# Saving the Future of our Past: Cultural Heritage Science at Elettra

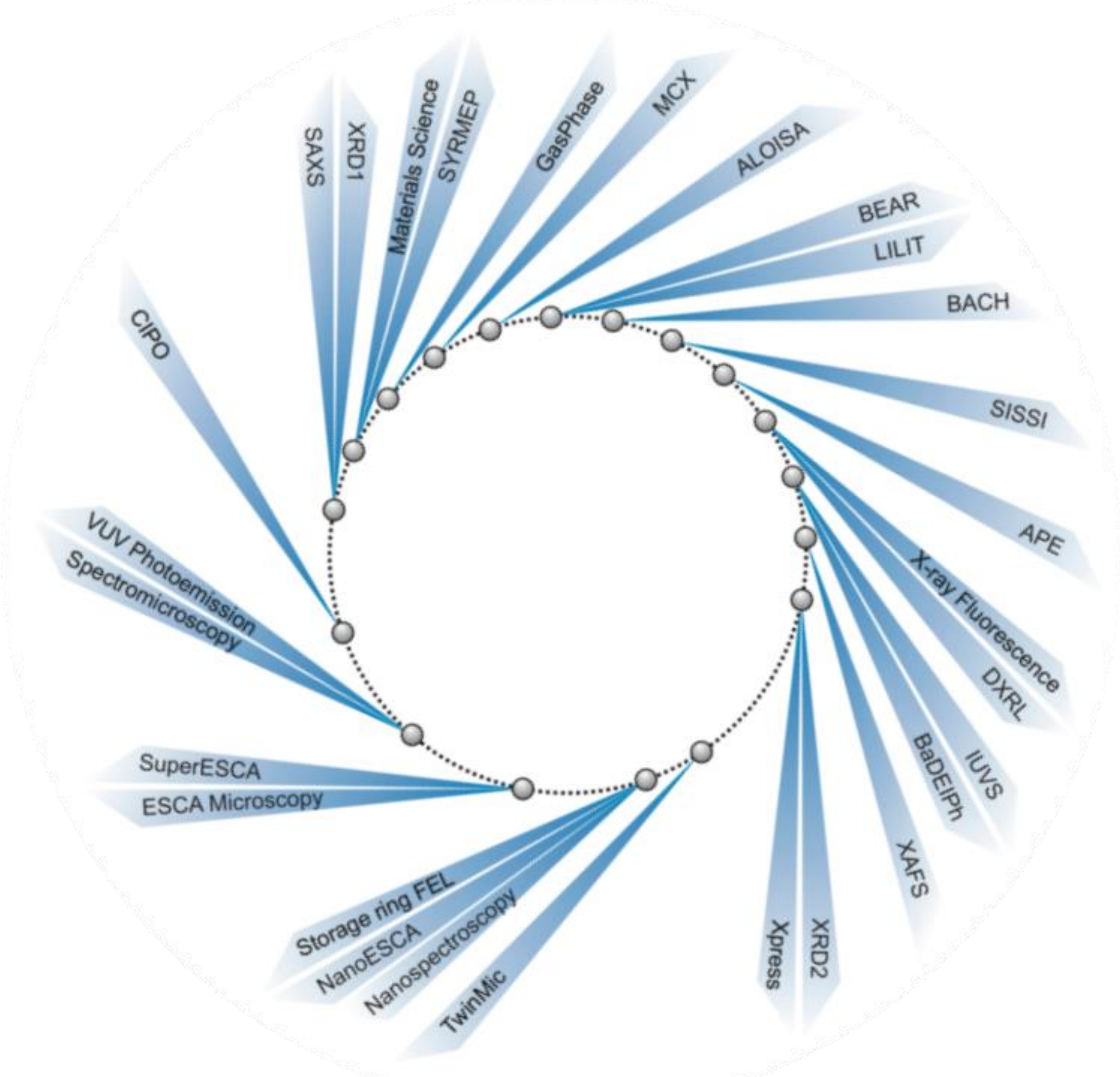
**Franco Zanini**

Elettra Sincrotrone Trieste

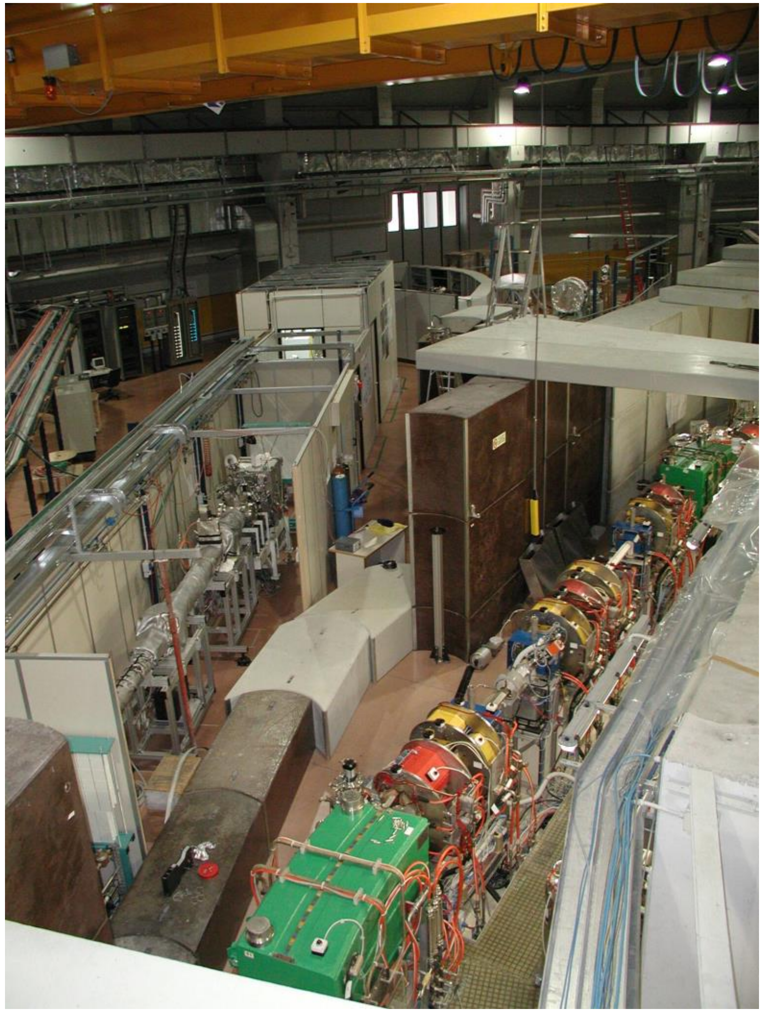
Postgraduate School of Archaeology
Universities of Trieste, Udine e Venezia Ca' Foscari

