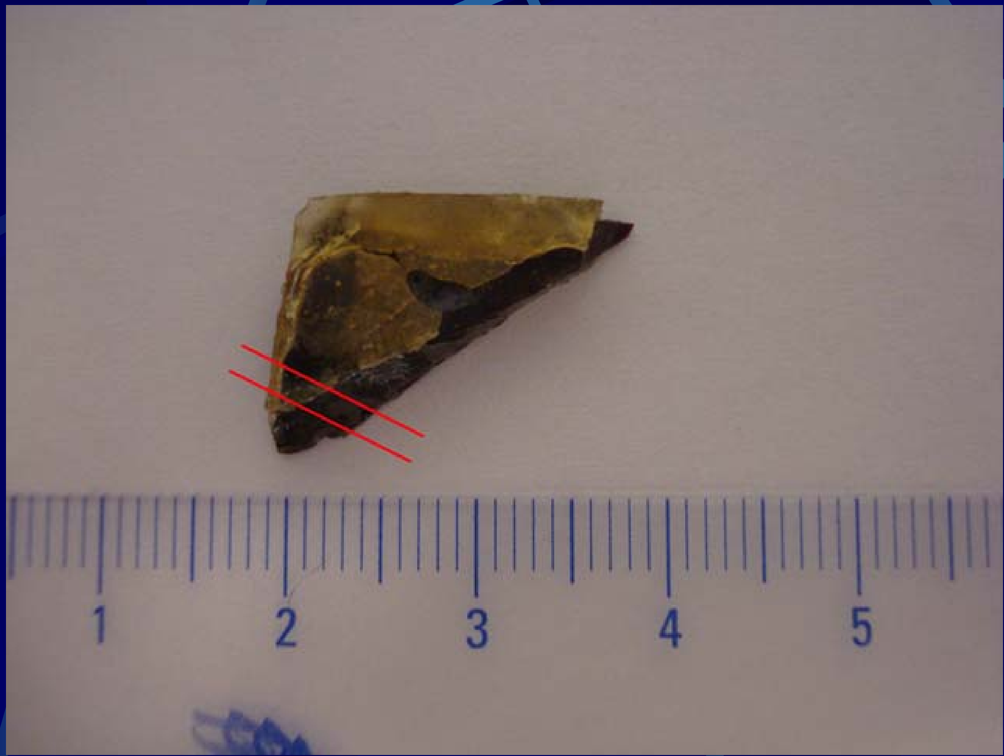






The microtomographic analysis shows a thick layer of resin on the glass surface and slight penetration of resin into the break. The break itself is soiled by external pollution. This explains the fact that the bonding is rather weak.

Sample CSRIV_01



Burgdorf Parish Church.

Vestry on south side of the choir, 16th century.

Window destroyed in 1707, fragments found in 1968, fragment panels mounted in 1971.

Red flashed piece in a triangle outline. The Araldite is yellowing and loses adhesion. Where it flakes off, it shows clearly different types of deterioration.

Sample CSRIV_01

For thin fragments with multiple cracks, simple bond edge was not considered sufficient, and a doubling method was used.

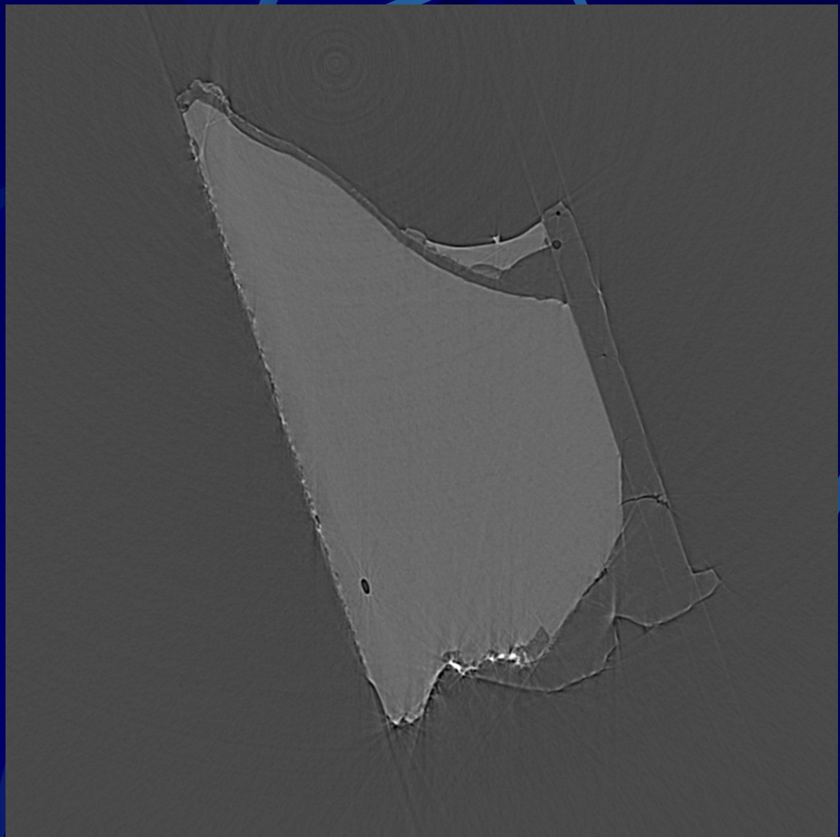
Araldite was poured on a thin carrier glass, the fragment was put on top and left under pressure. Araldite binder AY 103 by 100 parts, hardener hy951 by 9 parts.

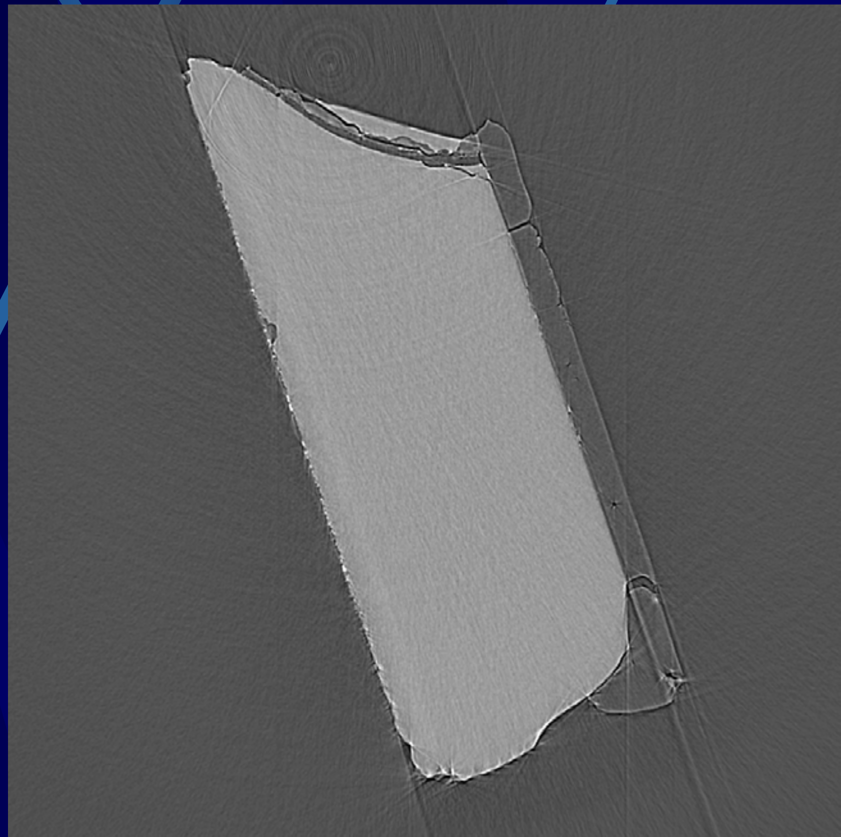
Sample CSRIV_01

For this sample the carrier glass was cut along the old crack of the original piece of glass. In this area the Araldite was only sticking to the old glass.

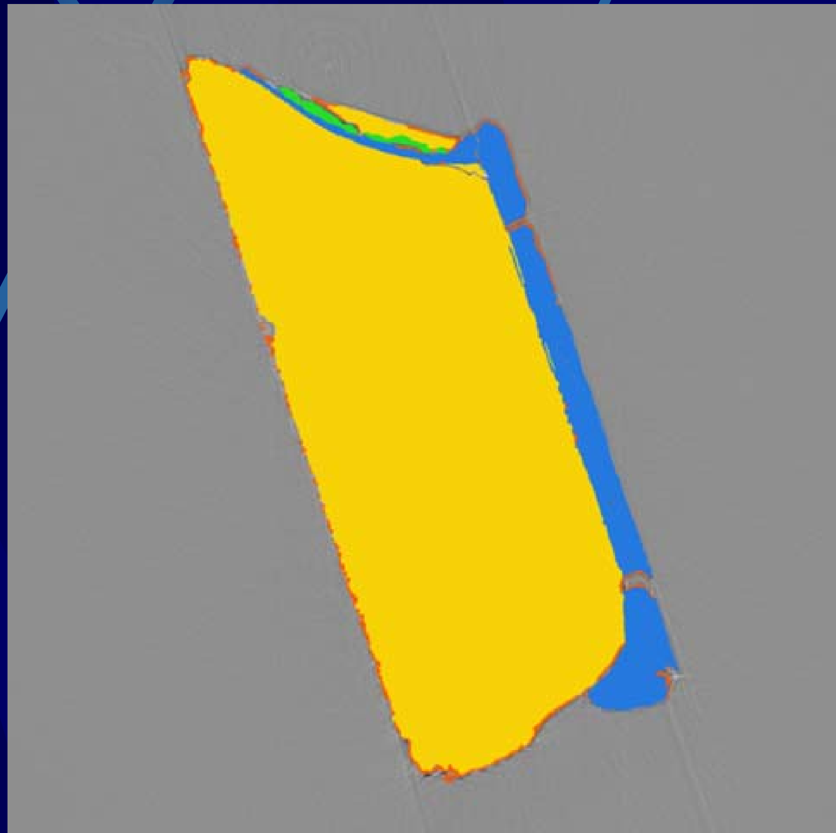
How far did the Araldite penetrate into the glass surface and eventually also thin external paint layers?
When peeling off, does the Araldite hurt the surface (with silver stain and eventual layers of grisaille?

Sample CSRIV_01



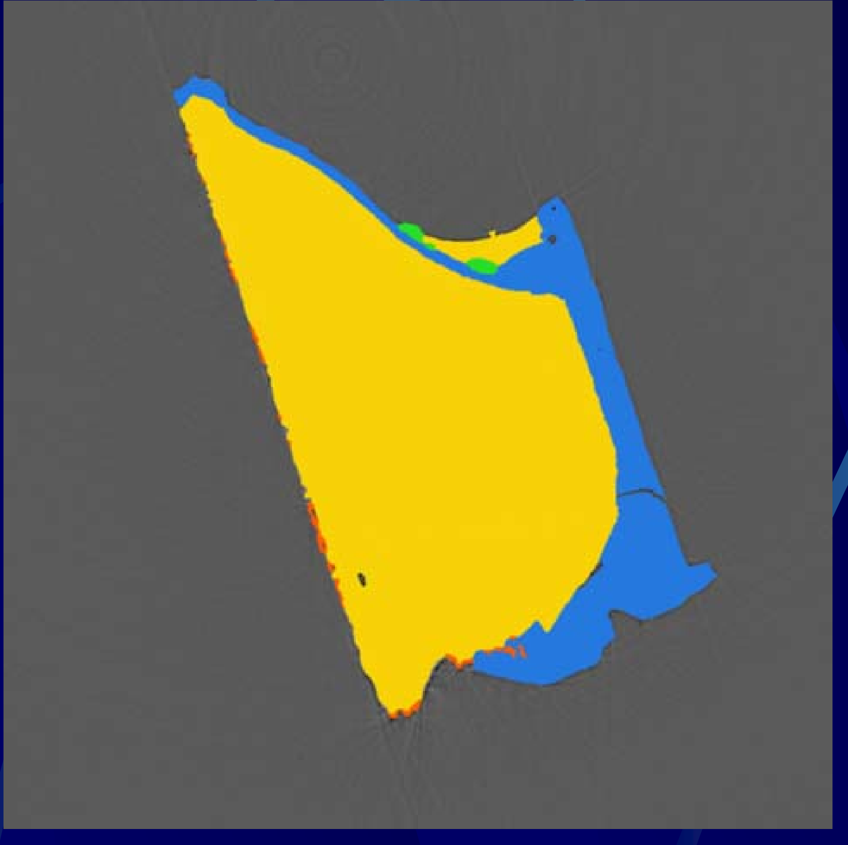


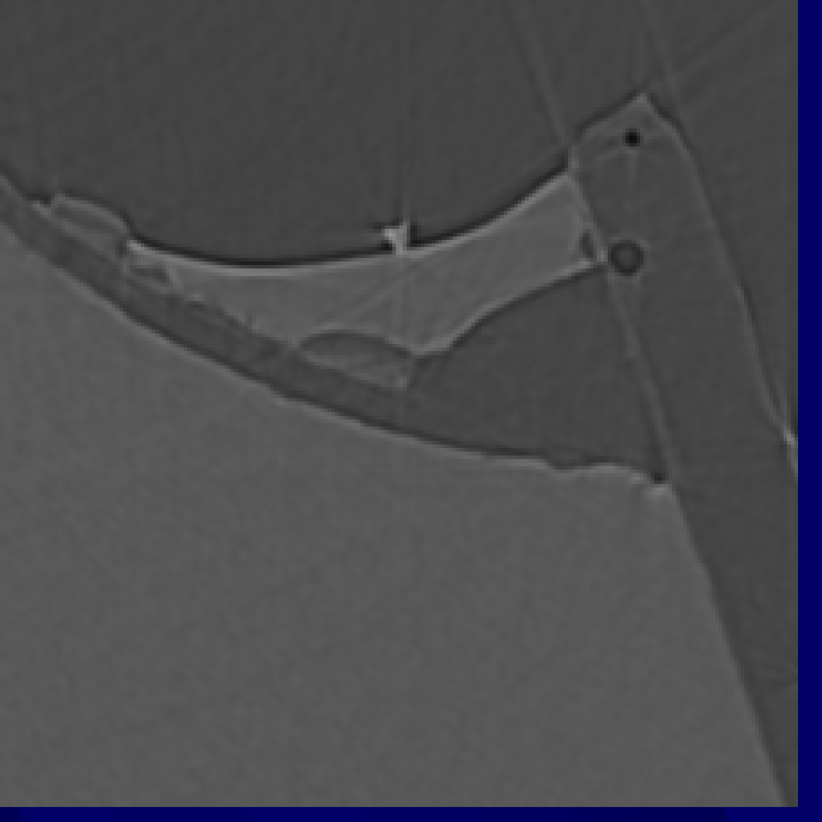
Sample CSRIV_01



Yellow = original glass Blue = Araldite
Green = degraded glass Red = metal

Sample CSRIV_01





Yellow = original glass   Blue = Araldite
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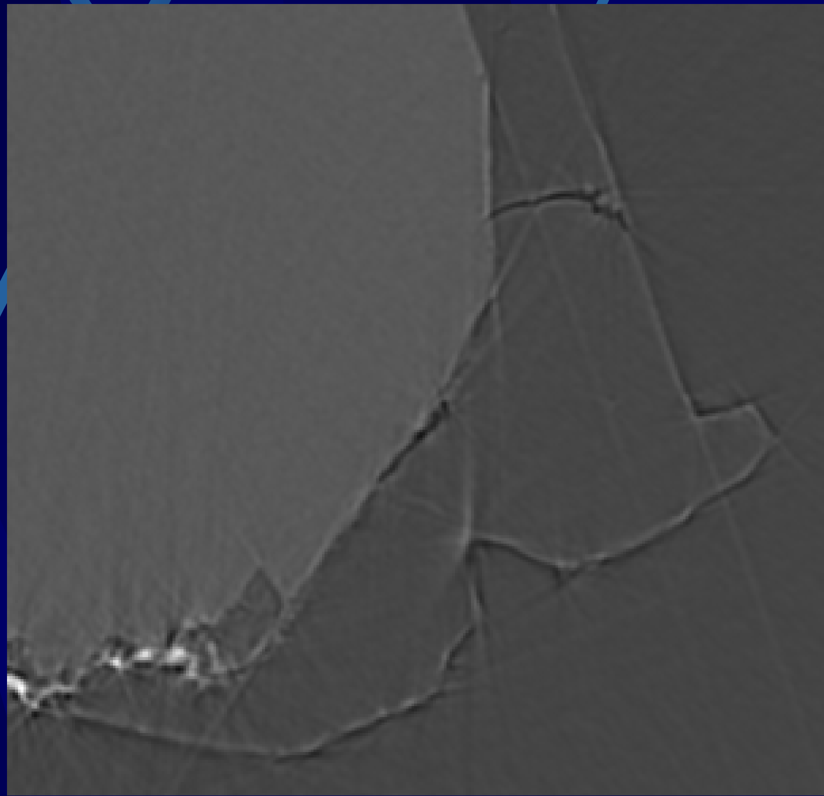
Sample CSRIV_01



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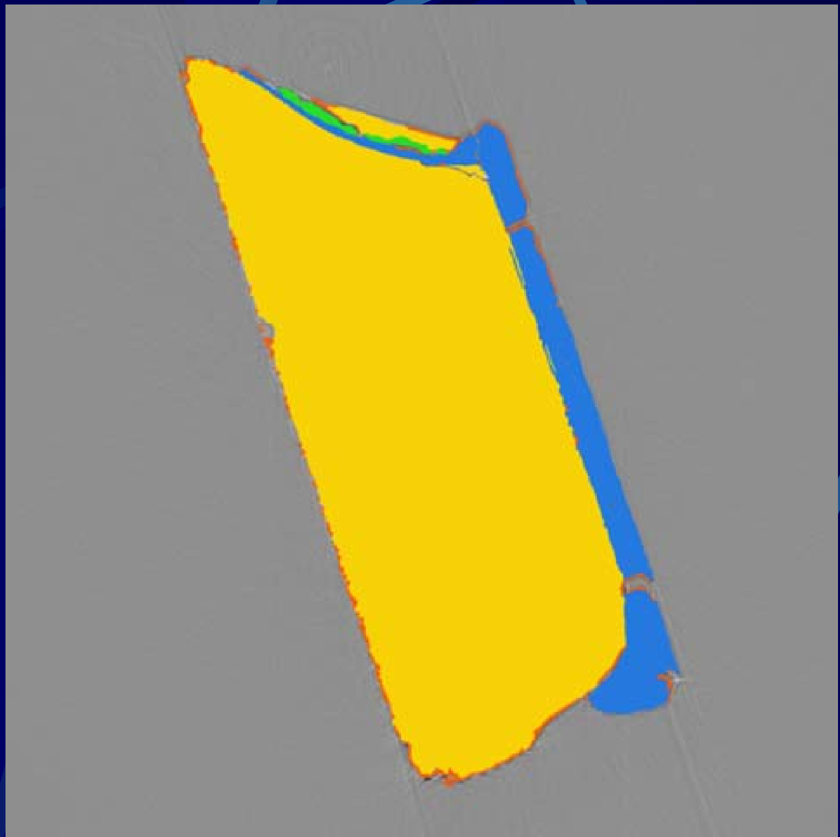