# The ELETTRA laboratory

# The beamlines at Elettra



# Why synchrotron light for CH?



1. **Brilliant** – several orders of magnitude more brilliant than conventional sources
2. **Continuous spectrum** – tunable from UV to X-rays
3. **Collimated** – can be focused to submicron dimensions
4. **Coherent** – allows the use of phase contrast techniques

# Cetervm censeo, mvndvm non delendvm esse

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

Artis monvmentorvm qvi vnvm vidit nvllvm vidit, qui mille vidit vnvm vidit

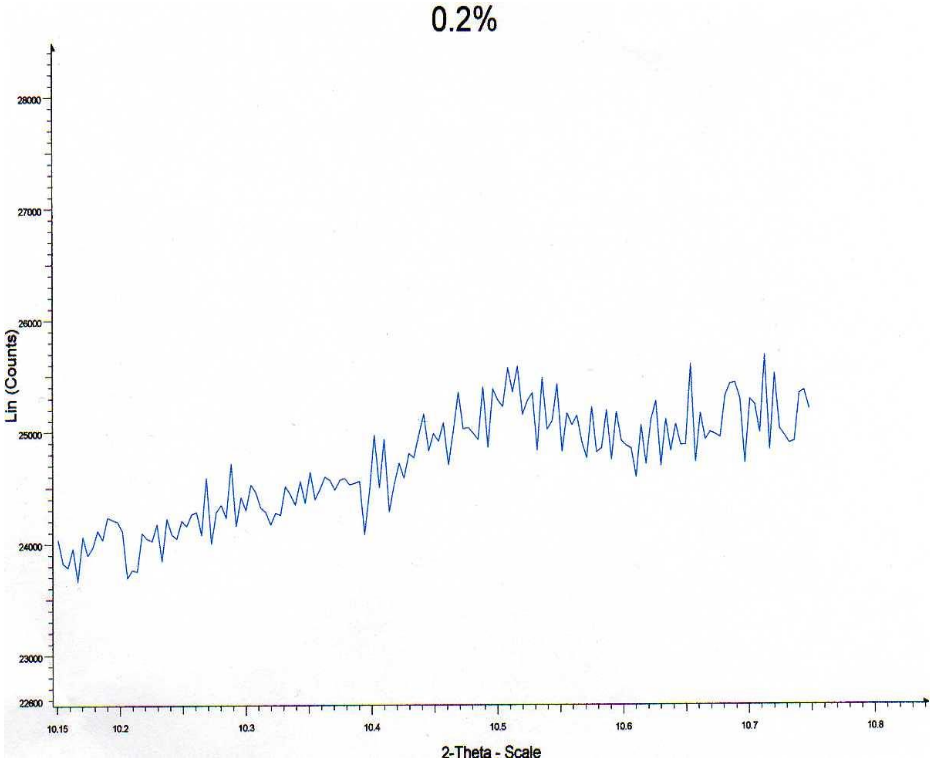
- Use of different analytical techniques
- Analysis of a large sets of similar samples

# What do we offer?

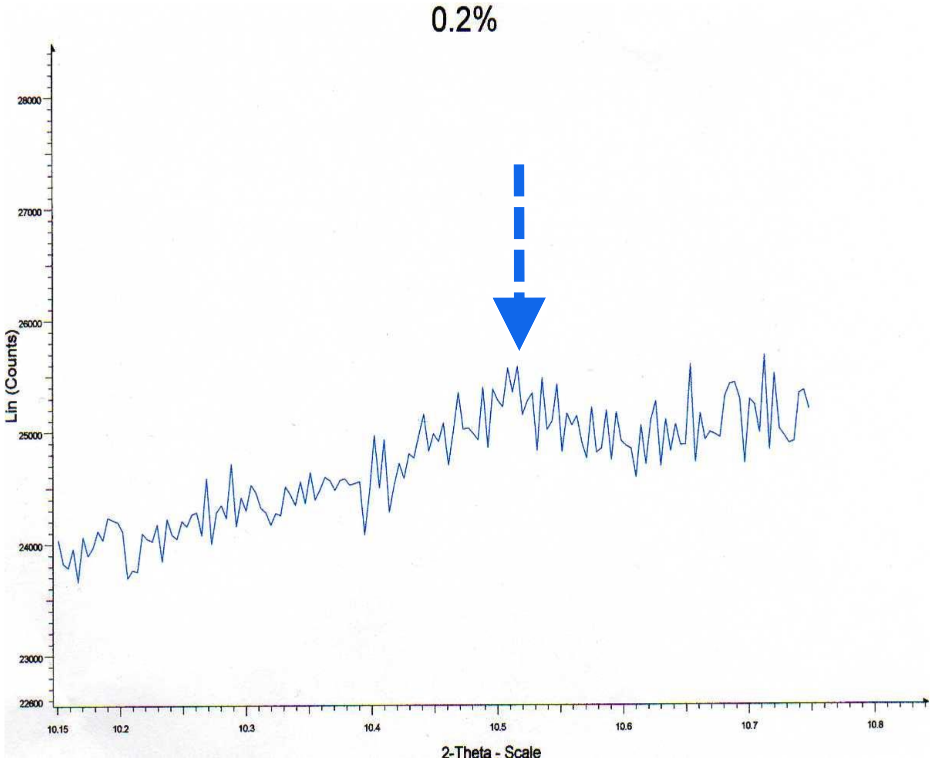
- A wide portfolio of techniques
- Most techniques are non-destructive or micro-destructive
- Synergies between conventional labs and large research infrastructures
- Access to thematic networks and search for funds