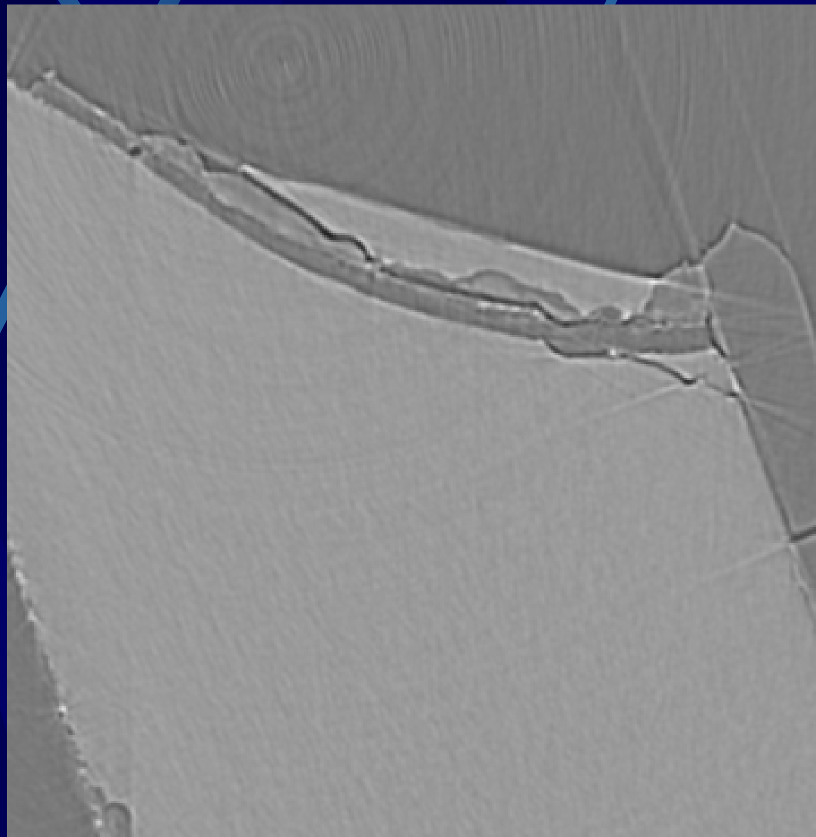

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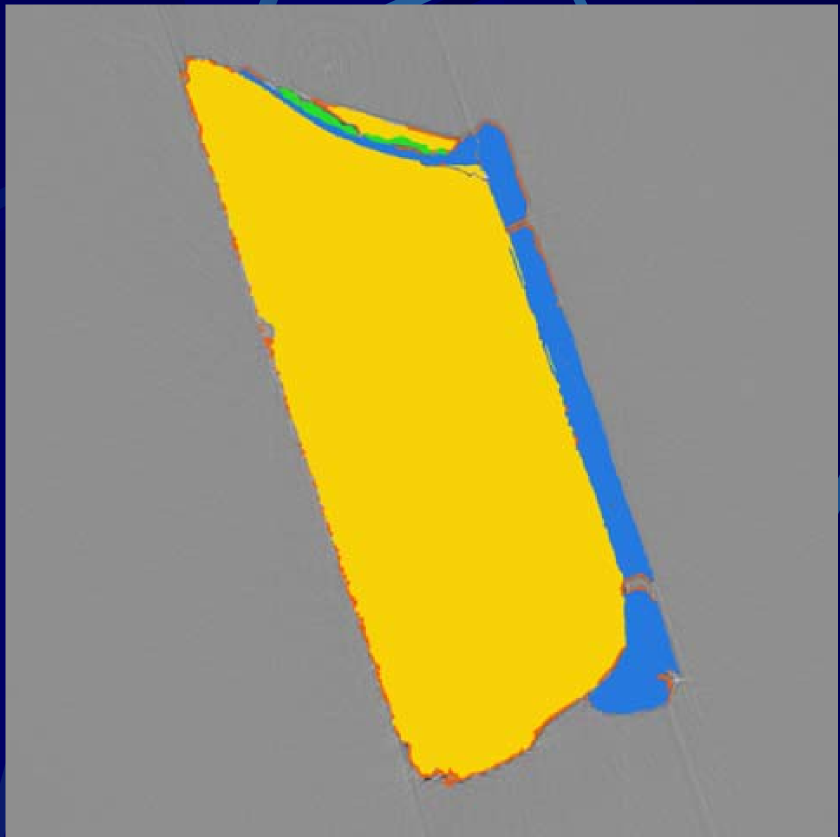
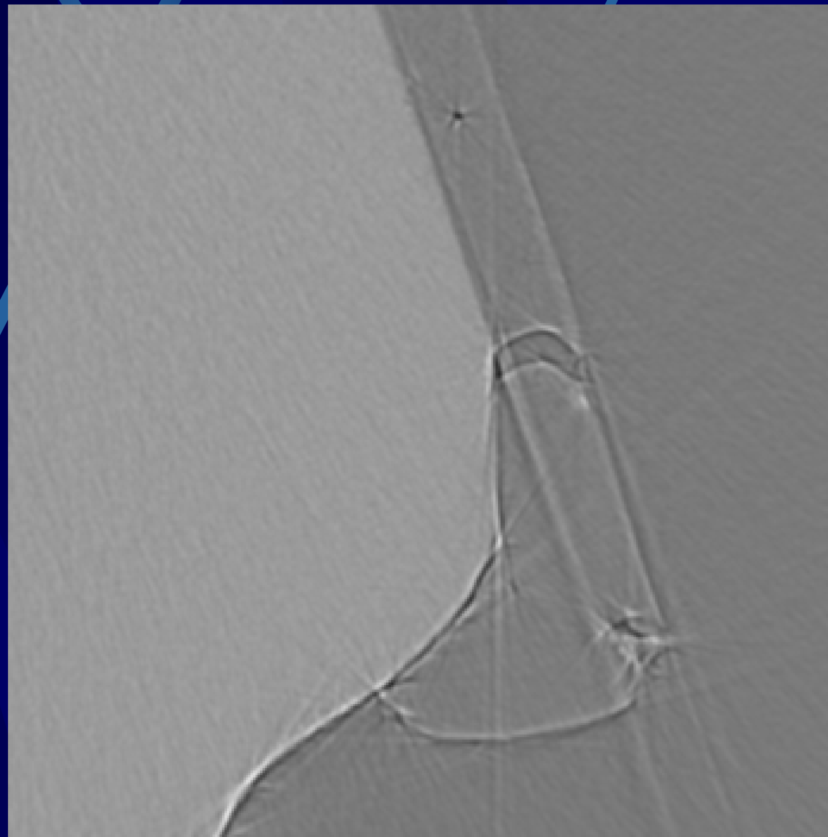




Yellow = original glass Blue = Araldite
Green = degraded glass Red = metal

Sample CAN 1a

Canterbury Cathedral.

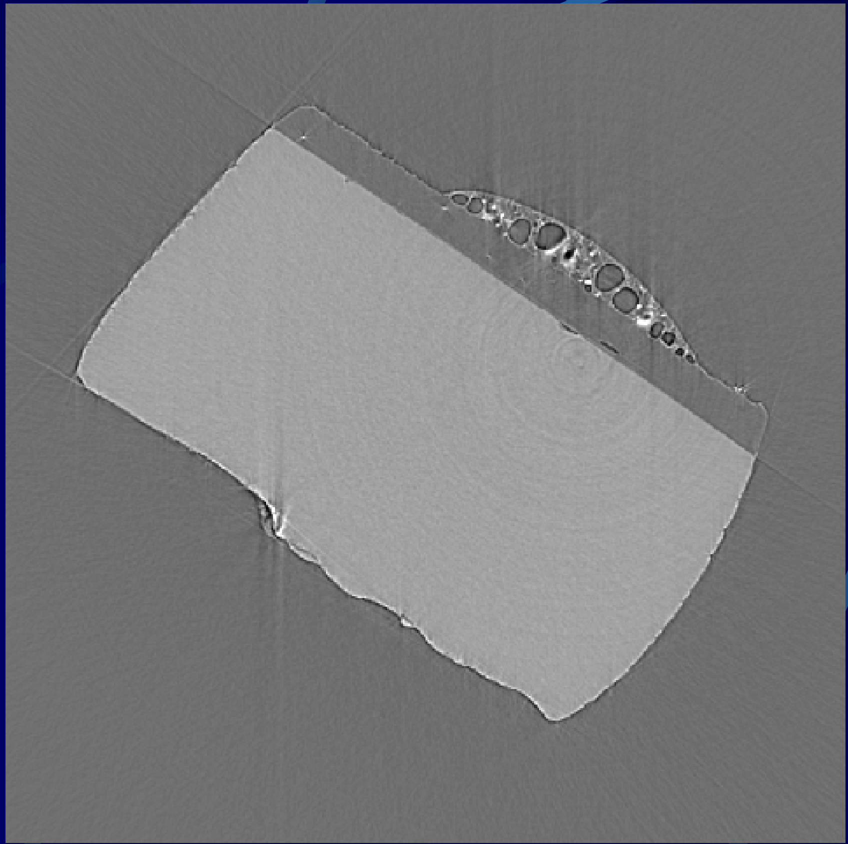
Unknown origin.

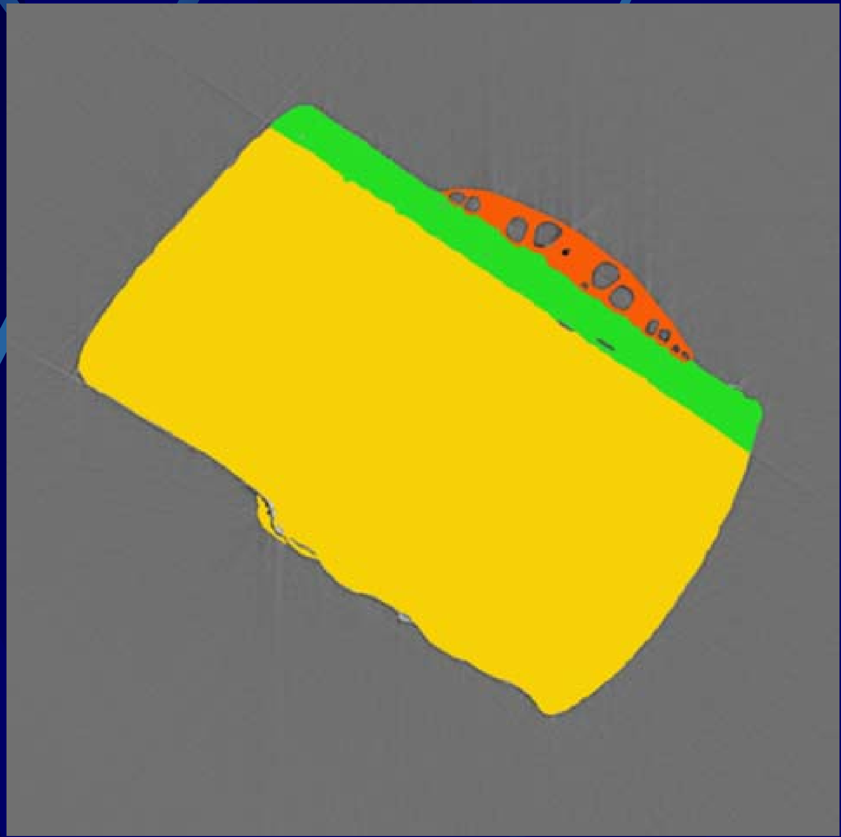
Fragment of medieval green tinted glass with slight surface corrosion.

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.

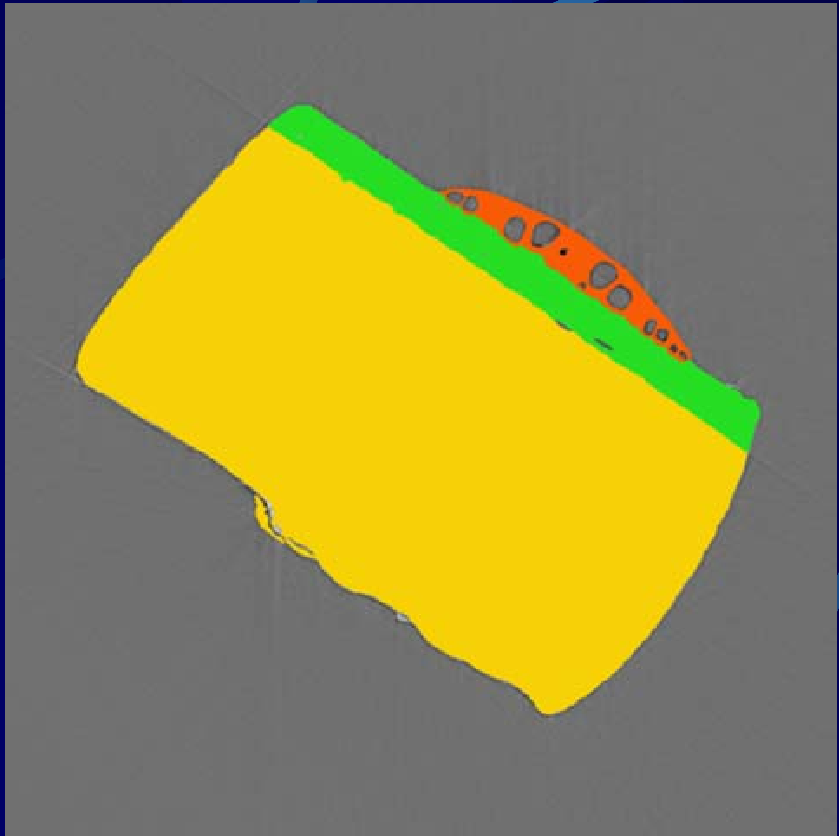
Sample CAN 1a

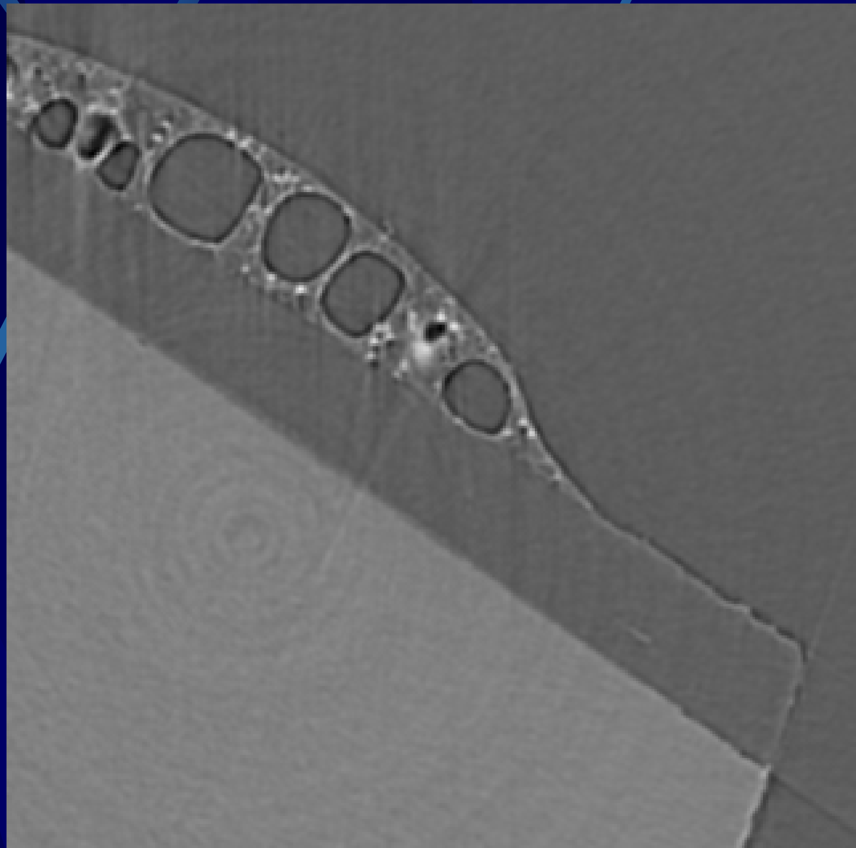




The wax was applied in 3 separate layers and left for three days. The Paraloid B72 was mixed together with raw umber pure powder pigment and this was applied with a small brush on the wax.

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.

Structural analysis of musical instruments

- Restoration and conservation
- Manufacturing techniques
- Acoustic analysis
- Economic evaluation
- Fabrication of replicas

Structural analysis of musical instruments

- Non - destructive analyses
- Samples of large dimensions
- Strict environmental conditions
- High spatial resolution
- Fast exposition times

The organ by Lorenzo da Pavia

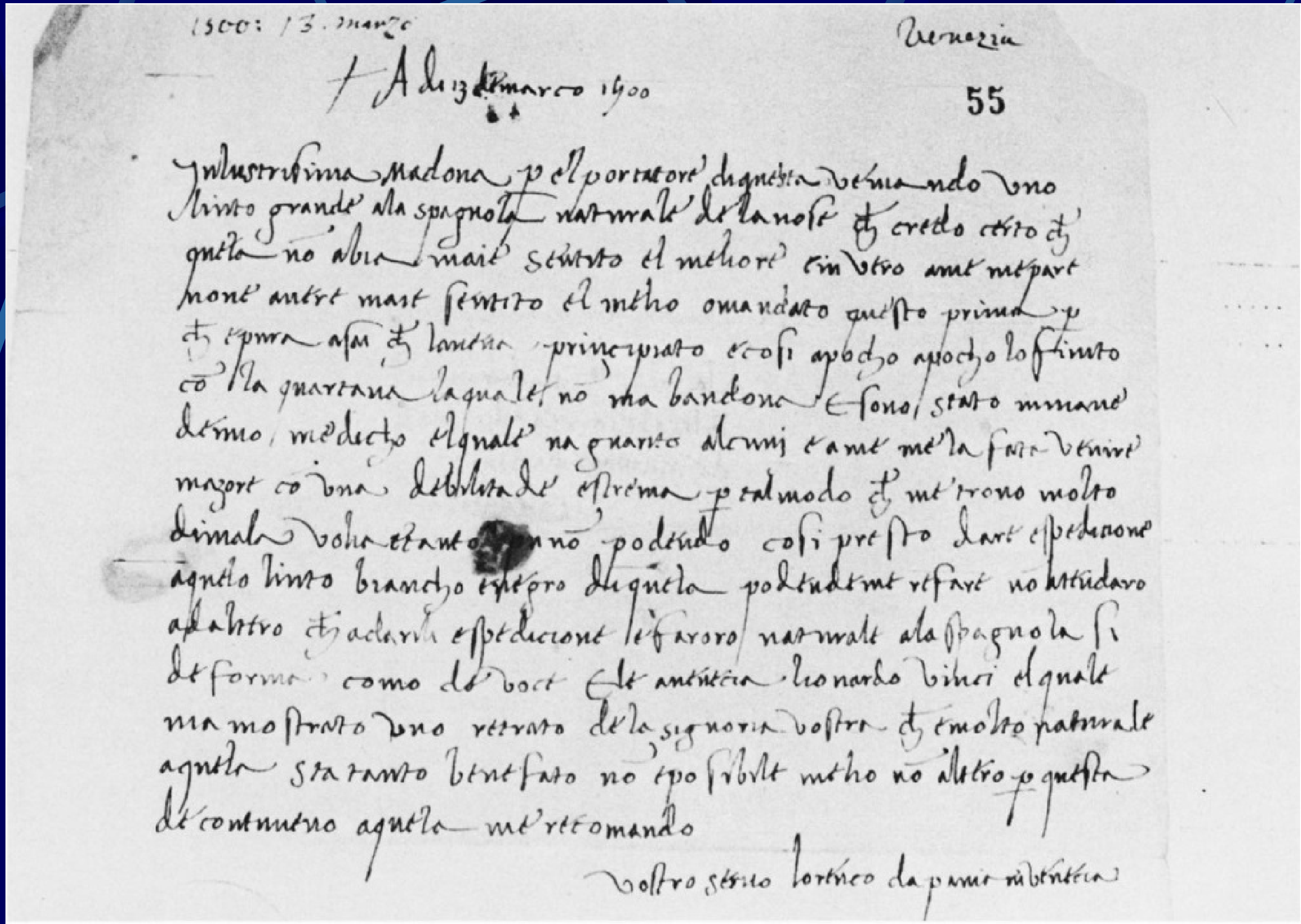
Organ by Lorenzo Gusnasco (1494)

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

+ Adì 13 de marzo 1500

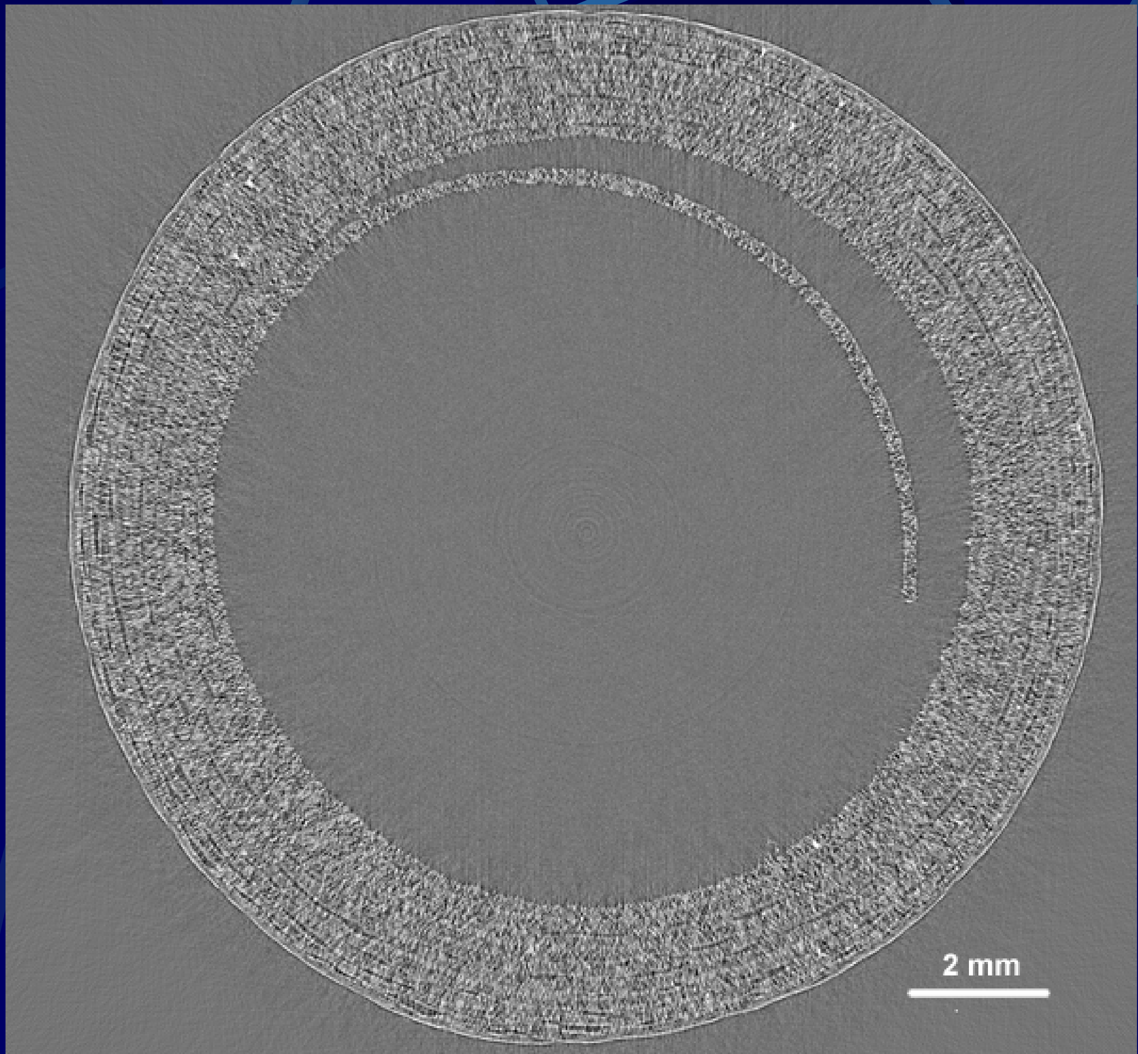
55

Illustrissima Madona p[er] el portatore de questa ve mando uno
liuto grande a la spagnola naturale de la noce che credo certo che
quello no abia mai sentito el meliore in vero a me me pare
non avere mai sentito el meio o mandato questo prima per
che eperava assai che l'arivera principiato e cosi a pocho a pocho l'o finito
co la quartana la quale no me abandona e sono stato mi manco
de mio medico el quale ne guarito alcuni e a me me la fa venire
magiore co una debilitade estrema per tal modo che me trovo molto
di mala voia e tanto [illegible] no podendo cosi presto dare espedicione
a quello liuto brancho intiero di quello podendome refare no studiaro
a pi altro che a darli espedicione l'e faroro naturale a la spagnola si
de forma como de voce. E a Venetia Lionardo Vinci el quale
m'a mostrato uno retrato de la signoria vostra che e molto naturale
a quella sta tanto bene fato no e posibile meio no altro per questa
de continuo a quella me recomando

Vostro servo Lorenzo da Pavia in Venetia

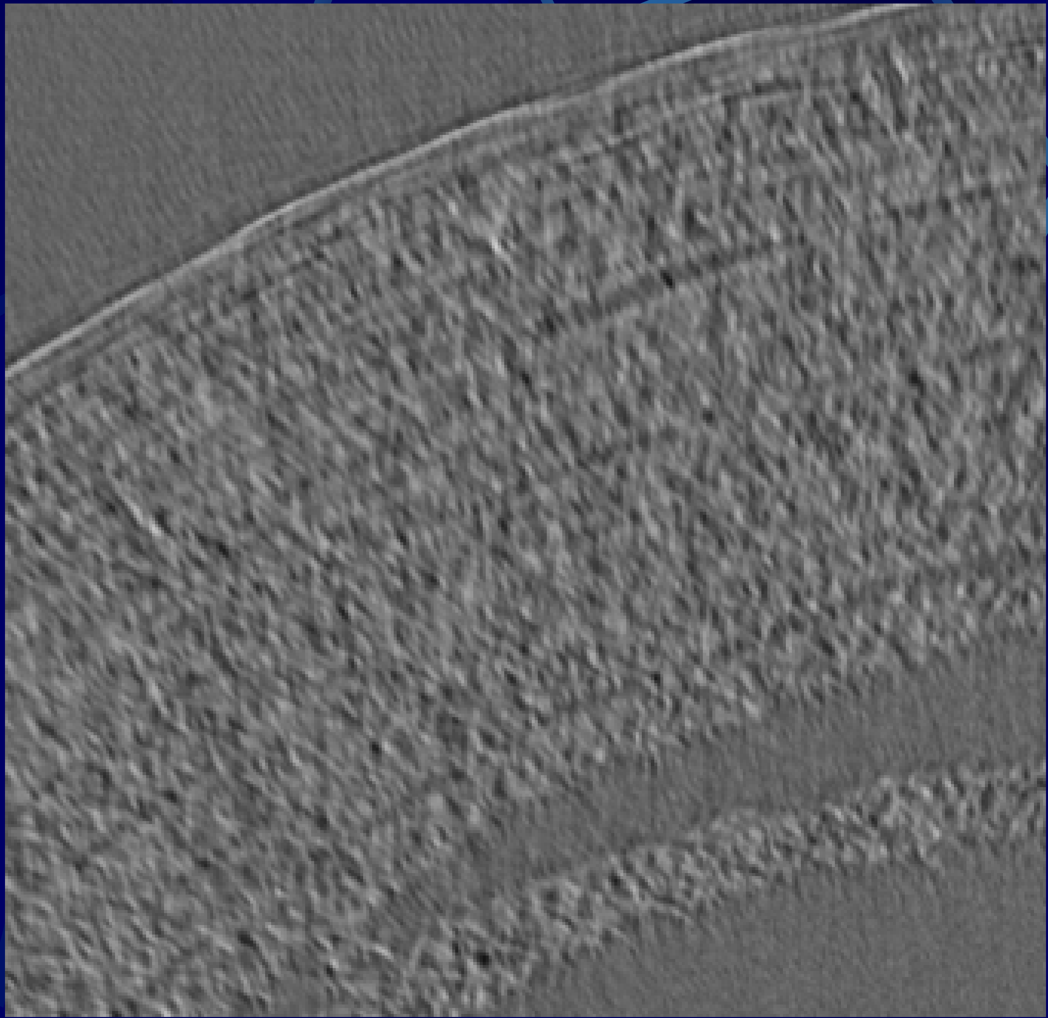
Correspondence between Lorenzo Gusnasco and Isabella d'Este

The organ by Lorenzo da Pavia



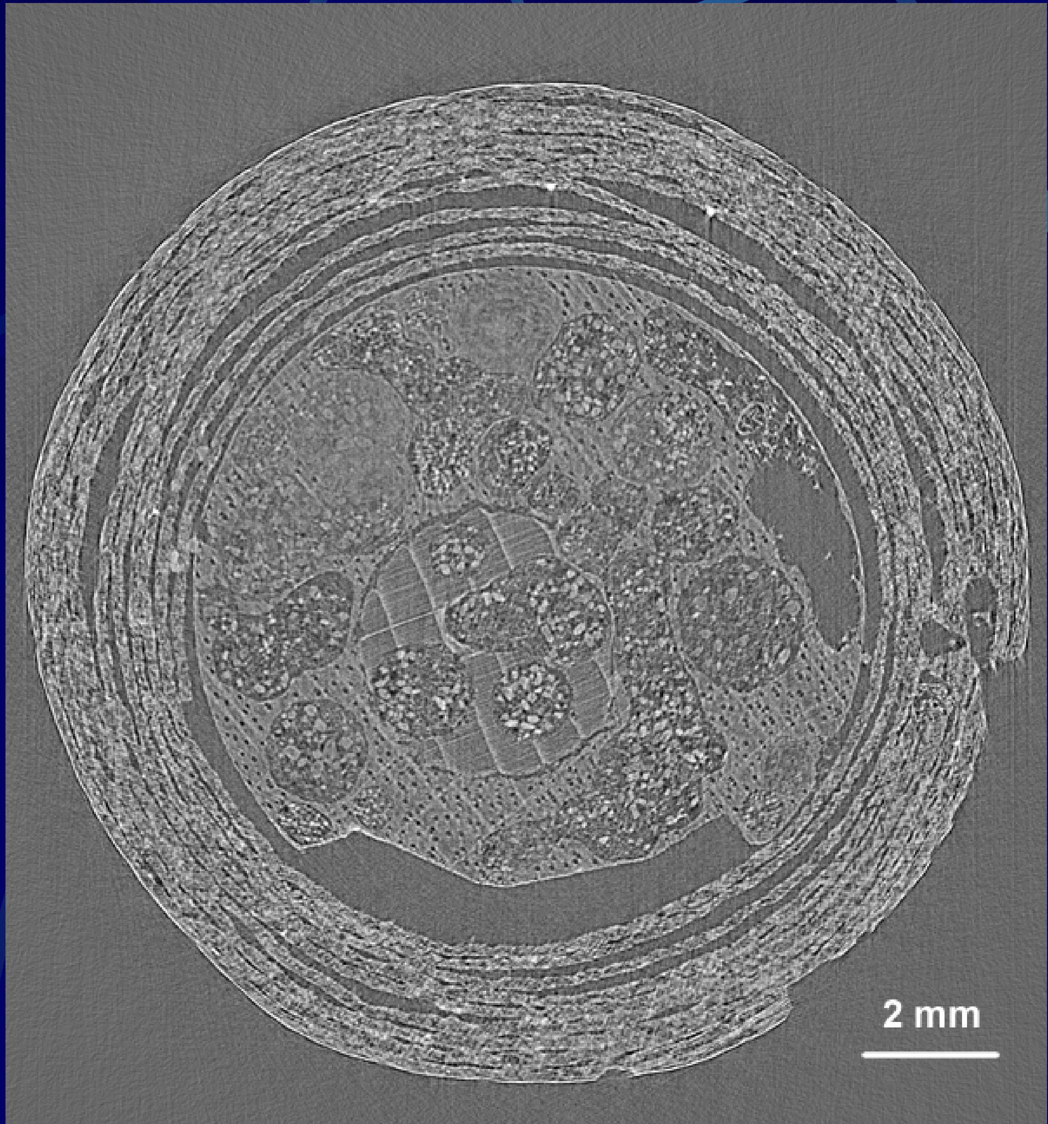
Virtual slice of a paper pipe with a spatial resolution of 9 microns

The organ by Lorenzo da Pavia



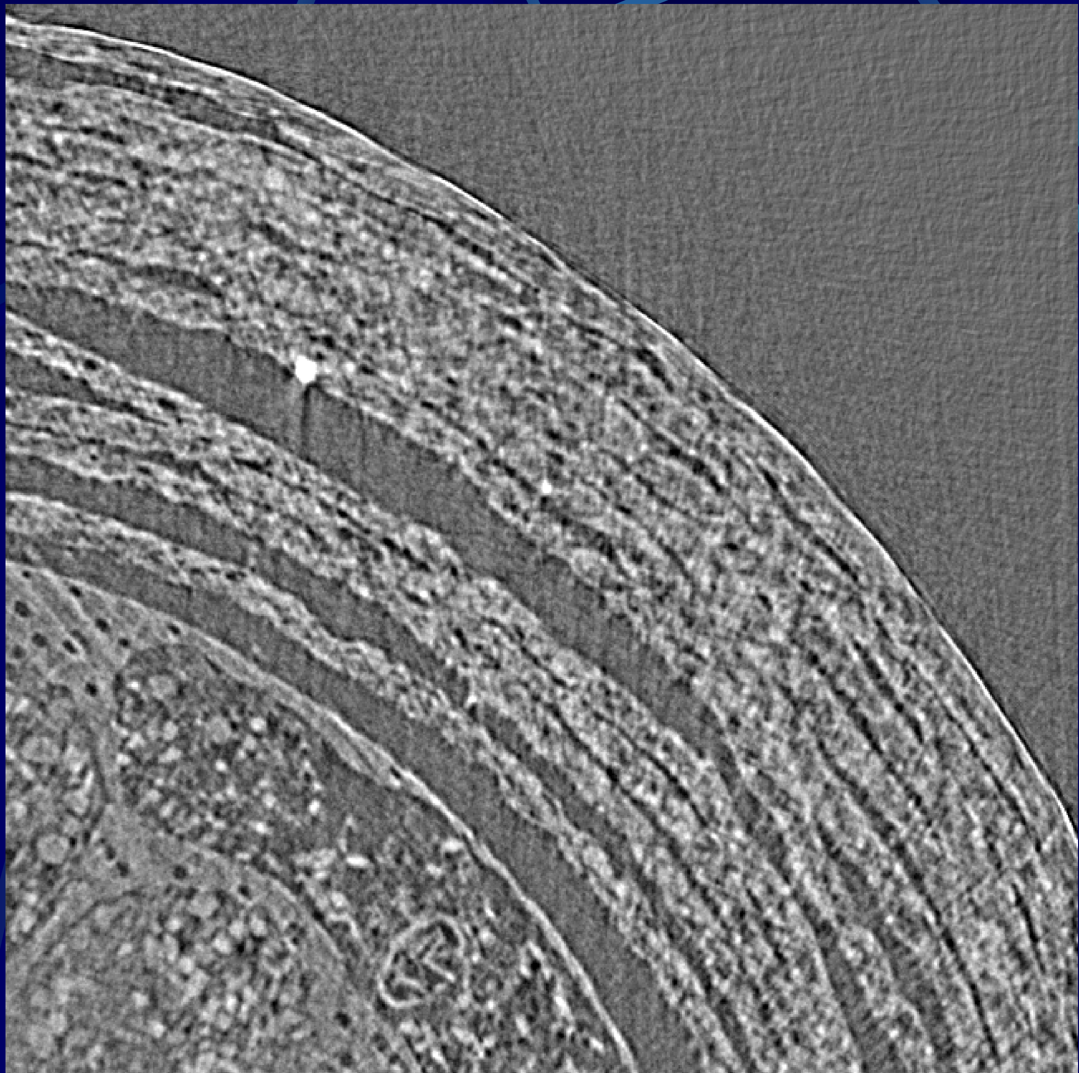
- Ten 0.25 mm layers
- Good quality of the external layer
- Good adhesion of layers, except the inner one

The organ by Lorenzo da Pavia



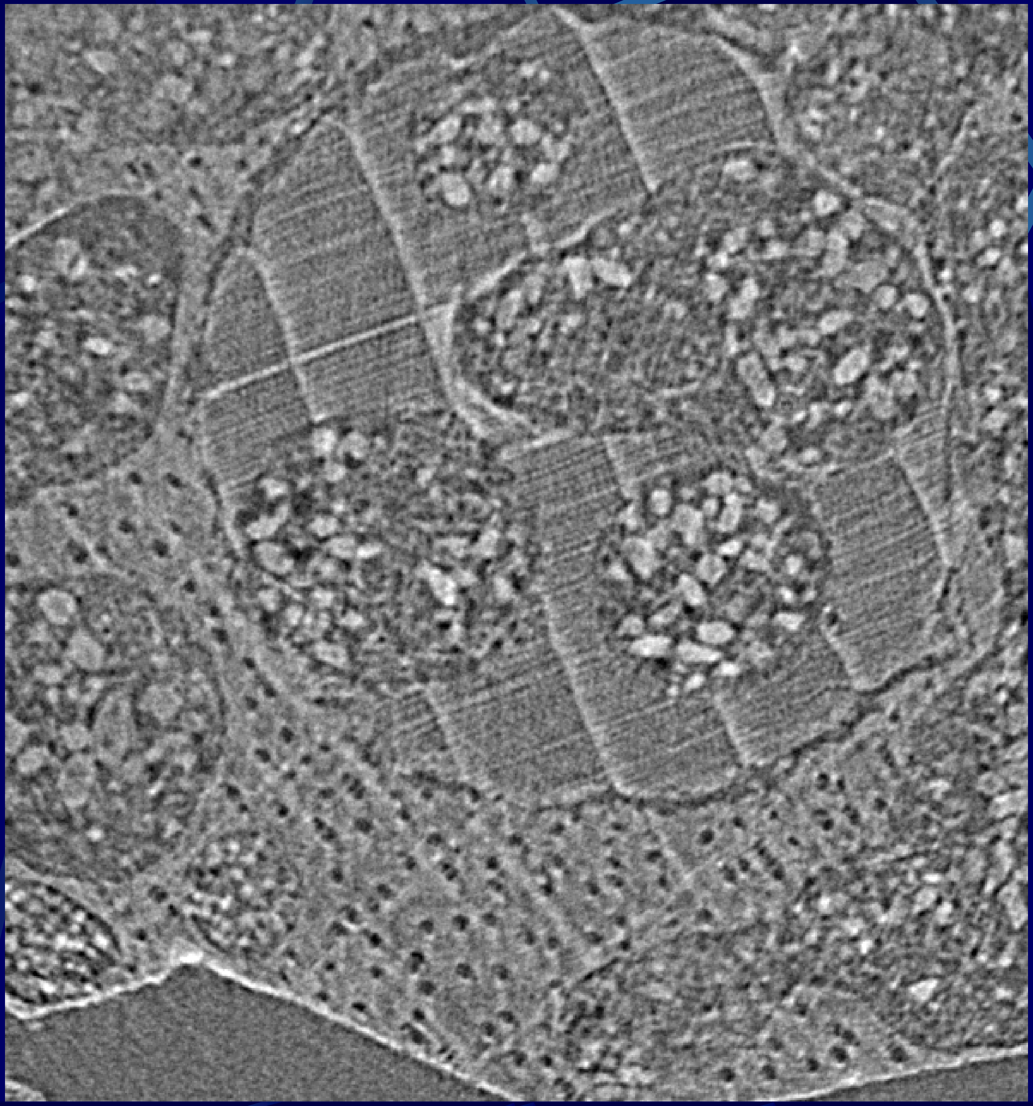
Slice at the wood foot position

The organ by Lorenzo da Pavia



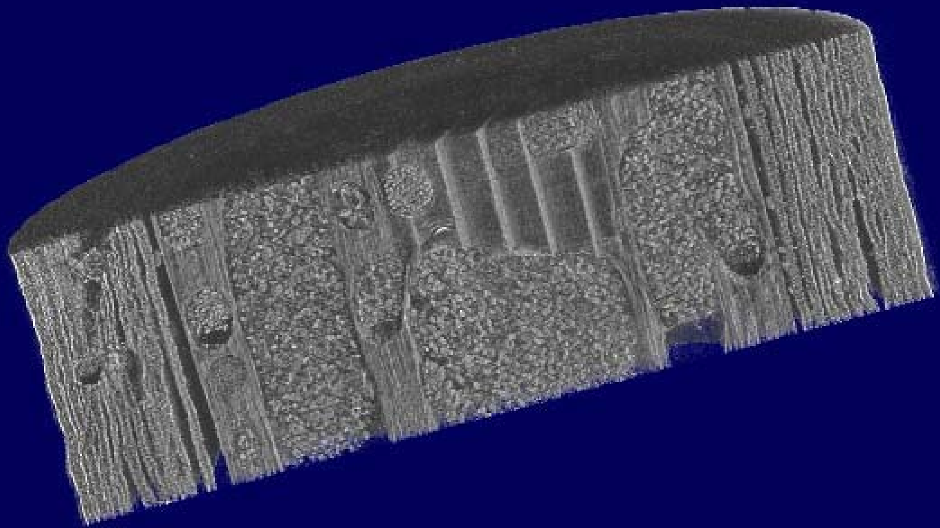
- Ten 0.25 mm layers
- Good quality of the external layer
- Evident degradation of the layers adhesion

The organ by Lorenzo da Pavia



- Possibility of wood species characterization
- Presence of larvae

The organ by Lorenzo da Pavia



Information is three - dimensional and digital, and can be displayed in several ways

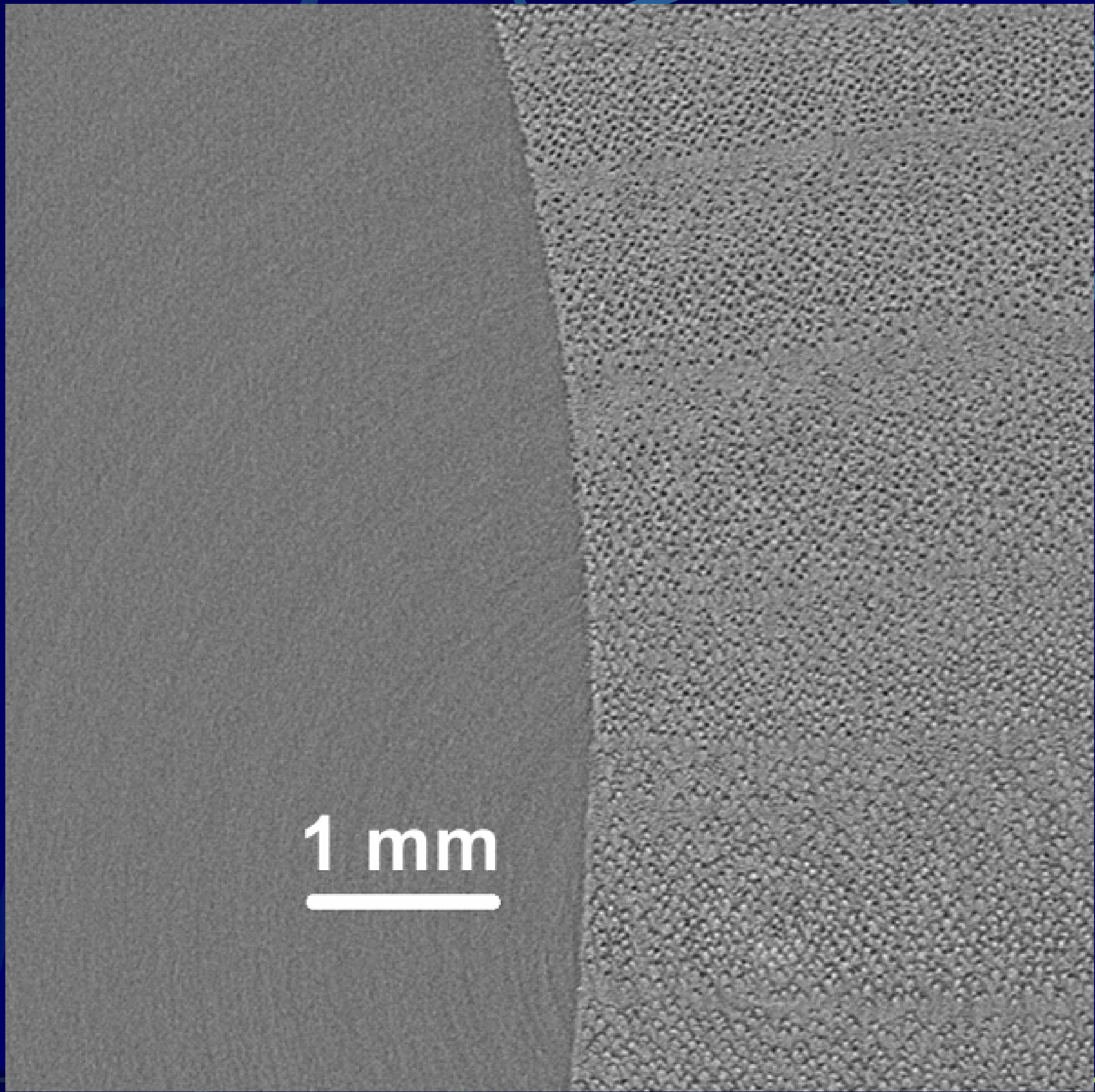
The recorder by J.C. Denner

Wooden recorder by J.C. Denner (beginning XVIII century)