Drug analysis

Active principles are often characterized by a crystalline structure

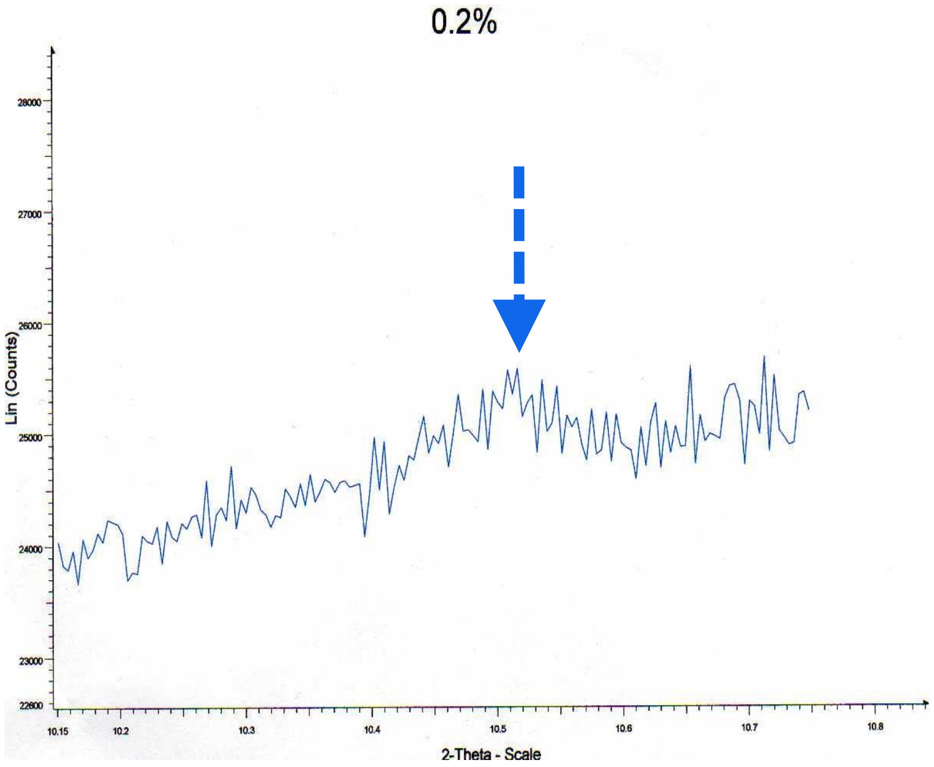


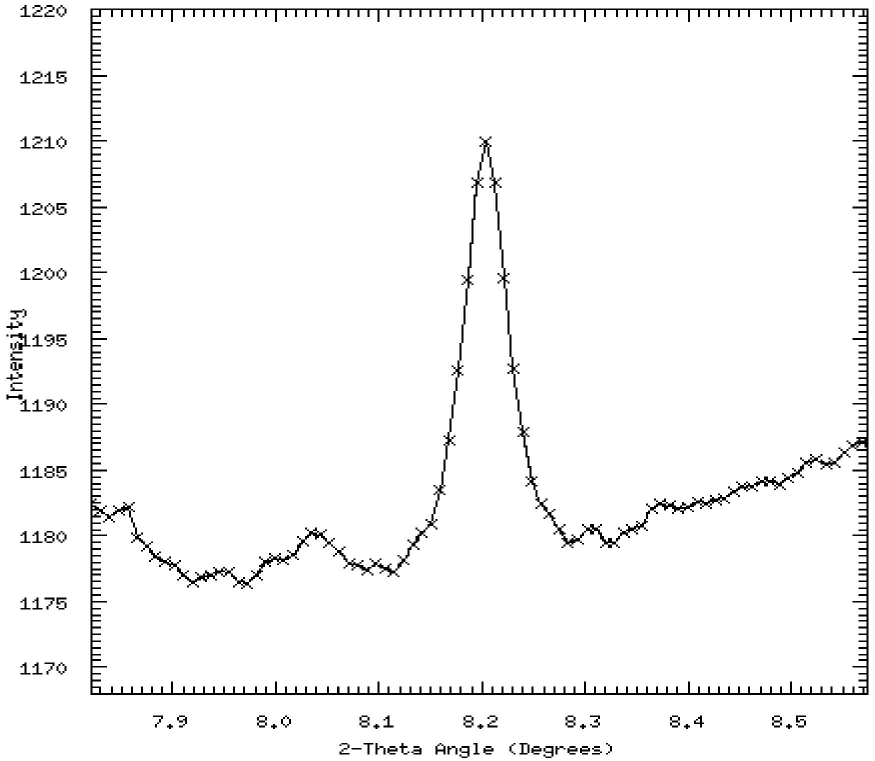
Drug analysis
Active principles are often characterized by a crystalline structure




Marker peak taken with a conventional source

Drug analysis
Active principles are often characterized by a crystalline structure




Marker peak taken with a conventional source

Marker peak taken with synchrotron radiation

# <u>ELETTRA MCX:</u> Materials characterization through X-Ray diffraction

Light source:

Bending magnet

Critical energy : 3.2keV (2.0) , 5.5keV (2.4)

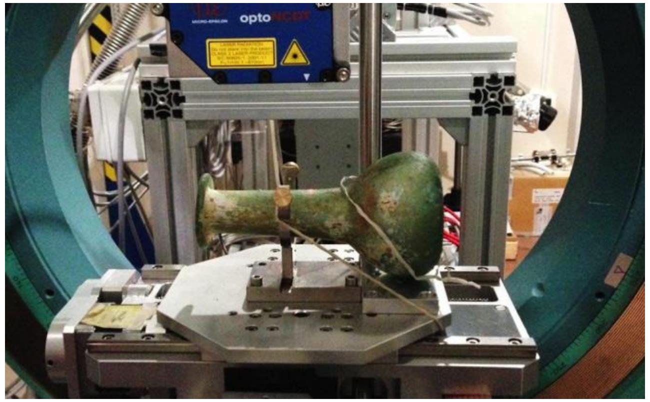
X-rays at sample:

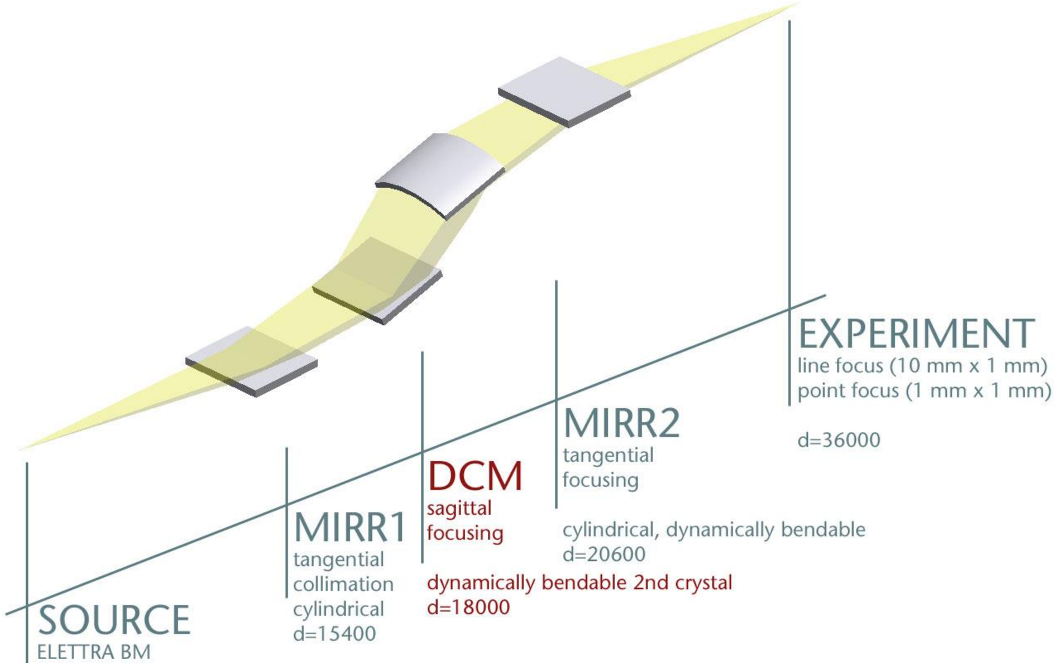
Energy range : 6-22 keV

Photon flux : $10^{11}$ photons/sec

Beam size at sample : 10x1 $mm^2$ - 0.3x

Energy resolution : ΔE/E $2x10^{-4}$





## MCX beamline

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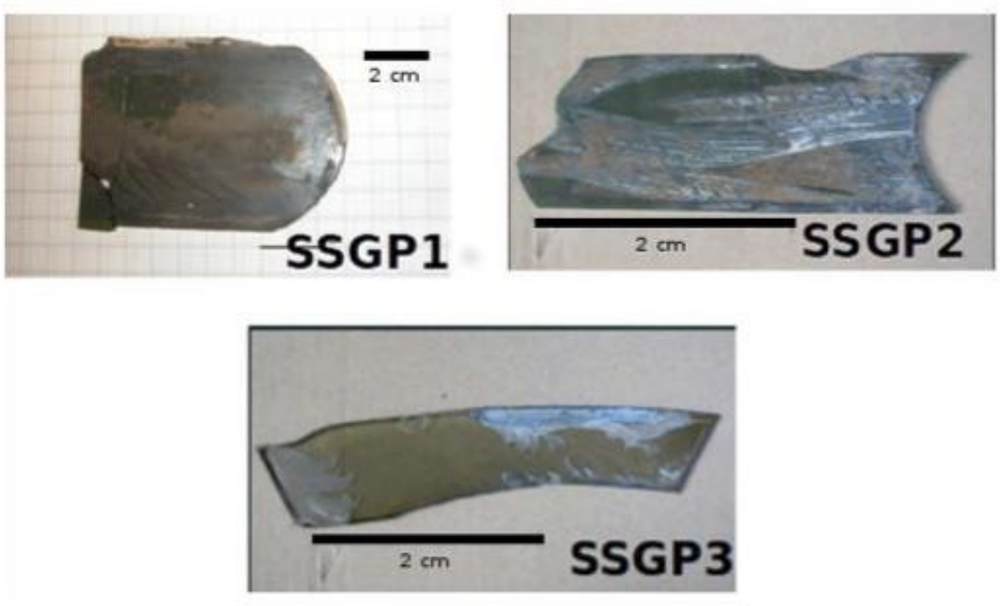
# The X-ray diffraction beamline MCX at Elettra: a case study of non-destructive analysis on stained glass

**Jasper R. Plaisier[1], Luca Nodari[2], Lara Gigli[1], Elena Paz Rebollo San Miguel[2], Renzo Bertoncello[3], Andrea Lausi[1]**