Structural characterization (restoration, conservation, previous restoration)

Instrument of great historical and artistic relevance

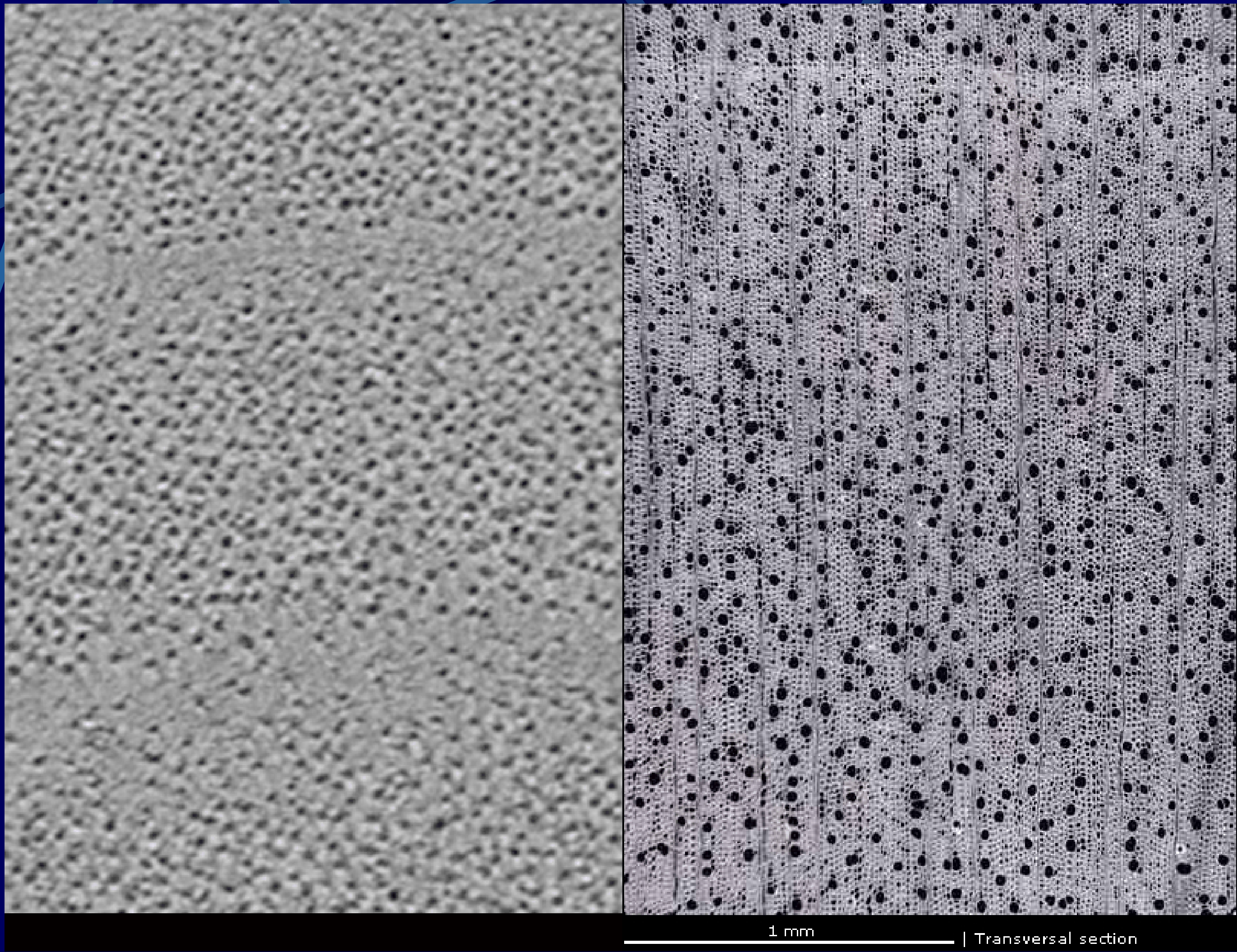
The recorder by J.C. Denner



Sample of great dimensions
Length 100 cm
Max diam. 7 cm

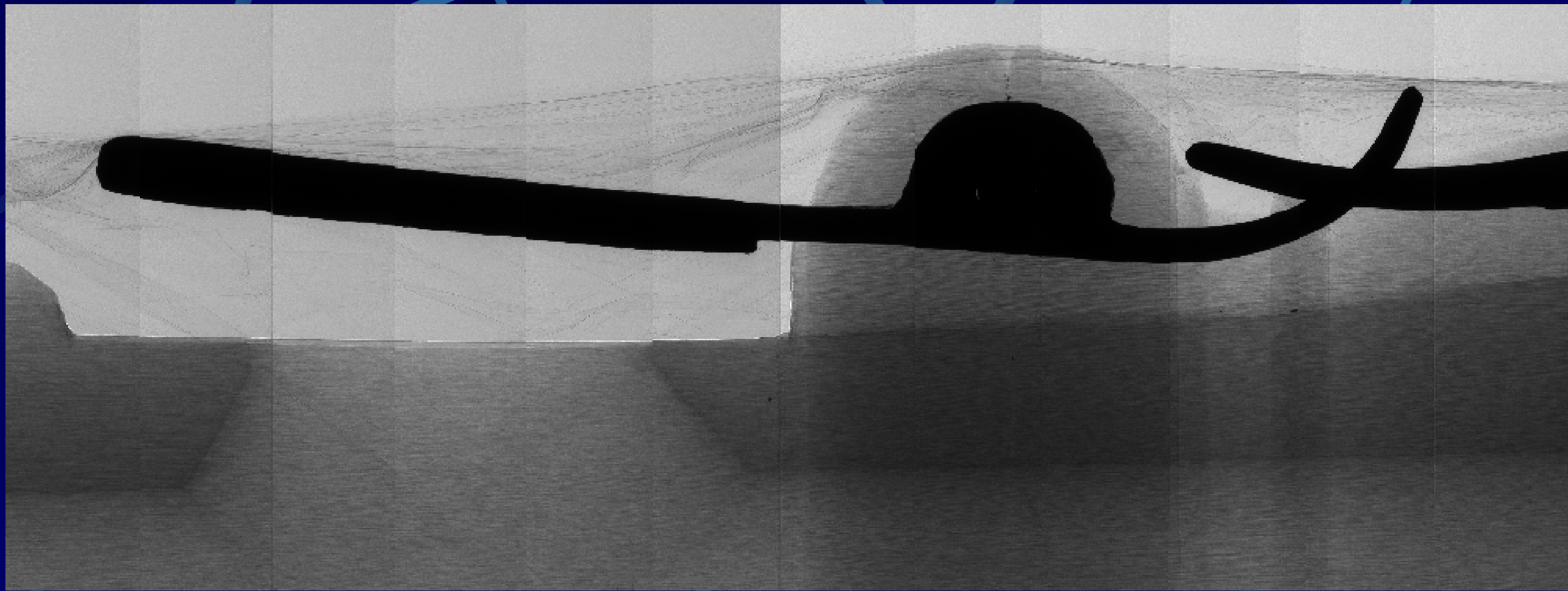
Necessity of *local area tomography* techniques

The recorder by J.C. Denner



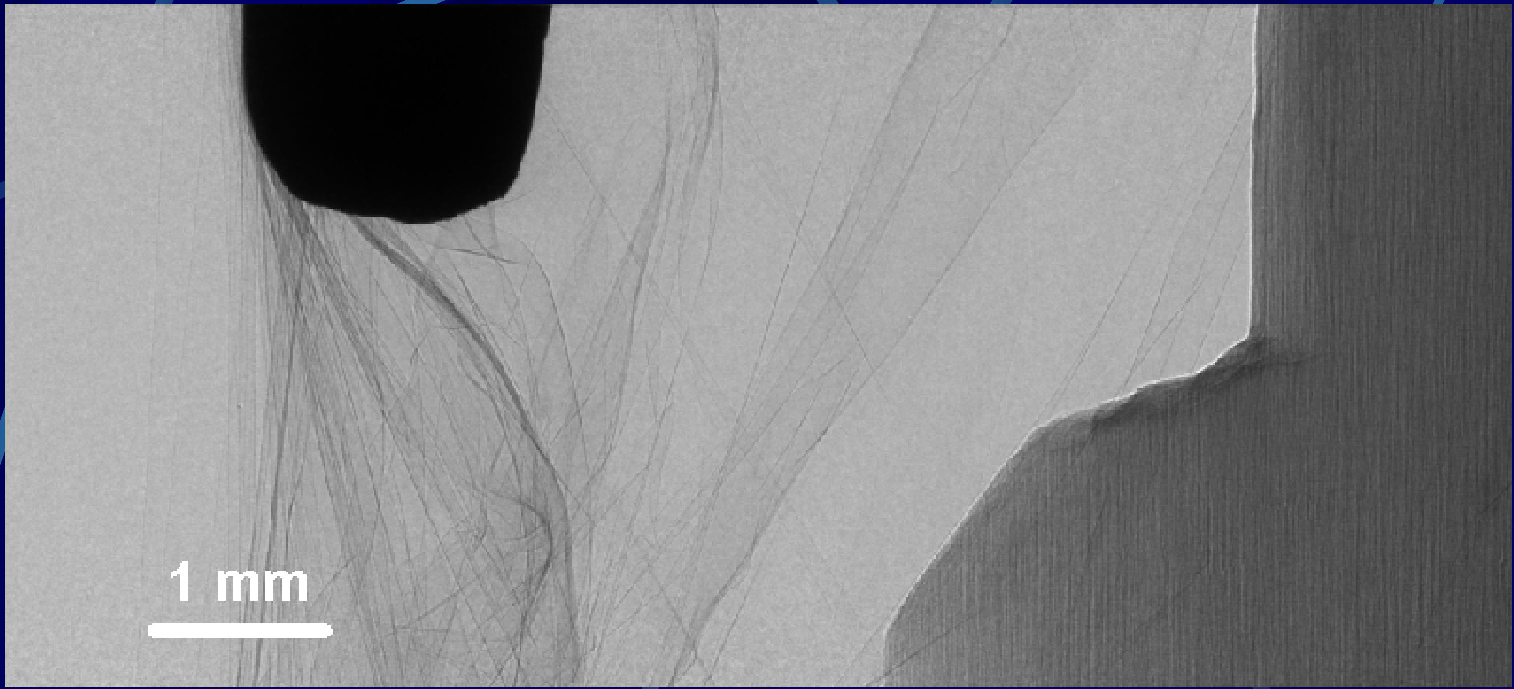
Comparison between tomographic data and a microscopic scan of boxwood

The recorder by J.C. Denner



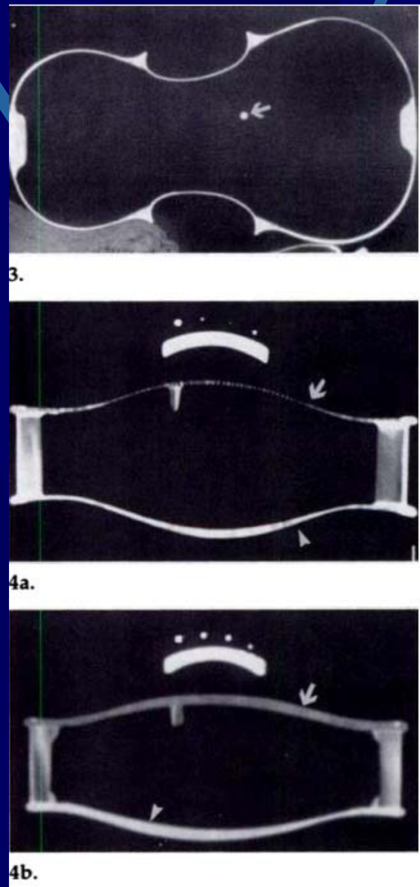
Planar scan of the recorder at the brass key (full dimension of the image: 5 cm (H) x 2 cm (V); spatial resolution of 9 micron)

The recorder by J.C. Denner



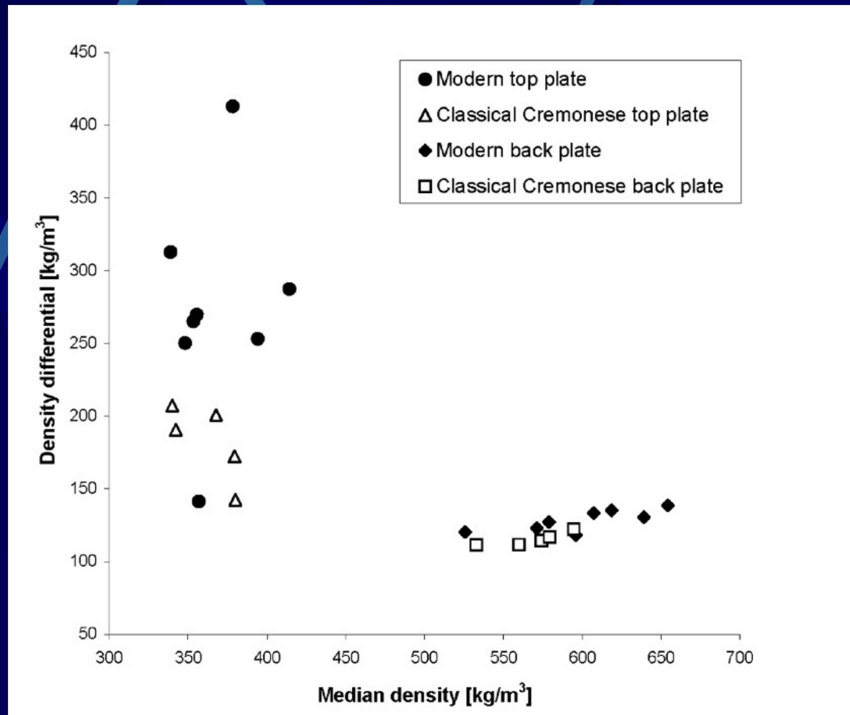
Detail of the recorder at the terminal part of the brass key. The veil that can be seen is due to the thin Parafilm layer used to protect the instrument during the data acquisition.

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.



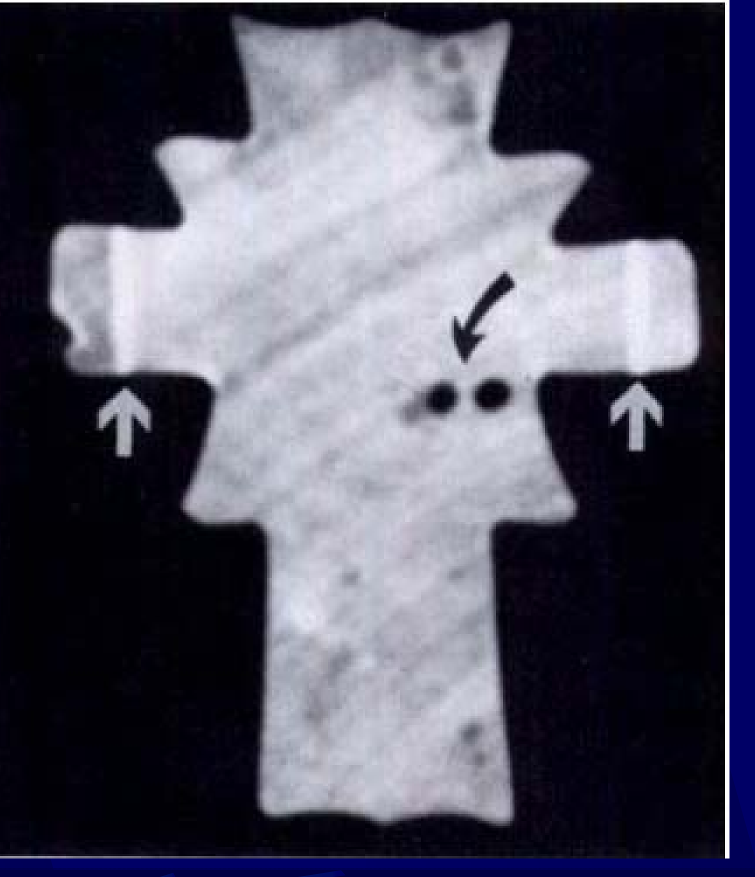
S.A. Sirr, J.R. Waddle, Radiology, 1997, **203**, 801

Clinical CT has also been used to measure the **density differences** between different points of violins in order to explain the acoustic differences between classical and modern violins.

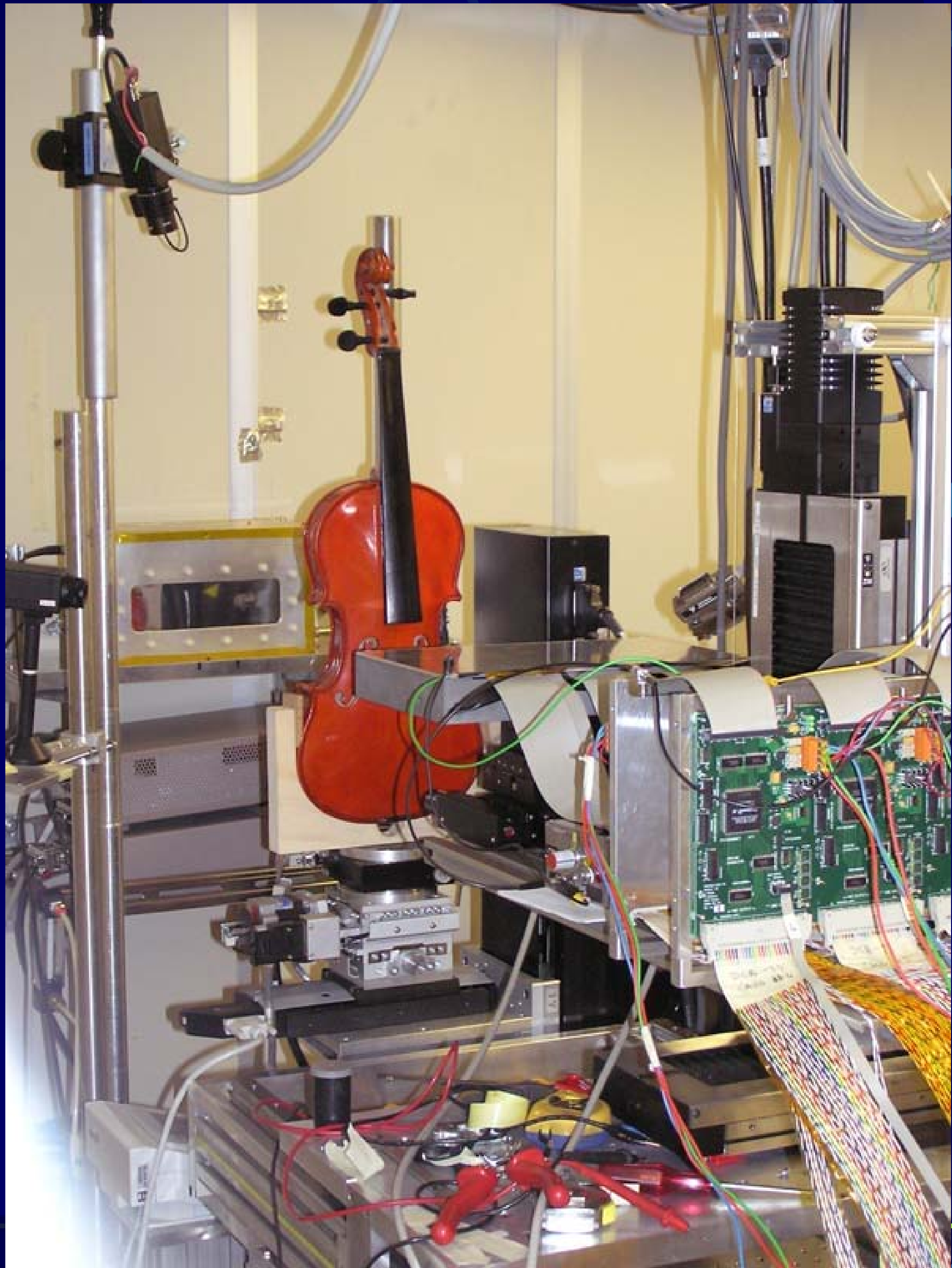


B.C. Stoel, T.M. Borman, PLoS ONE, 2008, **3**, 1 1

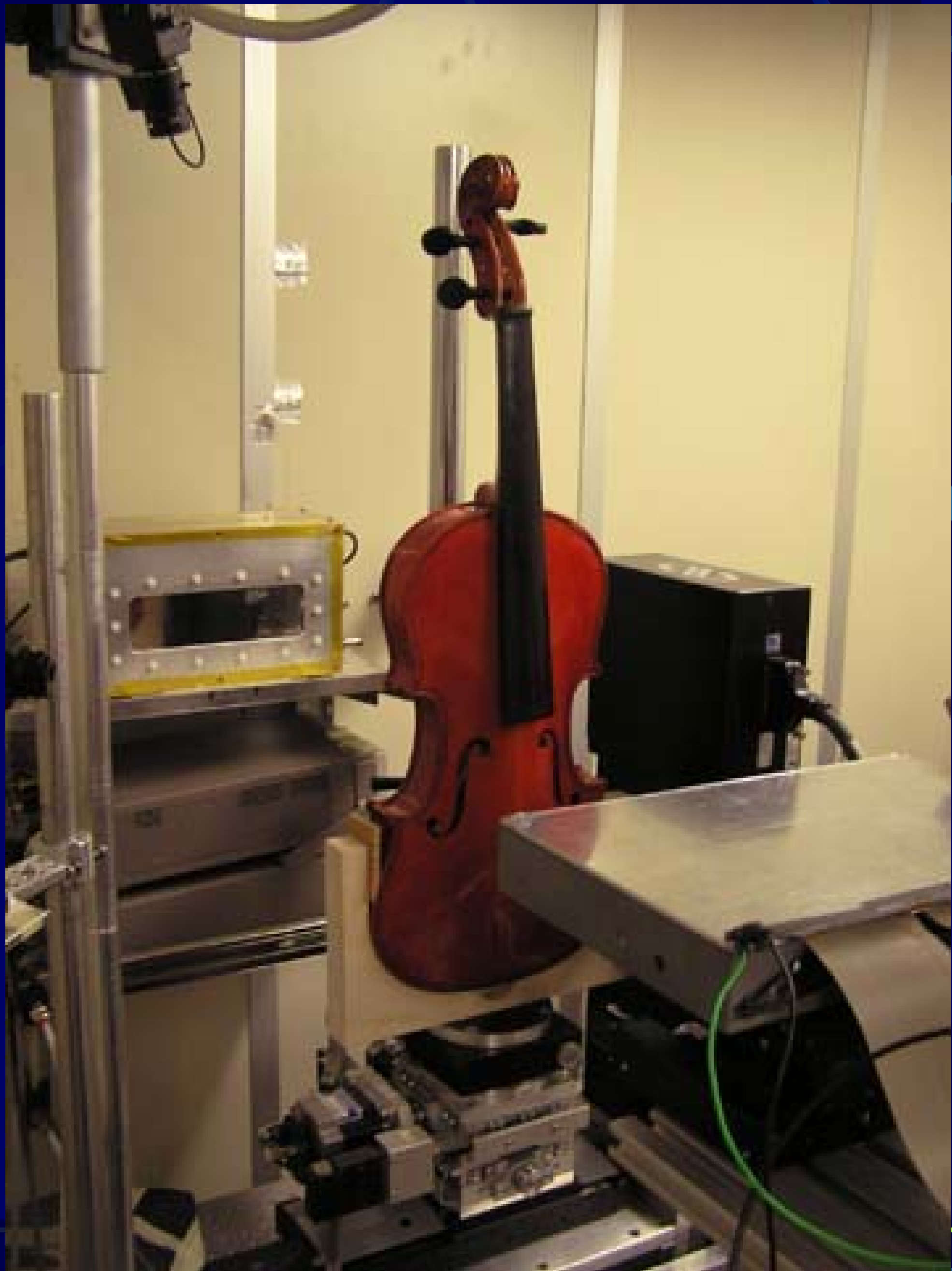
The main limitation in the application of clinical CT to the structural analysis of bowed instruments, however, is the **limited spatial resolution** of commercial instruments, (0.4x0.6x0.6 mm^3). Every defect with lateral dimensions smaller than this value **cannot be detected** with state-of-the-art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



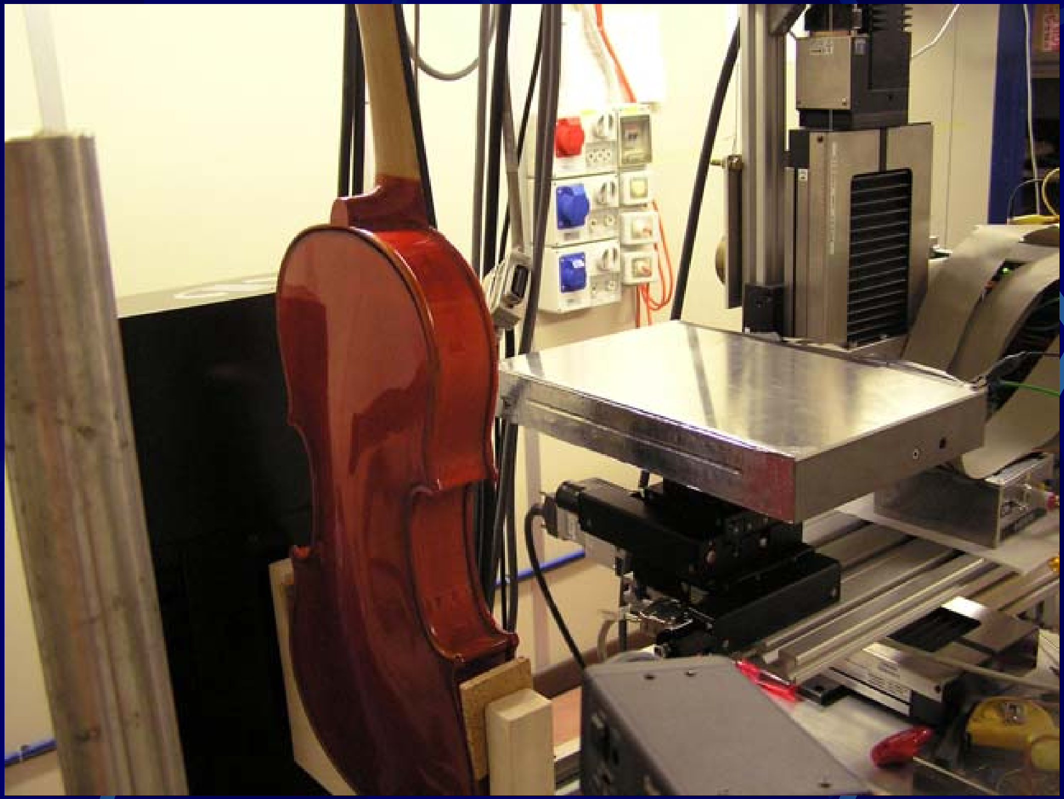
S.A. Sirr, J.R. Waddle, Radiology, 1997, **203**, 801



Advantages of synchrotron-radiation microtomography are well known, and include the choice of the ideal X-ray energy as a function of the sample, the reduction of acquisition times, the possibility to apply phase-contrast techniques in order to detect low-absorbing materials.

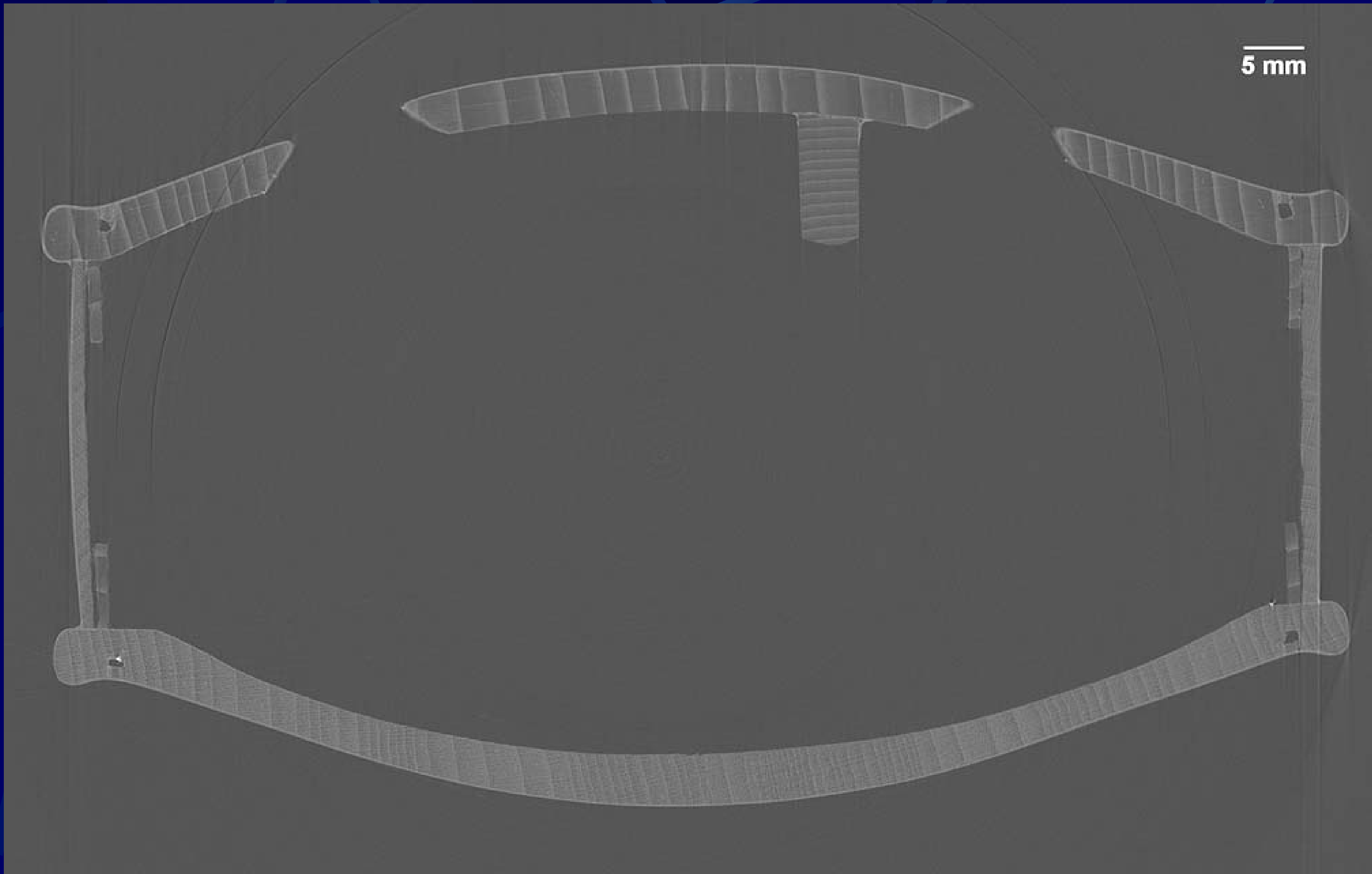


The main limitation in the use of synchrotron radiation is related to the reduced dimensions of the samples under investigation. The development of new X-ray detectors designed for the particular characteristics allows the researchers to overcome this kind of problems.

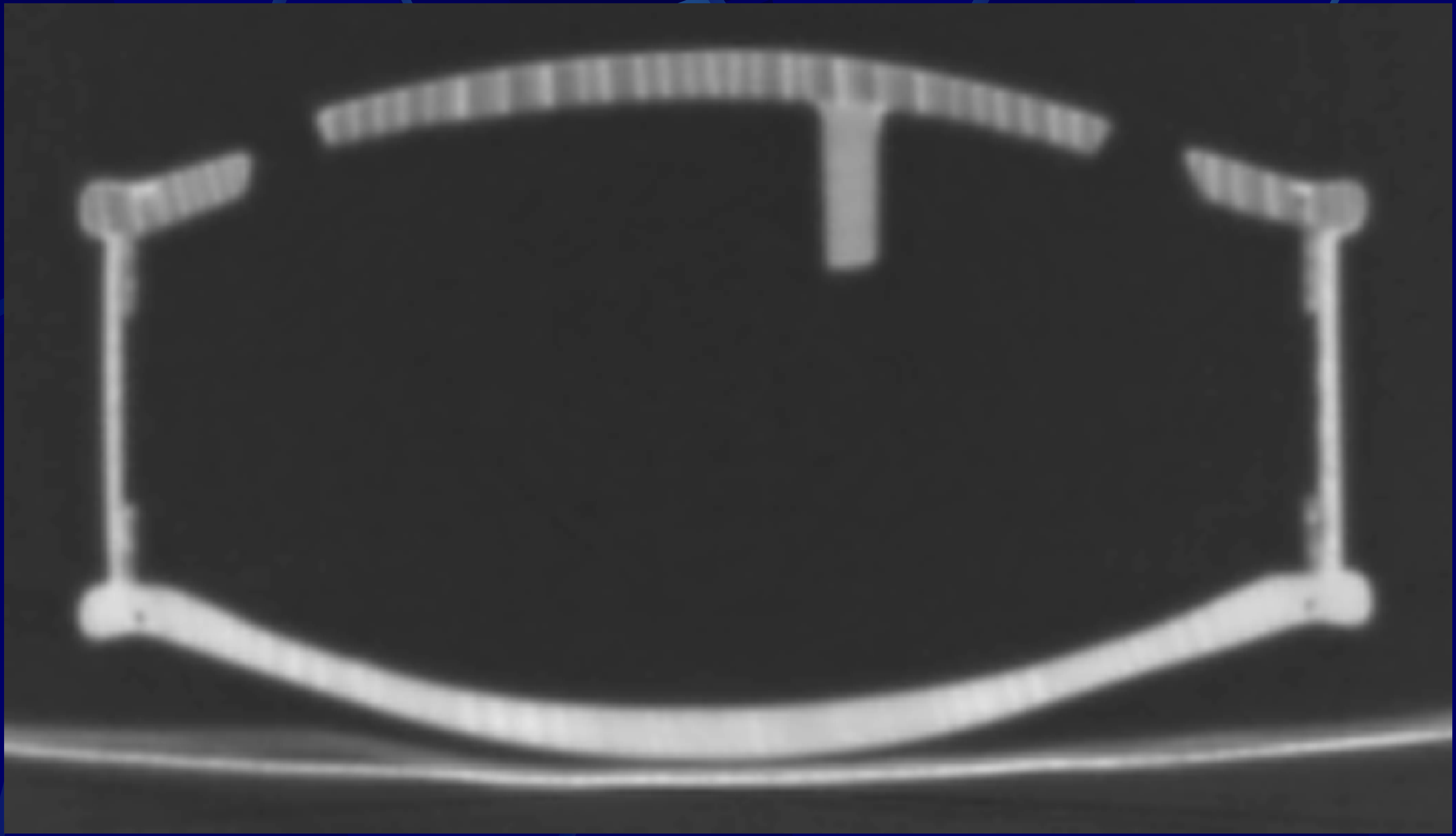


PICASSO (Phase Imaging for Clinical Application with Silicon detector and Synchrotron radiation) has been developed by the Istituto Nazionale di Fisica Nucleare (INFN). It is a silicon microstrip detector in “edge-on” configuration.

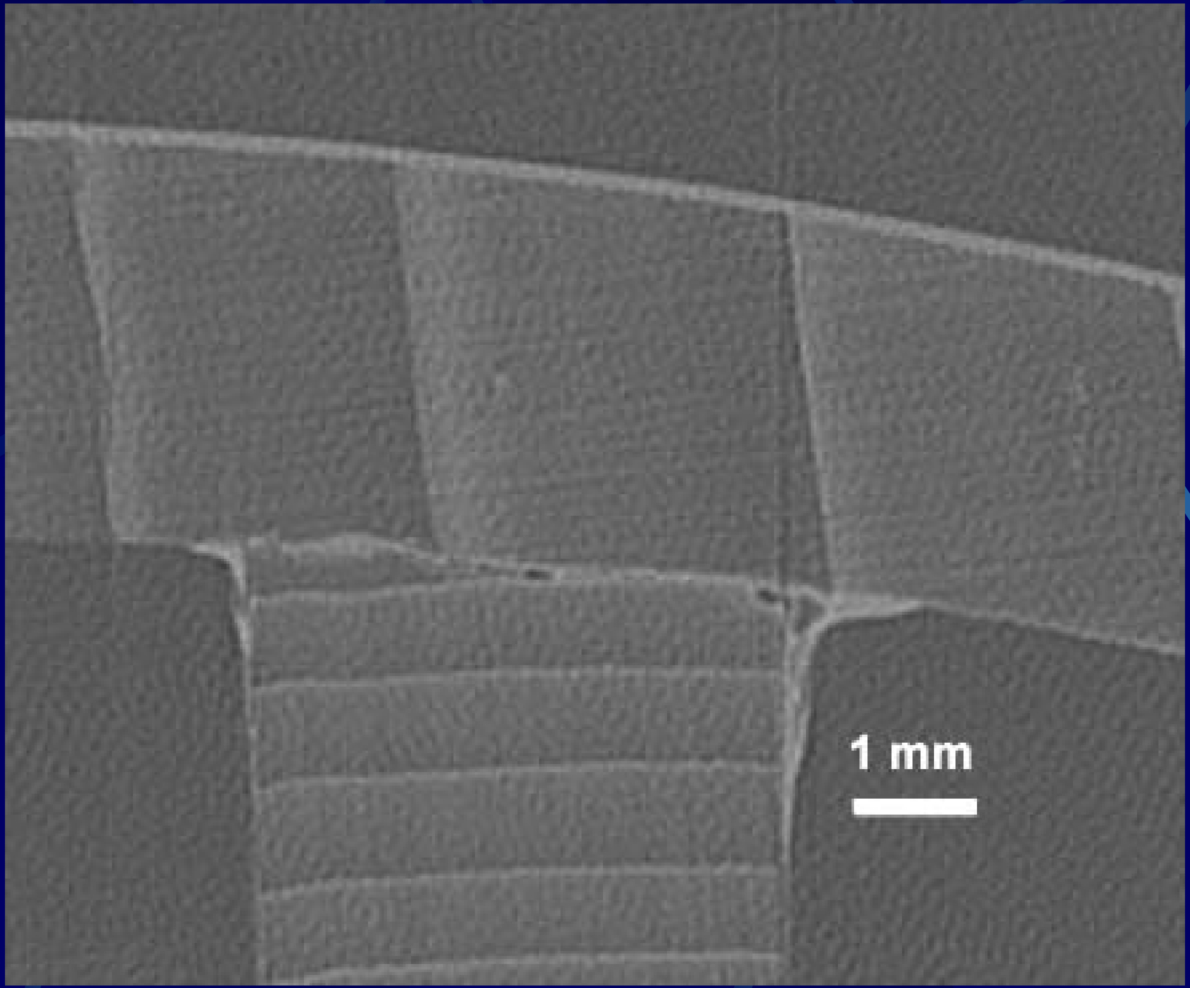
The aperture of each pixel is determined by the strip pitch (0.05 mm, H) and the sensor thickness (0.3 mm, V). The detector is operated in single-photon counting and it is read out by a high-rate electronics based on the Mythen-II application-specific integrated circuit (ASIC). Each pixel is wire-bonded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



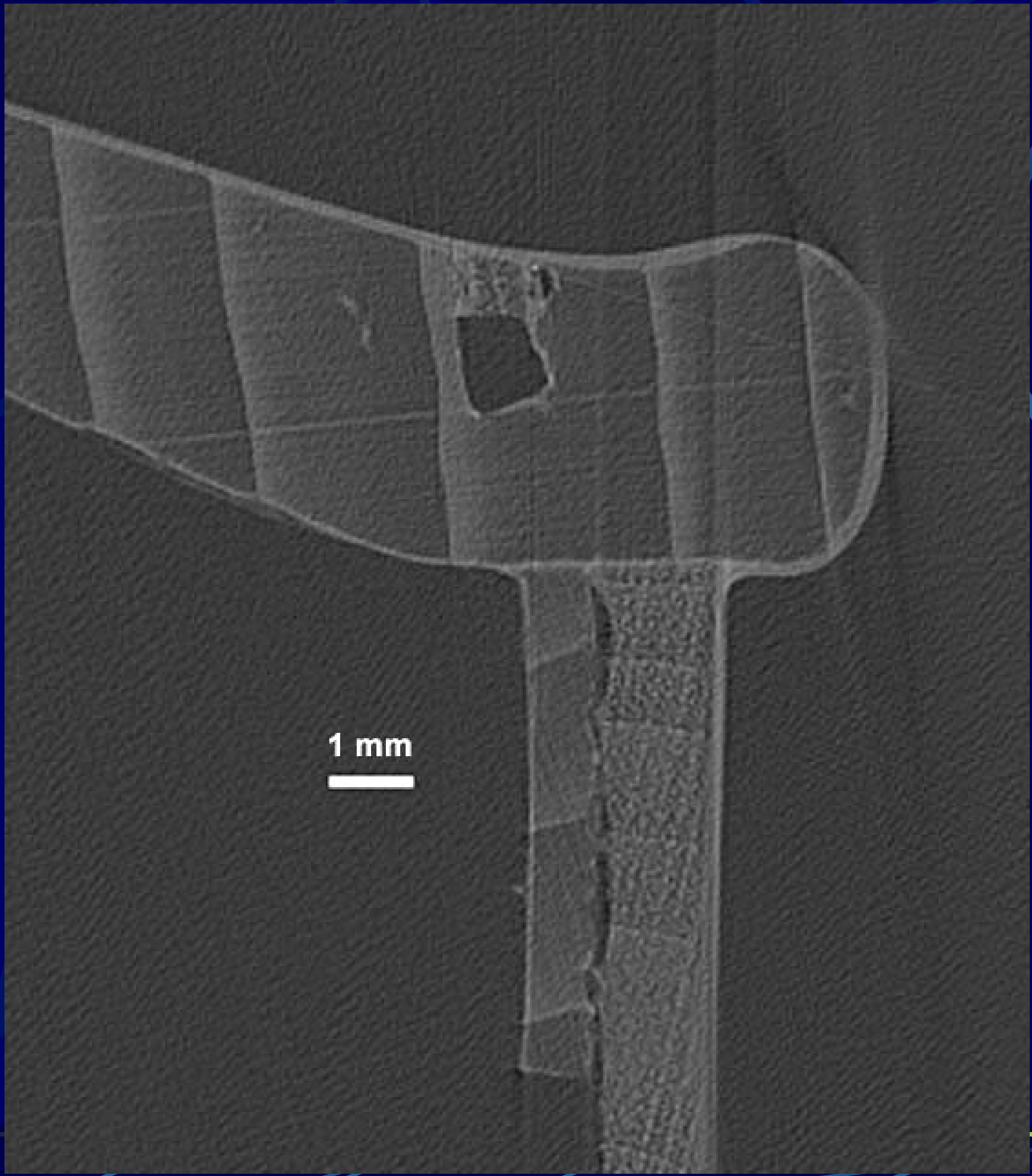
Transaxial μCT scan of a student violin taken at SYRMEP
(E = 23 keV, acquisition time = 1s, 3600 images)



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)



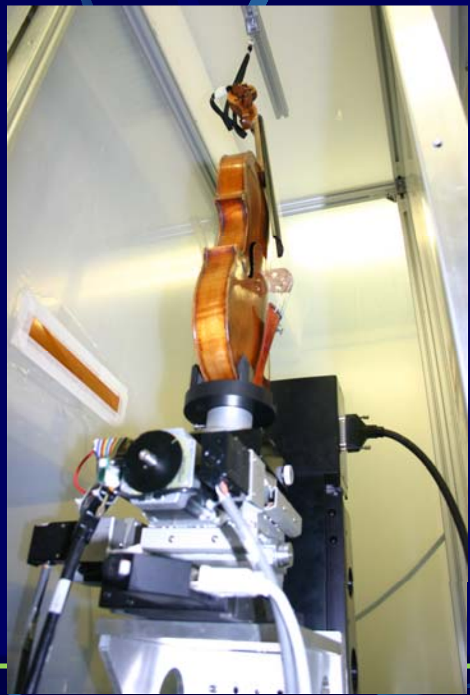
Detail showing the bass bar and the glue used
to attach it to the front plate.