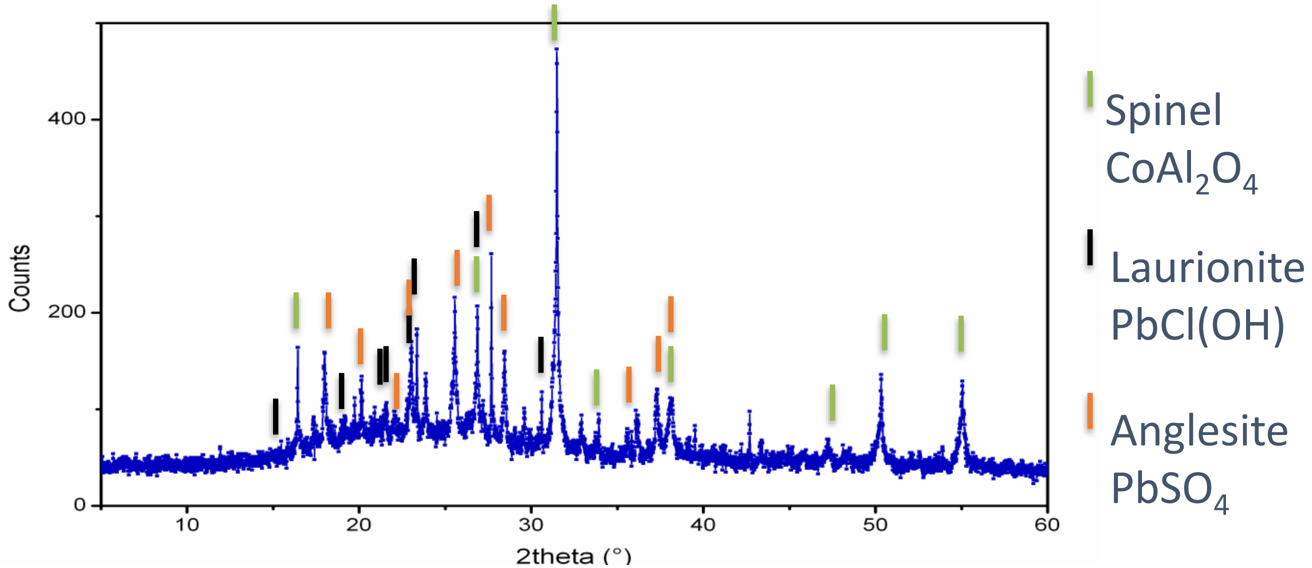
[1]*Elettra-Sincrotrone Trieste S.C.p.A., Strada Statale 14 - km 163,5 in AREA Science Park, 34149 Basovizza, Trieste, Italy*
[2]*Istituto di Chimica della Materia Condensata e di Tecnologie per l'Energia CNR– ICMATE, Area della Ricerca di Padova, Corso Stati Uniti 4, 35127 Padova, Italy*
[3]*Dipartimento di Scienze Chimiche, Università degli Studi di Padova, Via marzolo 1, 35131 Padova, Italy*

The long period of exposure to an increasingly aggressive environment in the immediate vicinity of seawater, makes the windows of the venetian Basilica di San Giovanni e Paolo an excellent case study for investigating the effect of pollution and climate change.




One of the strengths of MCX is the well-defined narrow instrumental profile, which allows accurate identification of phases in complex mixtures, which is often the case for objects or fragments that are being studied in the field of cultural heritage.
Three glass fragments from The Basilica di San Giovanni e Paolo in Venice were selected to study the alteration processes.

An aggressive environment with large thermal variation, can promote micro- cracking on the grisailles surface. These micro-breaks, together with the natural roughness of the grisailles layer, may favor the condensation processes on the surface. In this way, the formation of a deterioration-induced porous system can act as a series of micro-reactors for leaching phenomena with the subsequent salt precipitation.



Incoherent beam -> absorption contrast

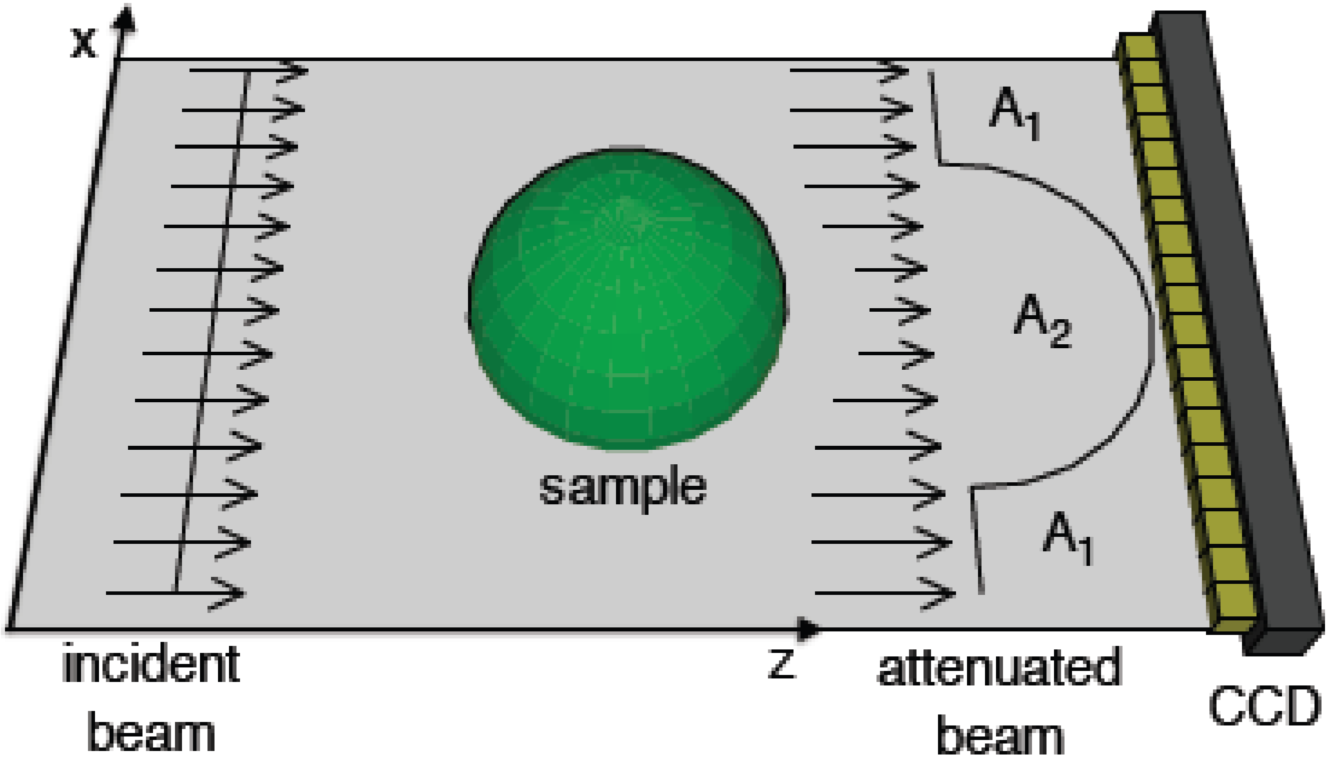
Coherent beam -> phase contrast

Phase contrast exploits differences in the refractive index of different materials, and allows to distinguish structures with similar absorption. The refractive index is expressed as a complex value:

$$n = 1 - \delta - i\beta$$

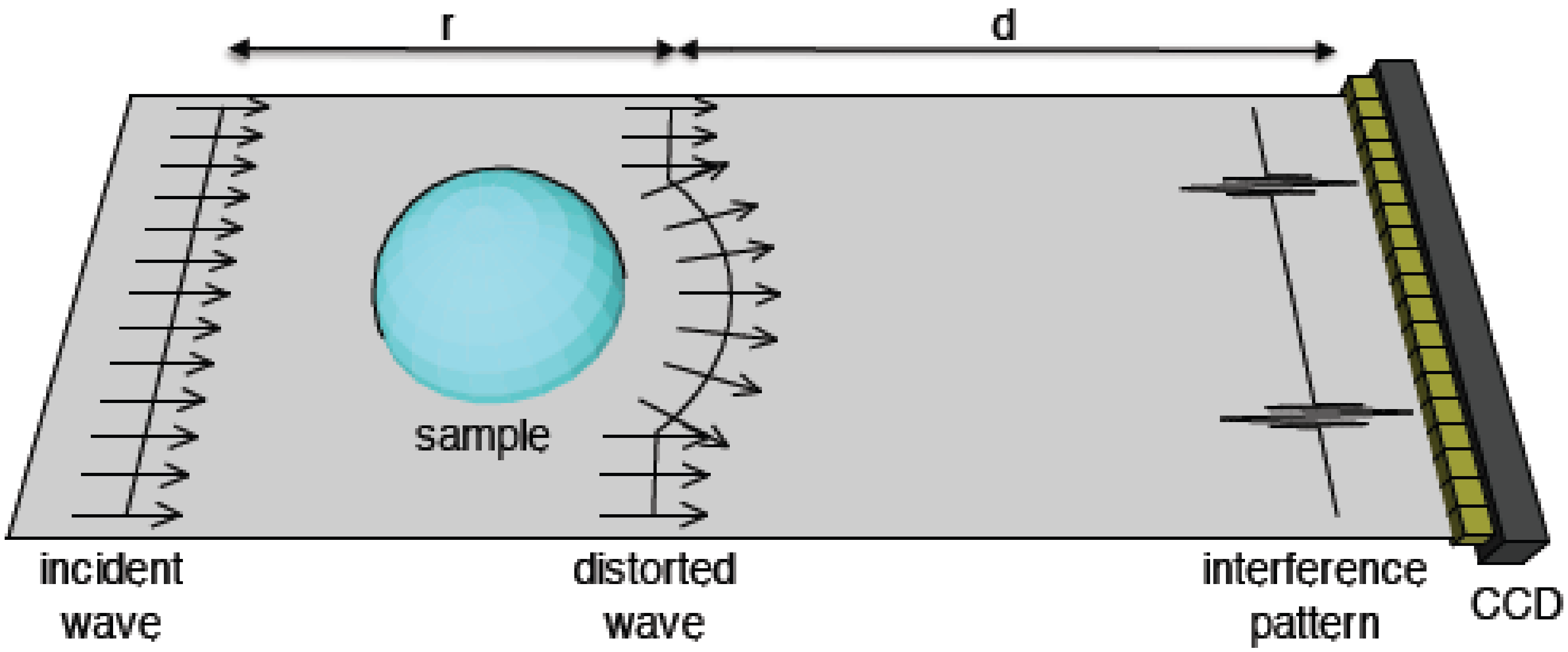
The $\delta$ term is linked to the phase shift, three orders of magnitude greater than the absorption term $\beta$. It therefore becomes possible to reveal phase effects even if the absorption (or the difference in absorption between different phases of a material) is negligible.

# Absorption Imaging



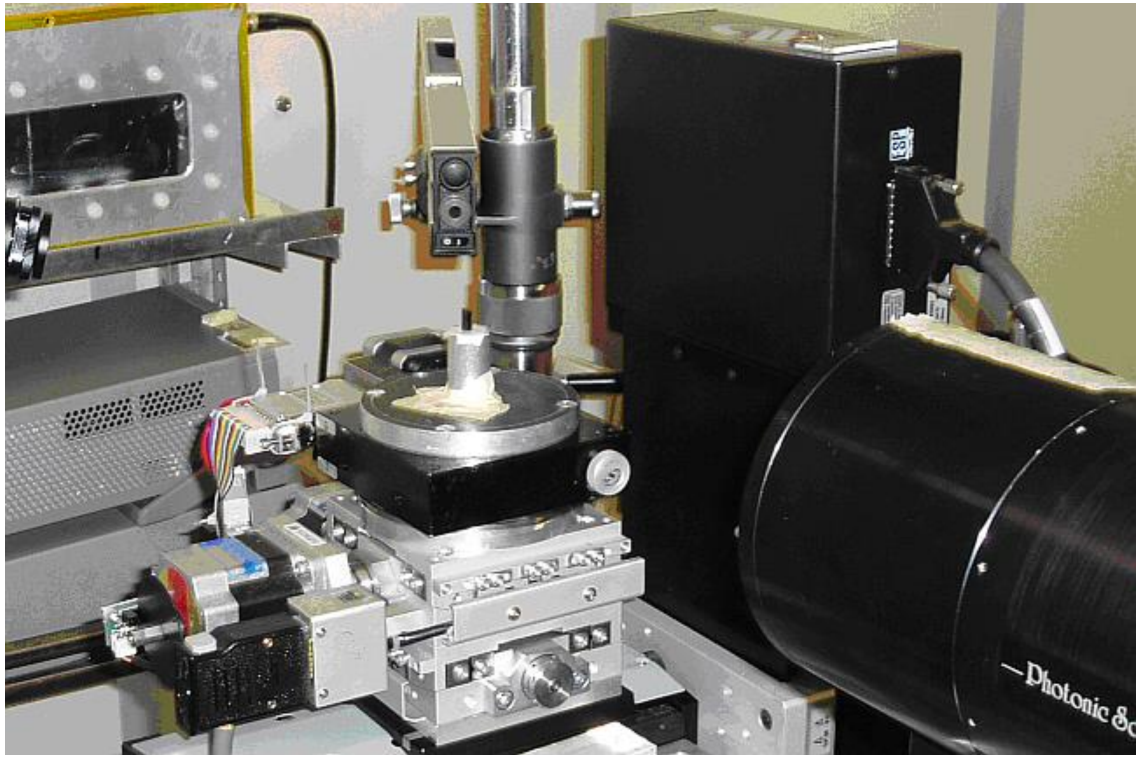

The incident beam is attenuated by the absorbing sample. The attenuation of the sample is recorded in area A2. The background signal is recorded in the A1 areas.

# Phase-contrast Imaging: propagation method

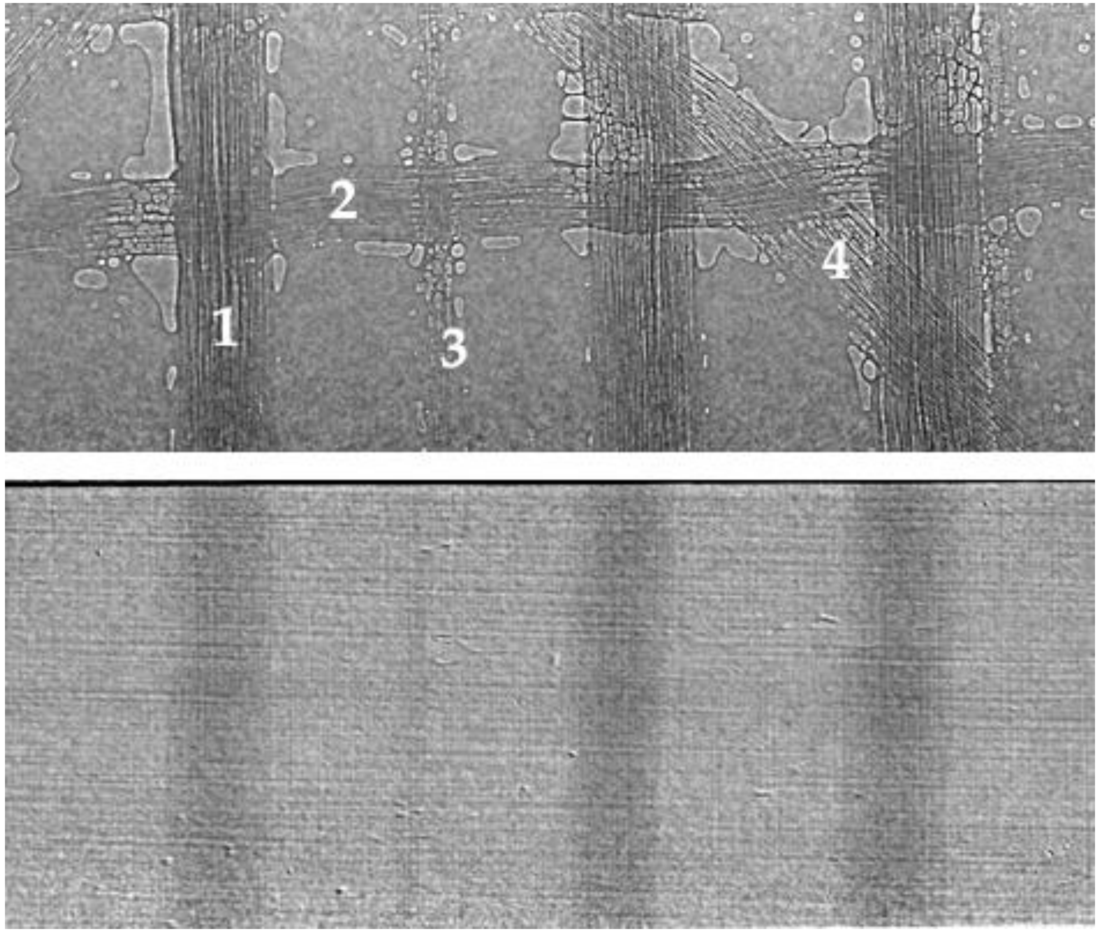



It is the simplest method, it does not require specific optics. Just chooses the correct sample - detector distance to get the signal. Without having to modify the reconstruction, the result is an “edge enhanced” image.

**<u>SYRMEP:</u> SYnchrotron Radiation for MEdical Physics**



X-ray Image taken with synchrotron radiation



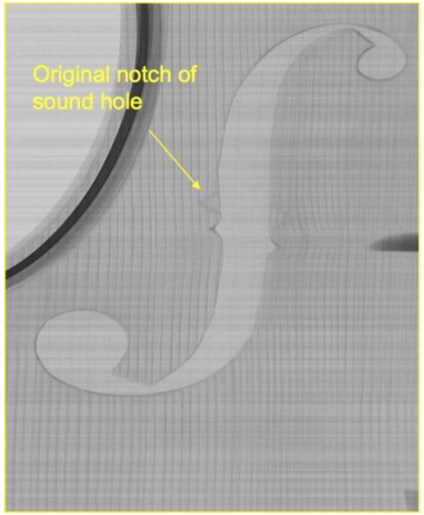

X-ray Image taken with a conventional source

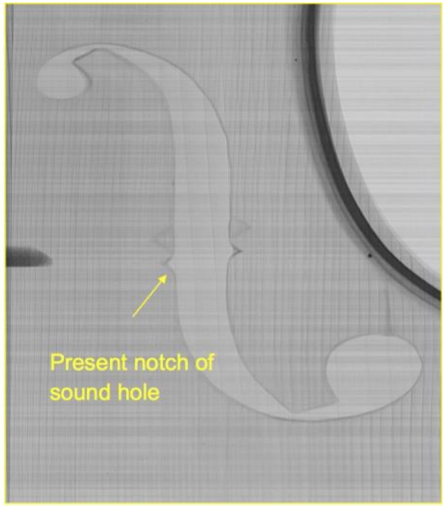
America's Cup
high performance sails

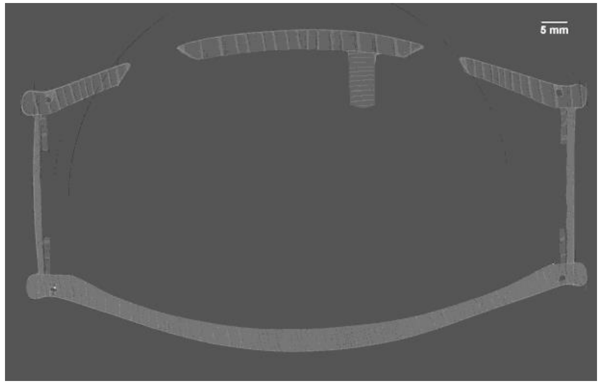
1. High modulus Twaron® fill
2. High modulus Twaron® warp
3. Spectra® ripstop fill
4. Spectra® diagonal axis