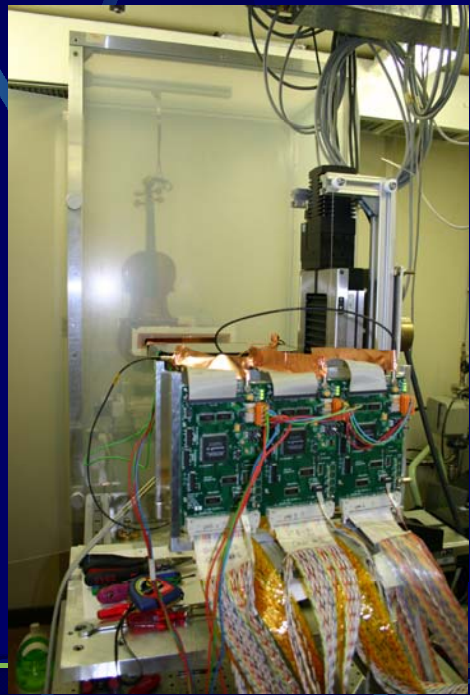


Detail showing the front plate, one rib and the relative lining

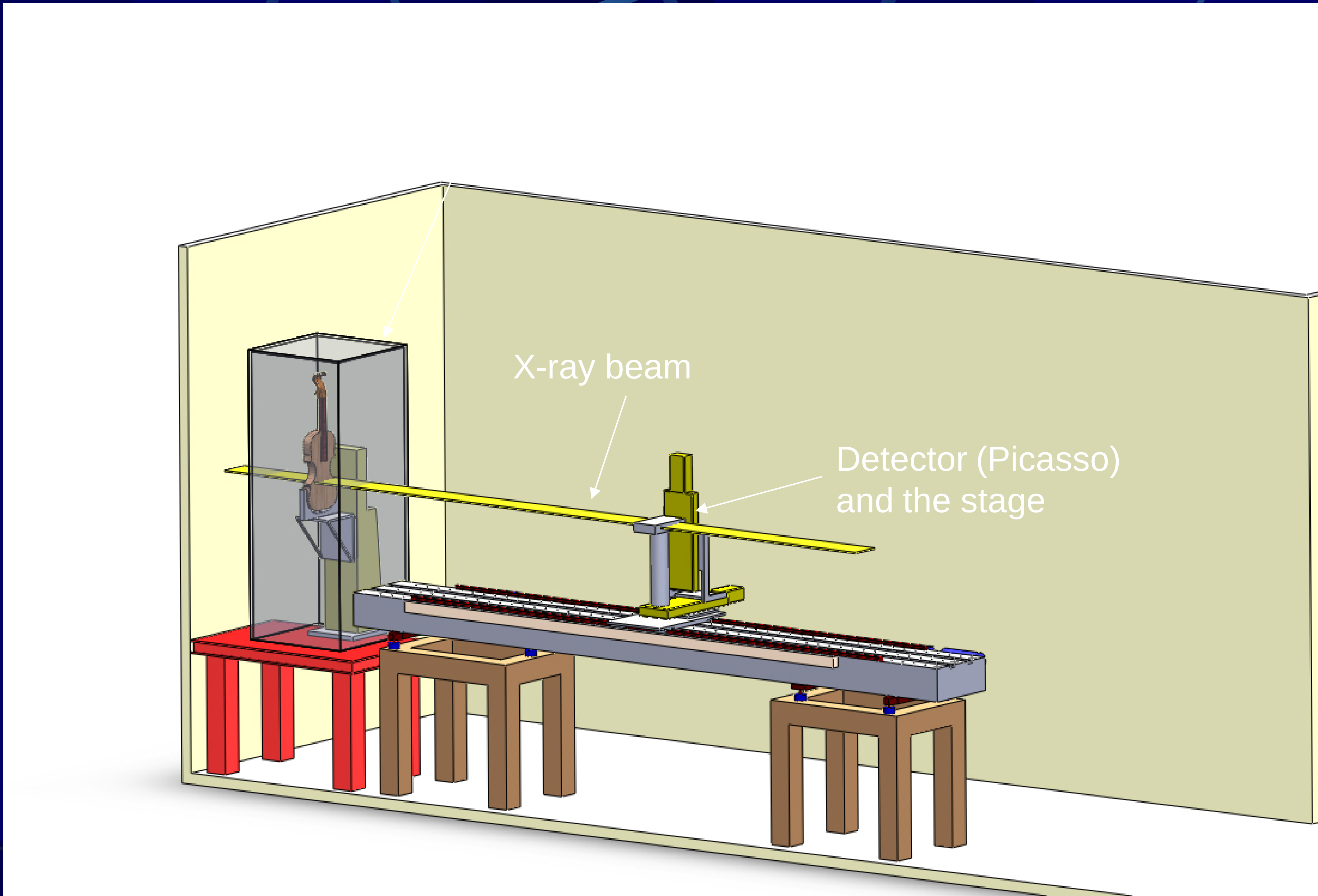
Peter Herresthal and his Giovanni Battista Guadagnini (1753)

Peter Herresthal and his Giovanni Battista Guadagnini (1753)

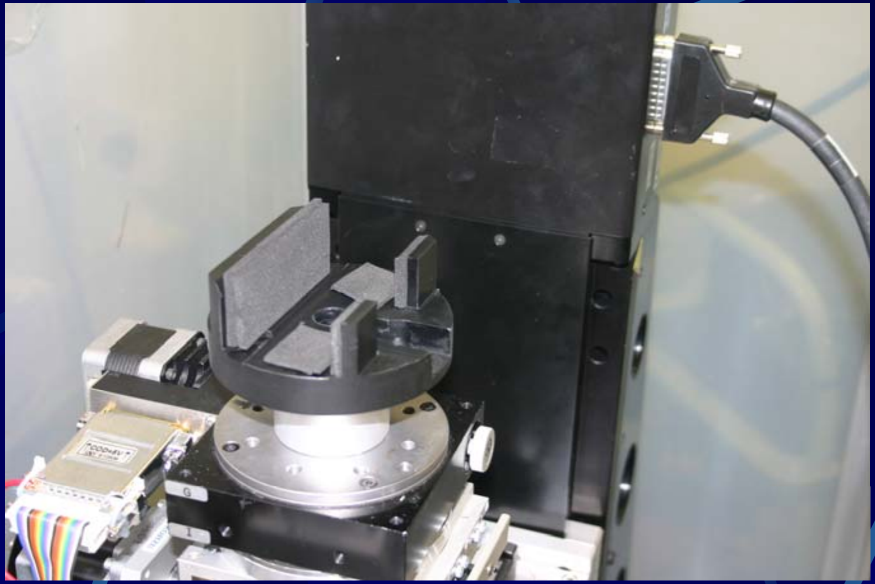




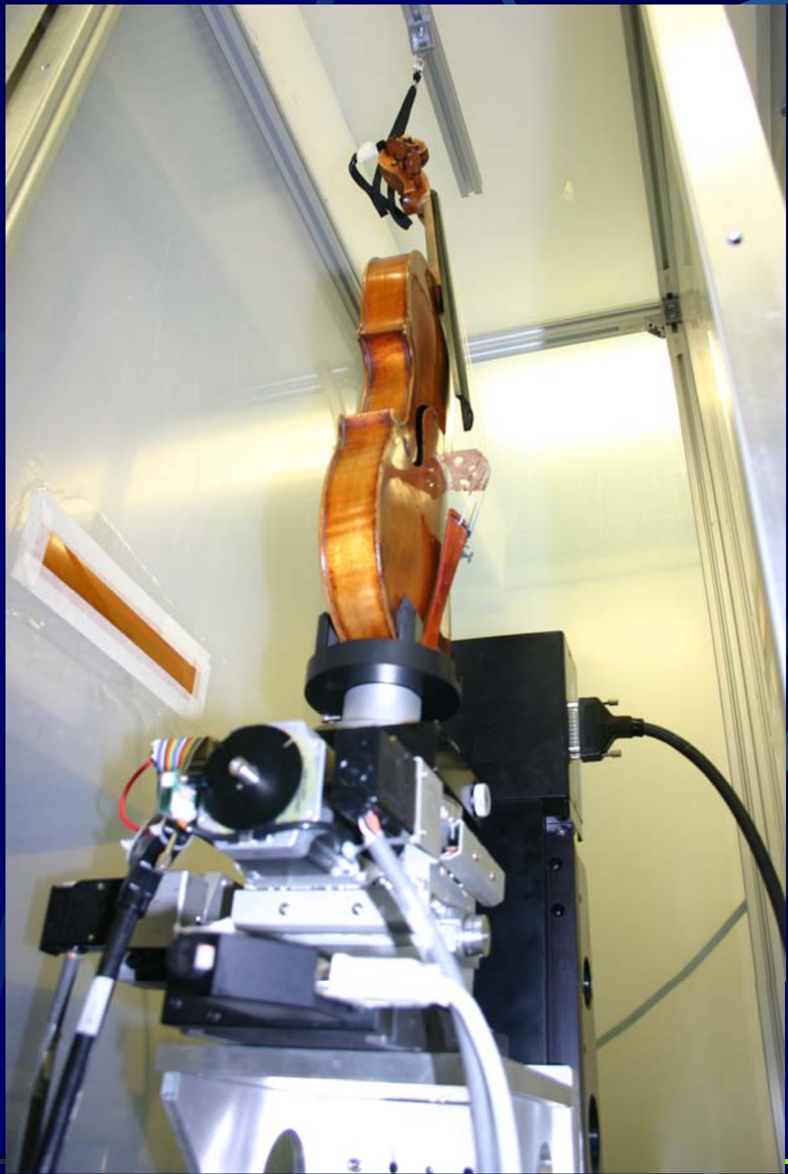
The experimental hutch

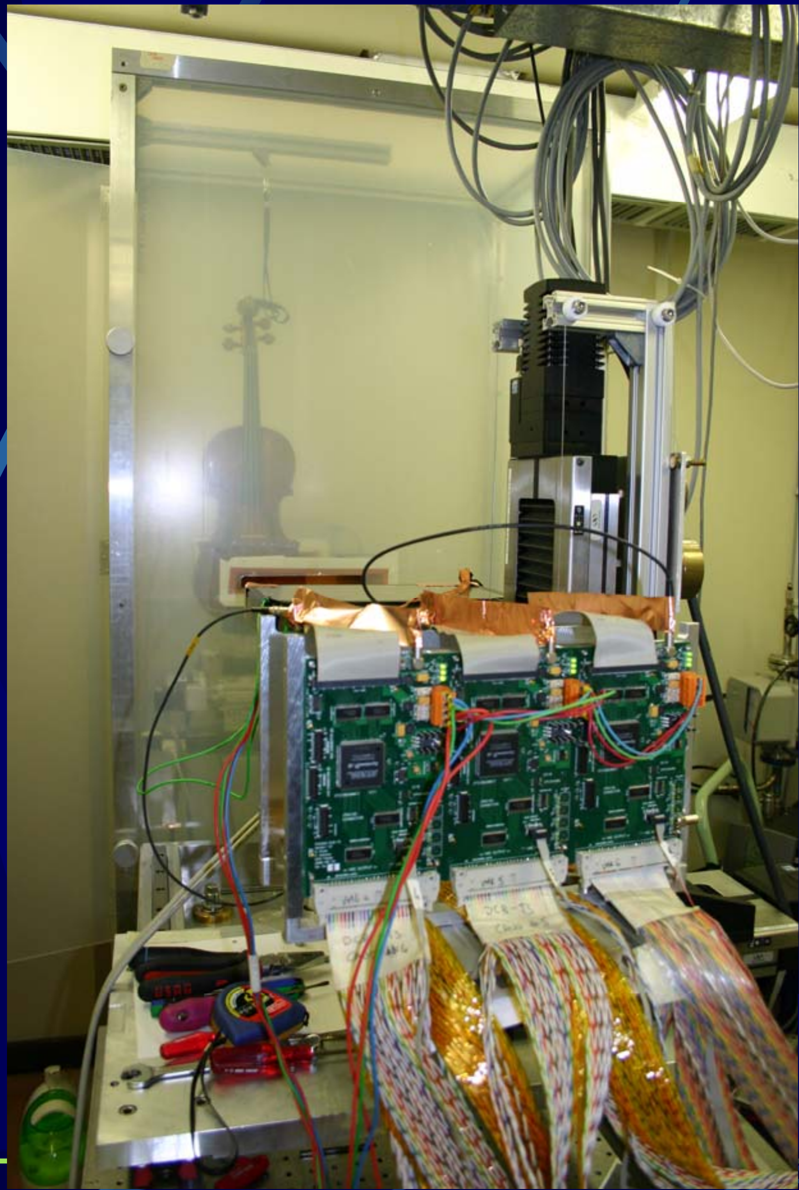


The experimental hutch

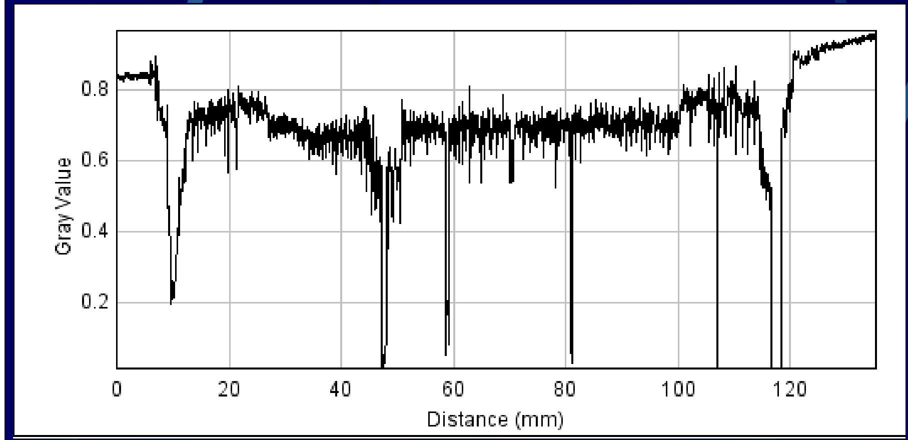


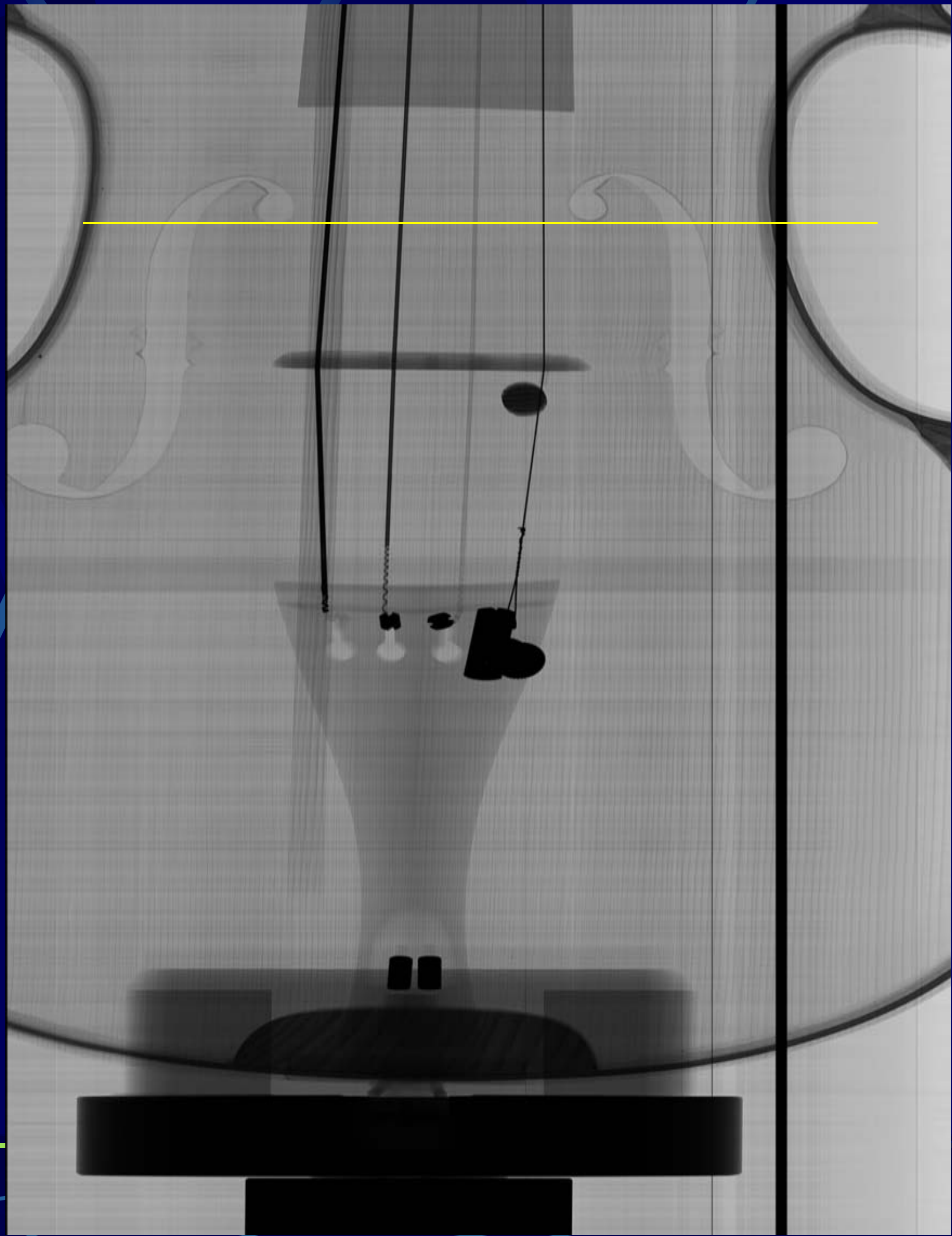
The experimental hutch



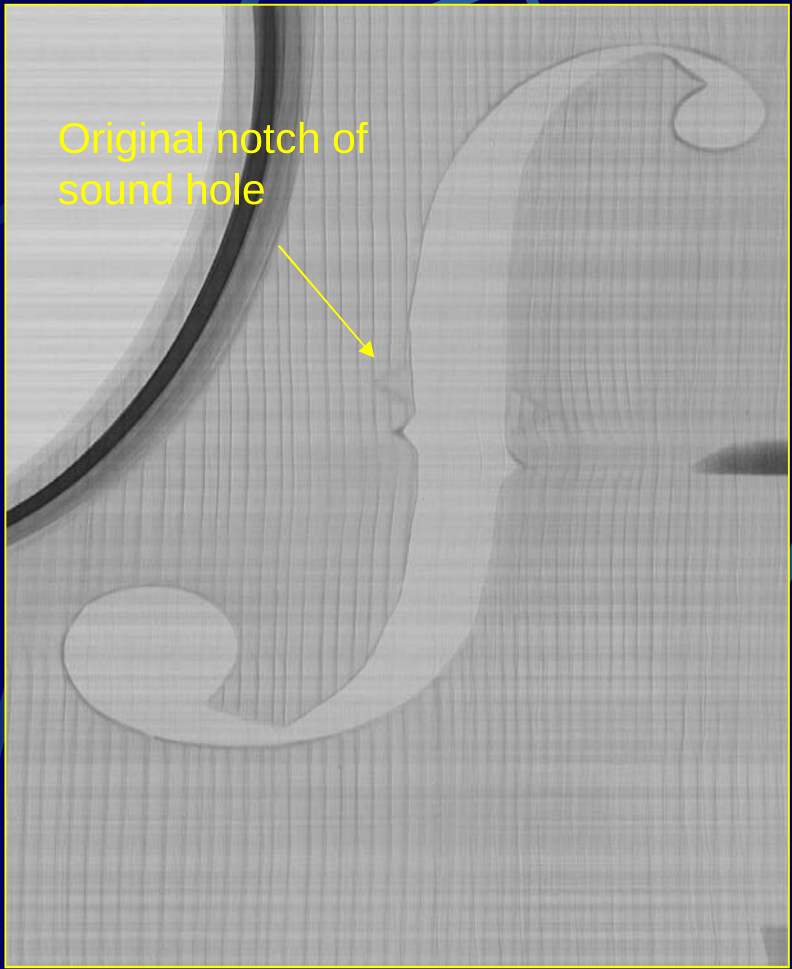


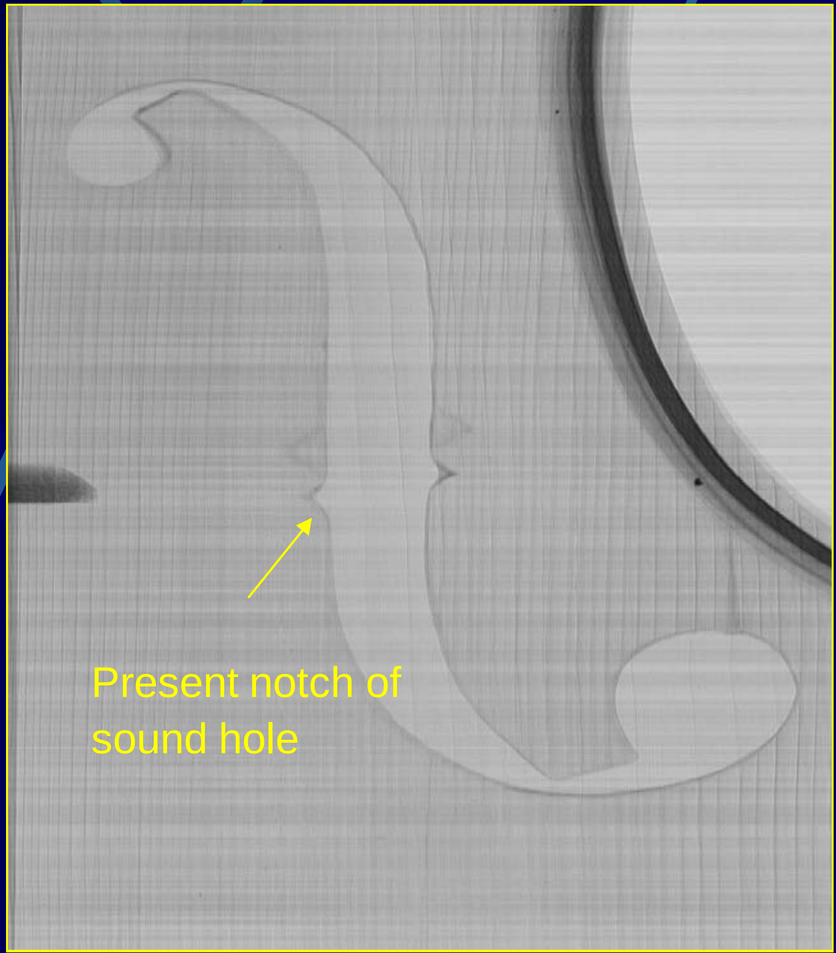
The planar image

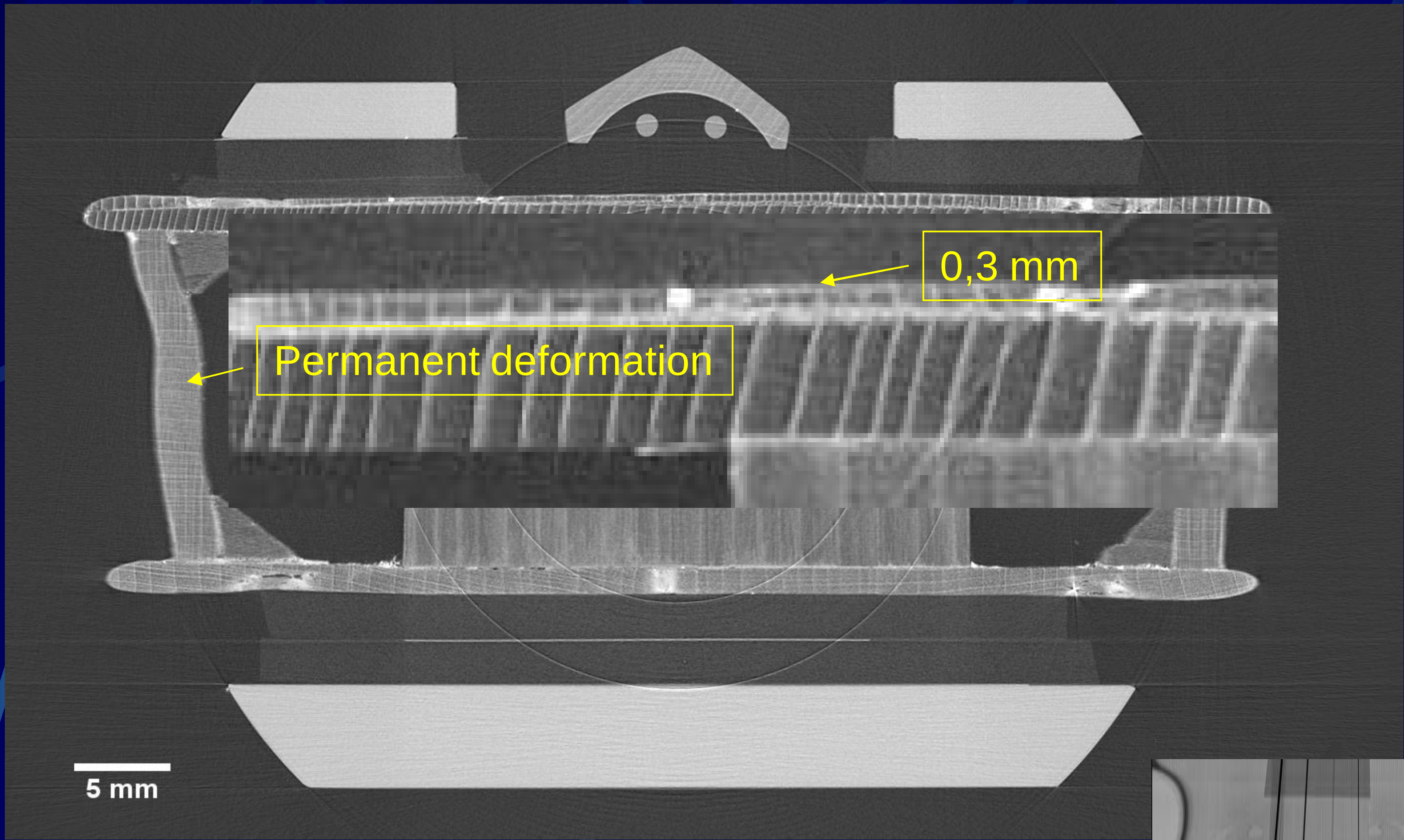


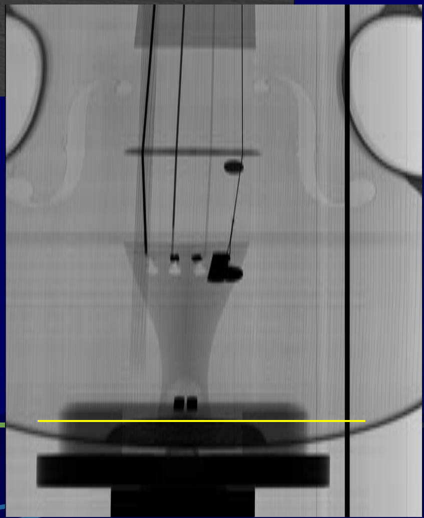


The planar image





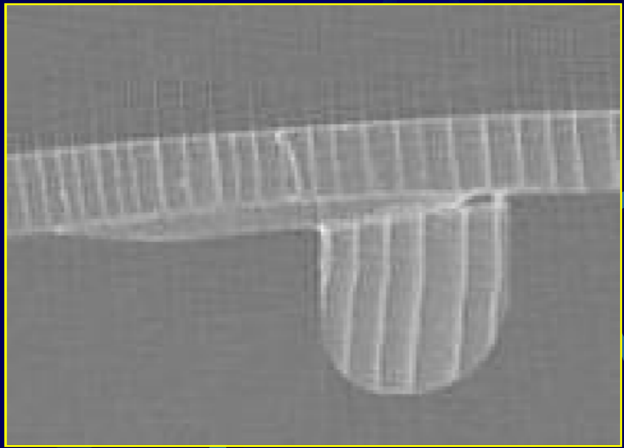


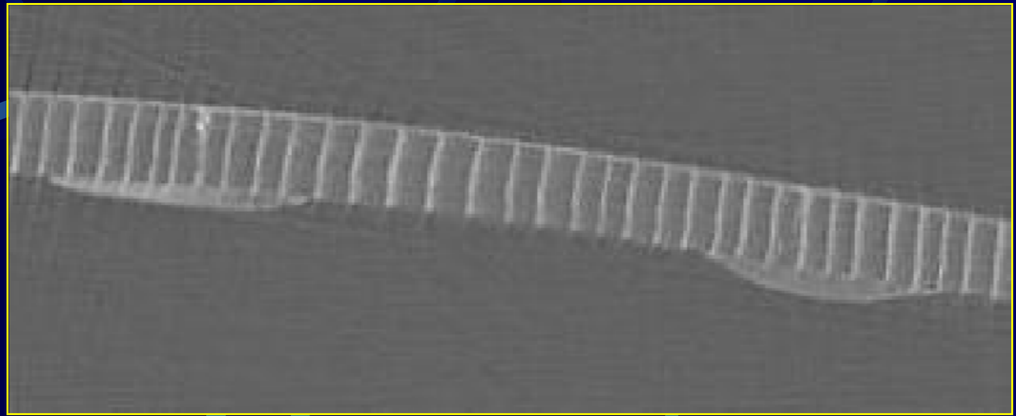


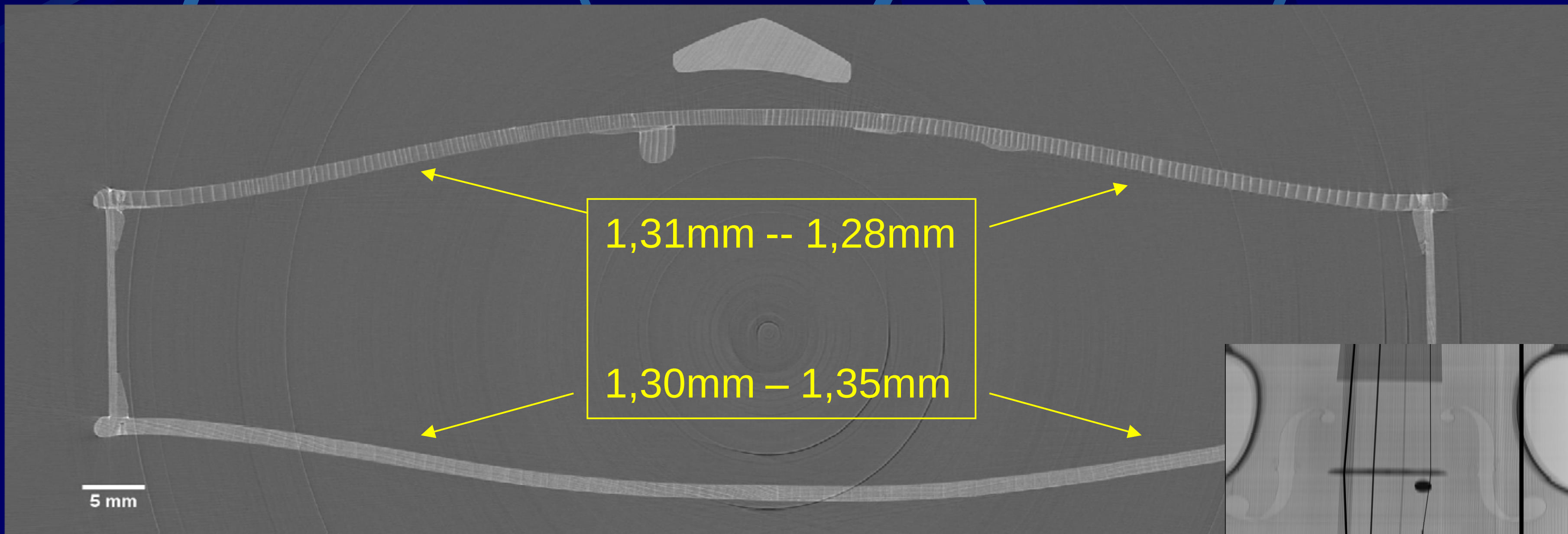
- The original top plate is very thin, *expecially* on the left side;

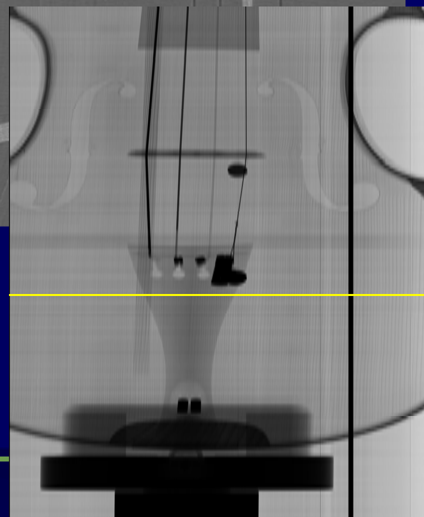
-The grain of the patch below the top plate is not straight (probably obtained by cutting and not by splitting);

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

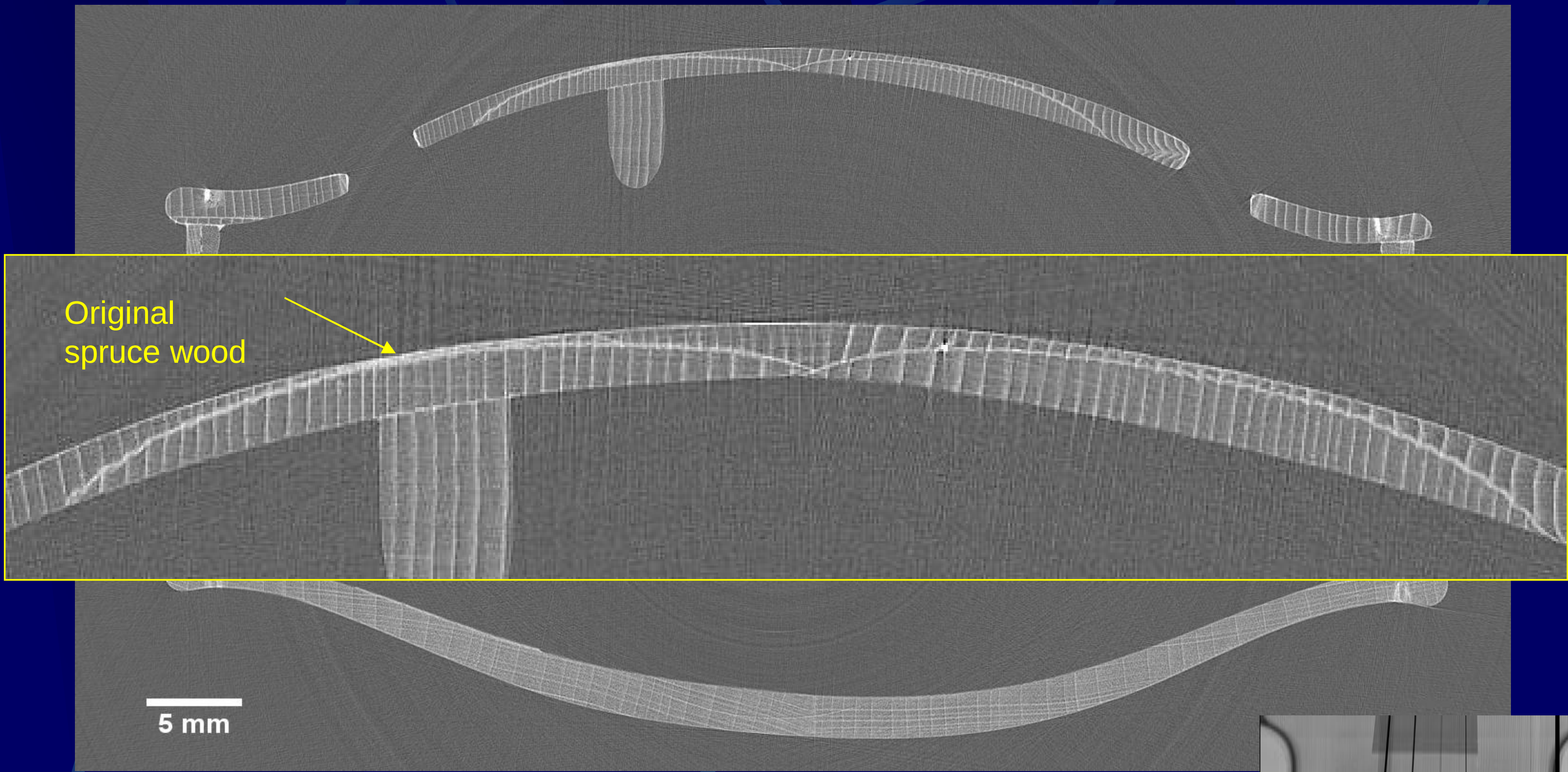


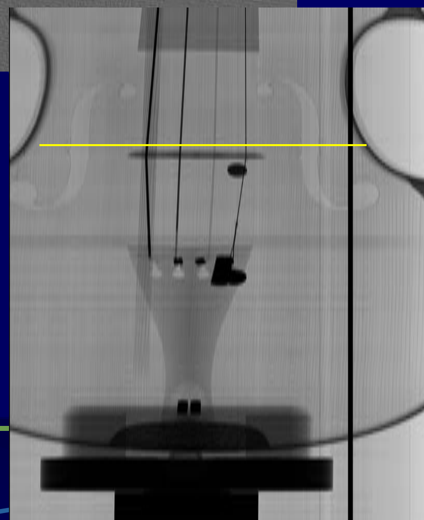




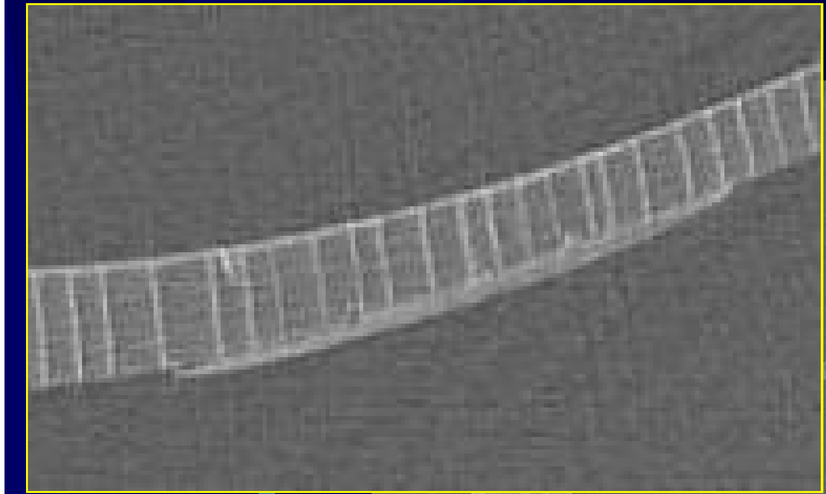


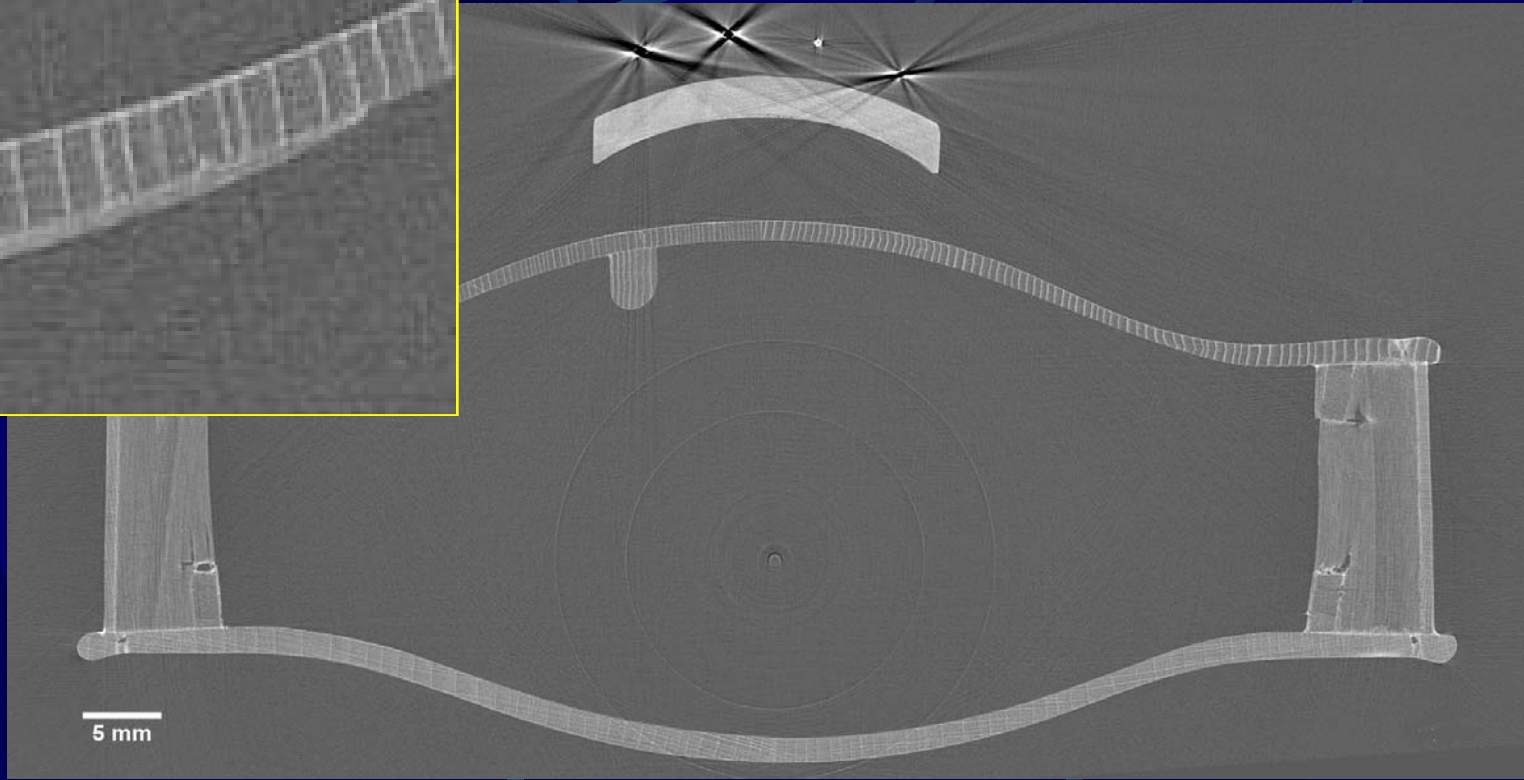
- Thickness of top and back plate
- Bass bar is glued on a patch;
- Two patches on the right part of top plate.



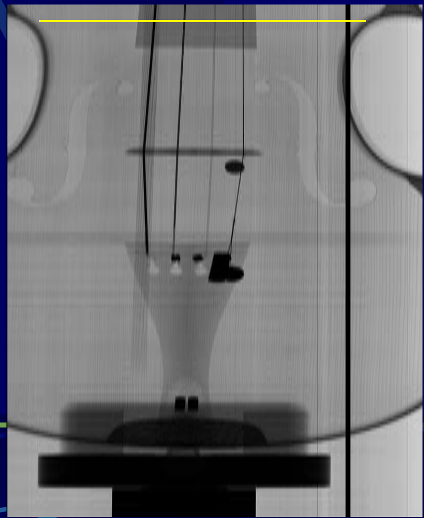


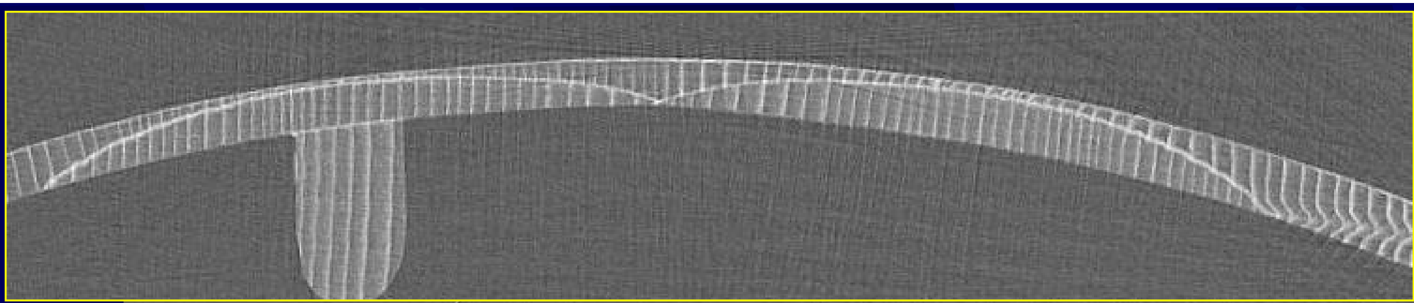
- Two patches are glued on the top plate;
- The grain of the patches is good:
- Thickness of the back is very thin (mm)

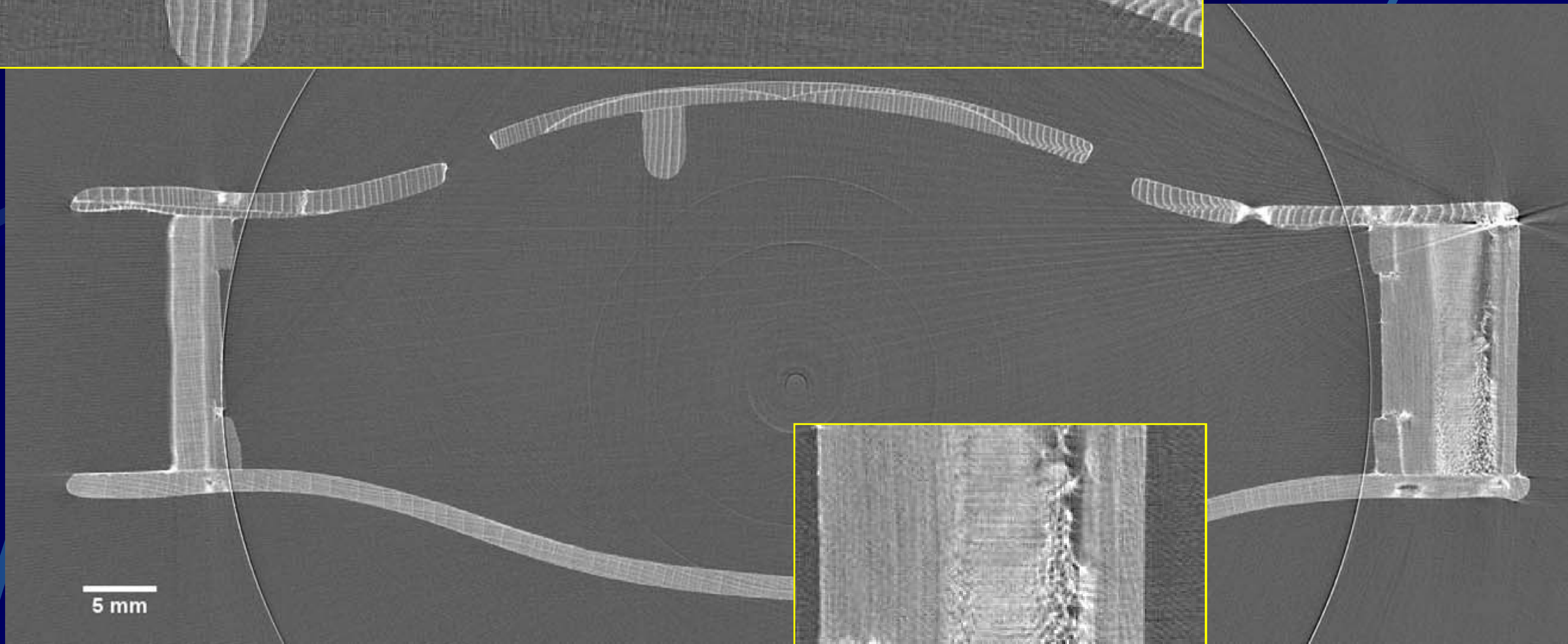


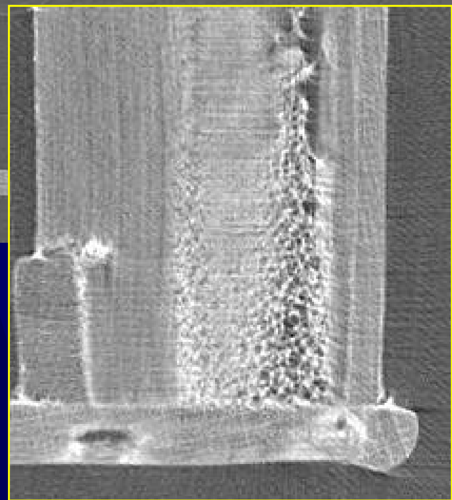


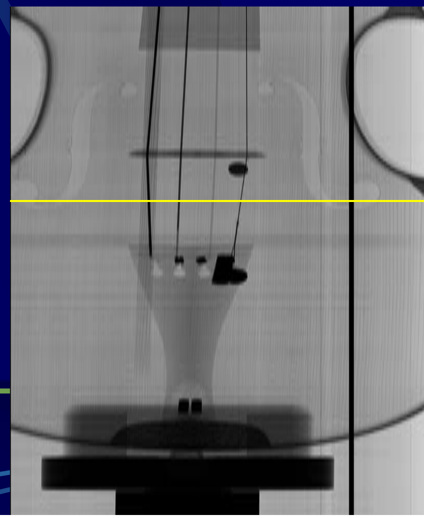
- The front and back plate are highly arched;
- Reduced thickness of top plate (~1mm);
- A very thin patch (probably paper) on the left side of top plate



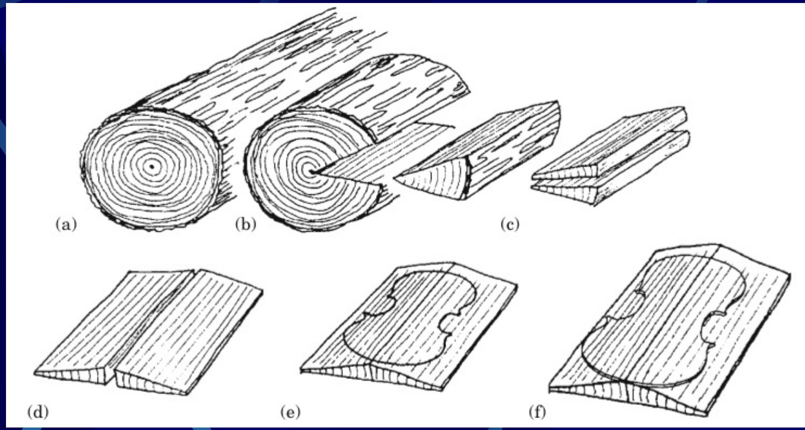








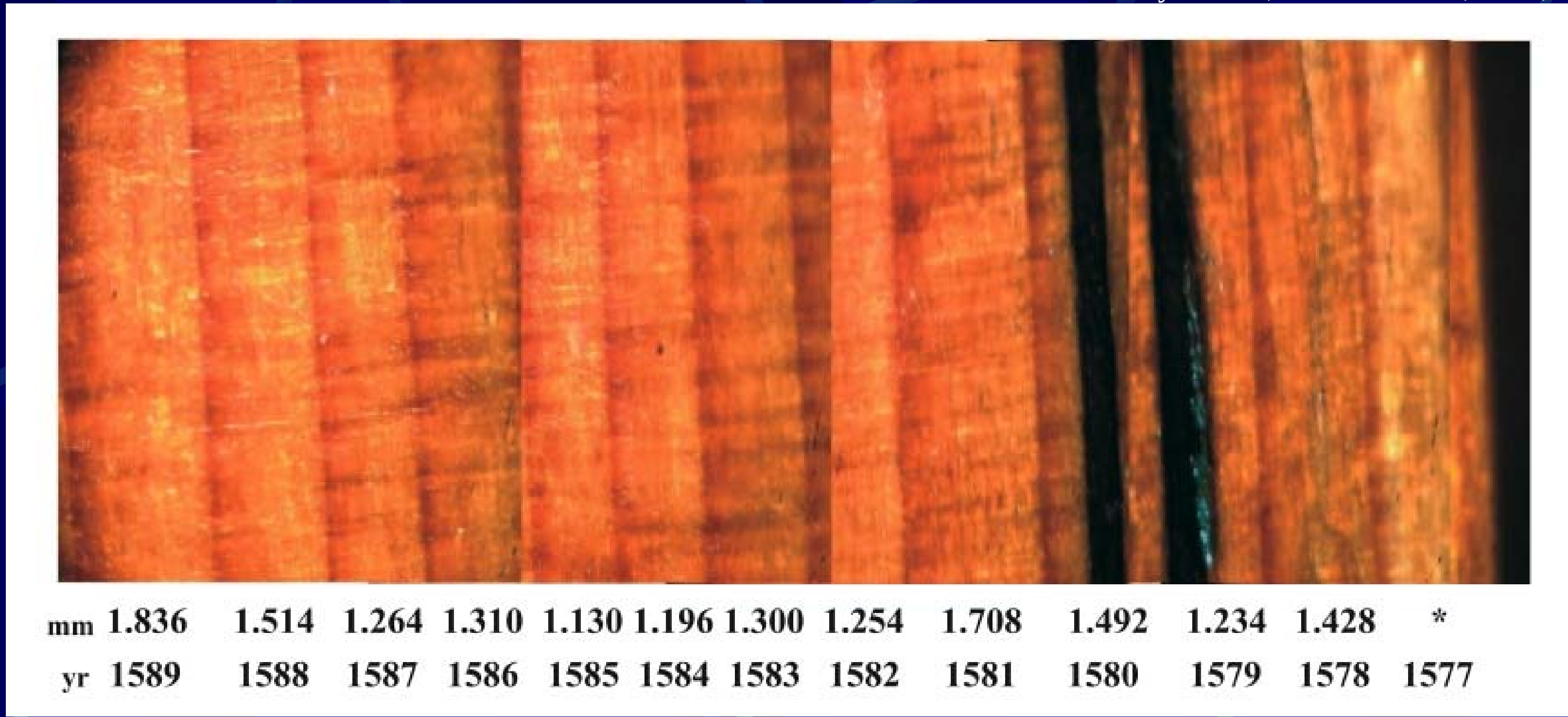
- Reinforcement patch on the left lining;
- Presence of filler;
- Woodworm between the corner block and rib on the right;
- Good quality patches on the top plate.



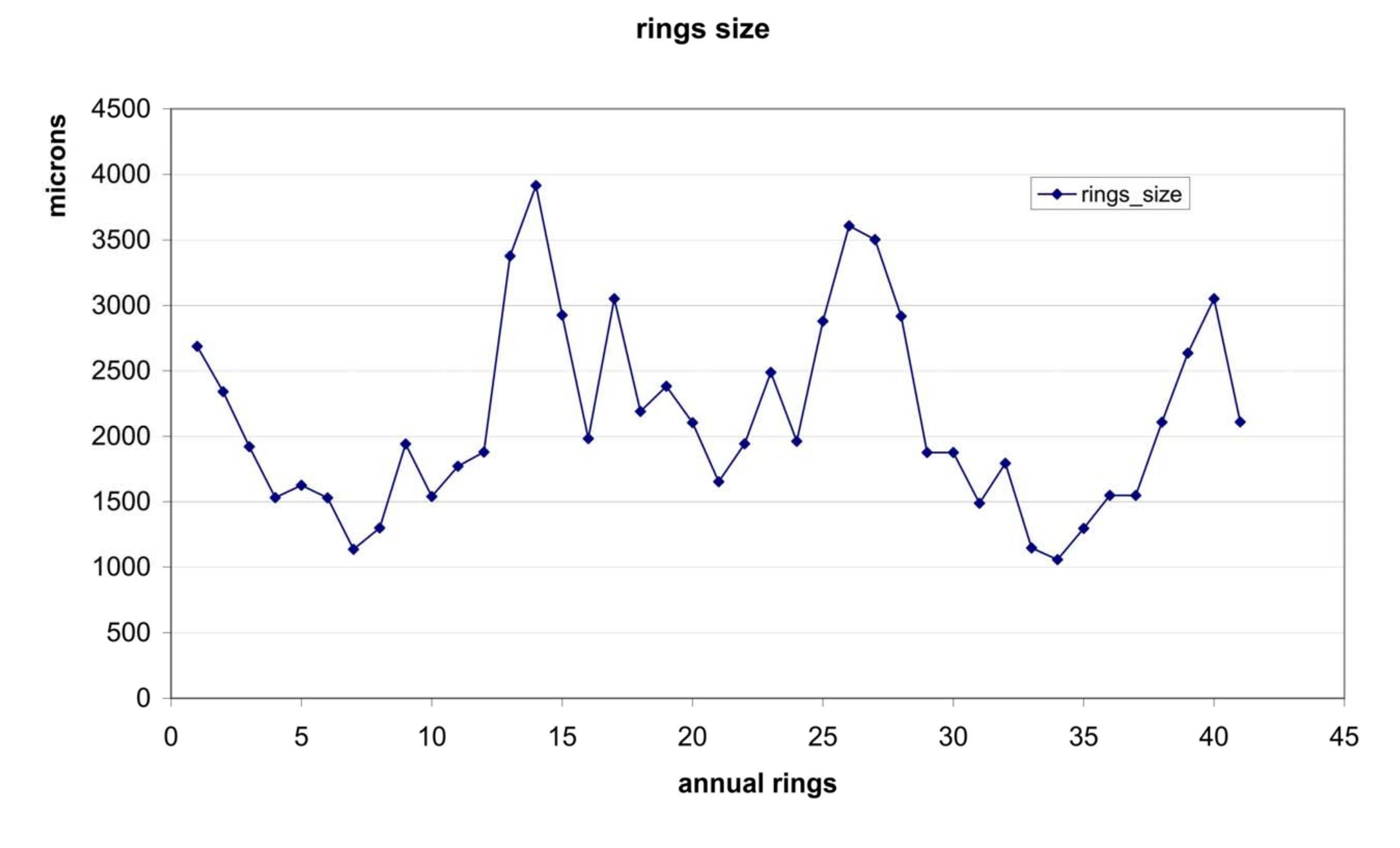
Dendrochronology is a scientific wood dating technique that studies the growth rings of trees in relation to time, which today represents the principal method of dating and studying wooden artefacts.

M. Bernabei et al., J. Archeo. Sci., 2010, **37**, 192

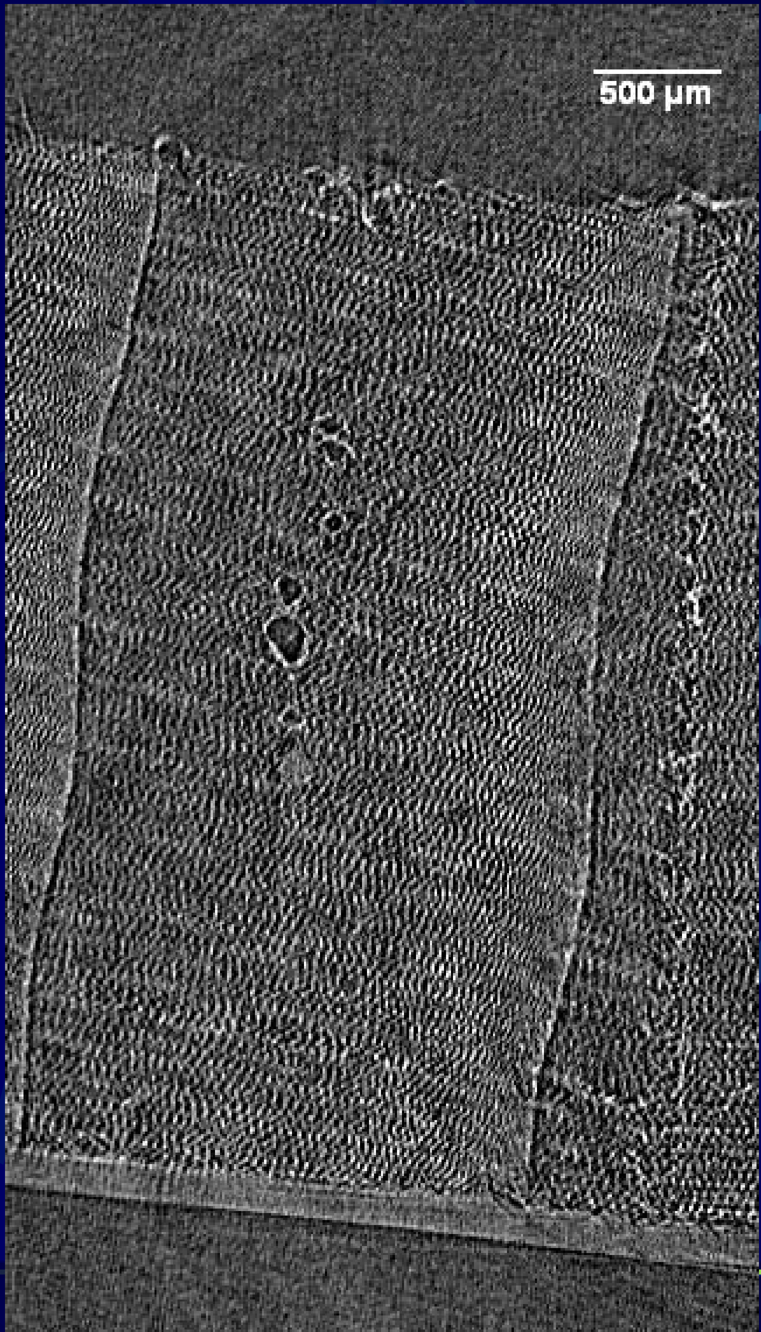
H.D.. Grissino-Mayer et al., J. Archeo. Sci., 2004, **31**, 167



- The object must contain an adequate number of clearly visible and measurable rings.
- The object must be made of a tree species suitable for dendrochronological dating.
- There must be a reference chronology for that particular species and geographical area.



Tree rings measurement taken from the front plate μCT scan. The ring widths are indicated by the dark points on the curve.



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

Concluding remarks

Synchrotron radiation phase-contrast micromography has proven to be an effective tool for the non-destructive visualisation of glass and consolidants.

The same technique has been successfully applied to other fields of cultural heritage: archaeological wood, historical musical instruments, paleonthology, etc.

The Constglass project has paved a way for a new form of collaboration between the communities of human and natural sciences, stimulating the formation of new professional figures.