FOV = 5.6 mm x 13.5 mm
Resolution = 20 μm
X-ray energy = 15 keV

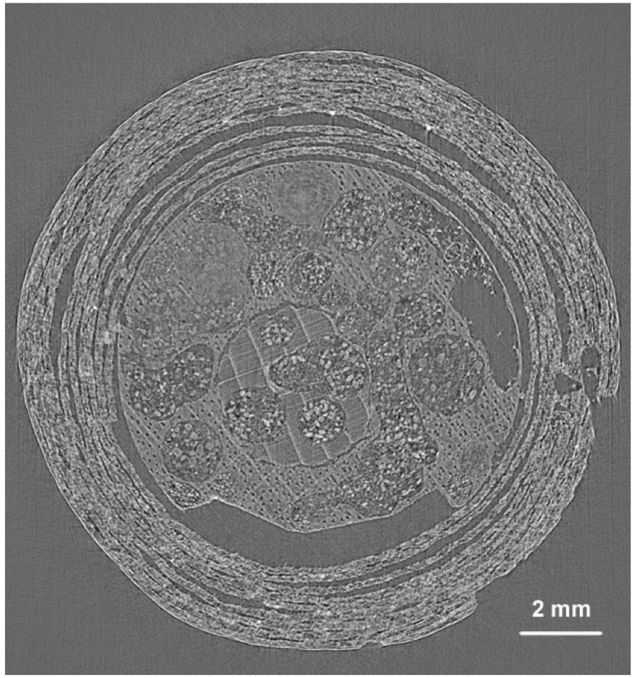
F. Zanini, Strad 123 (2012) 36




**G. B. Guadagnini
Herrestal violin,
Milano 1753**




**Paper-pipes organ, Lorenzo Gusnasco da Pavia (1494)**

B. Bentivoglio-Ravasio et al., J. Ent. Acarol. Res. 43 (2011) 149

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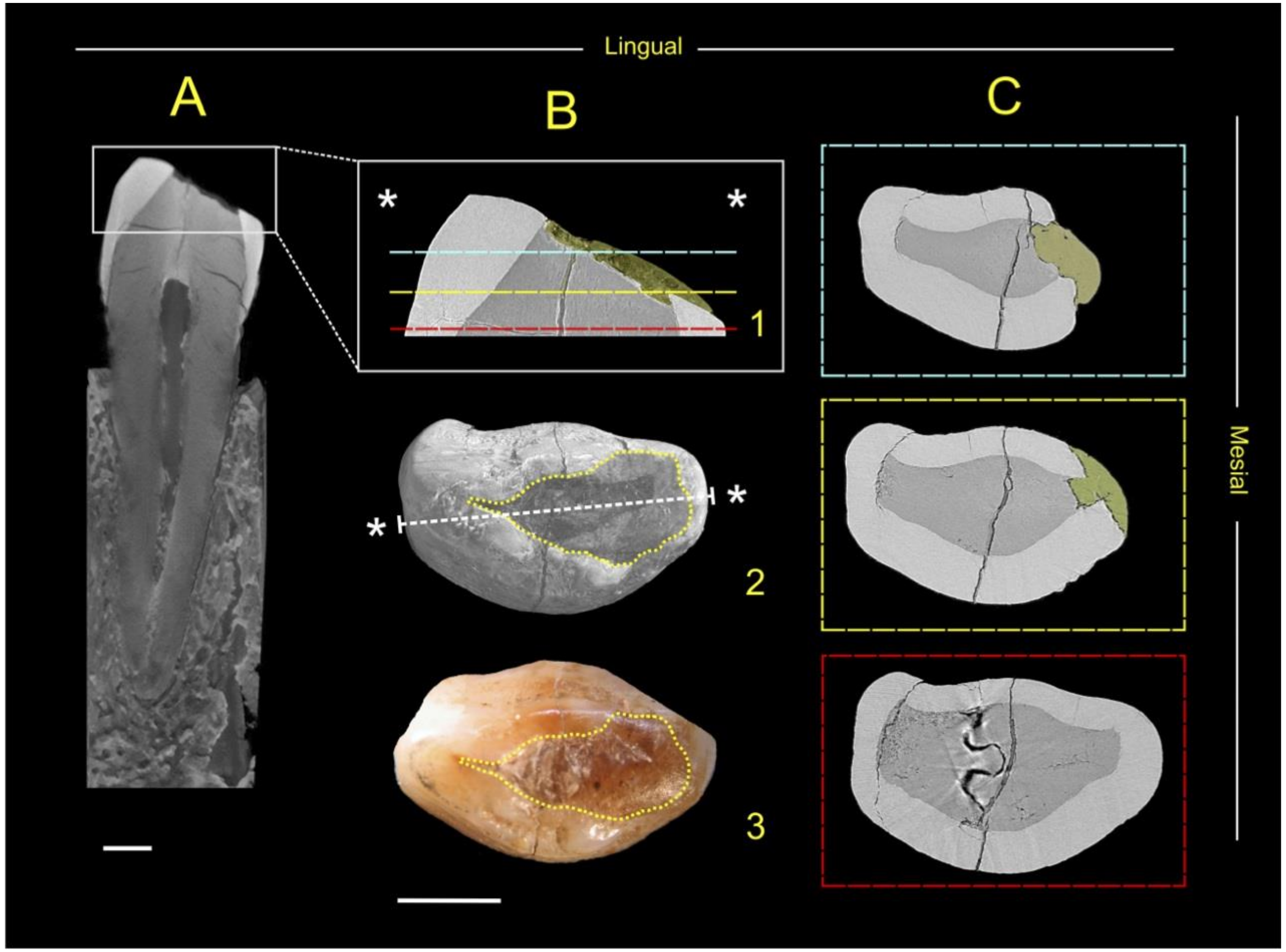
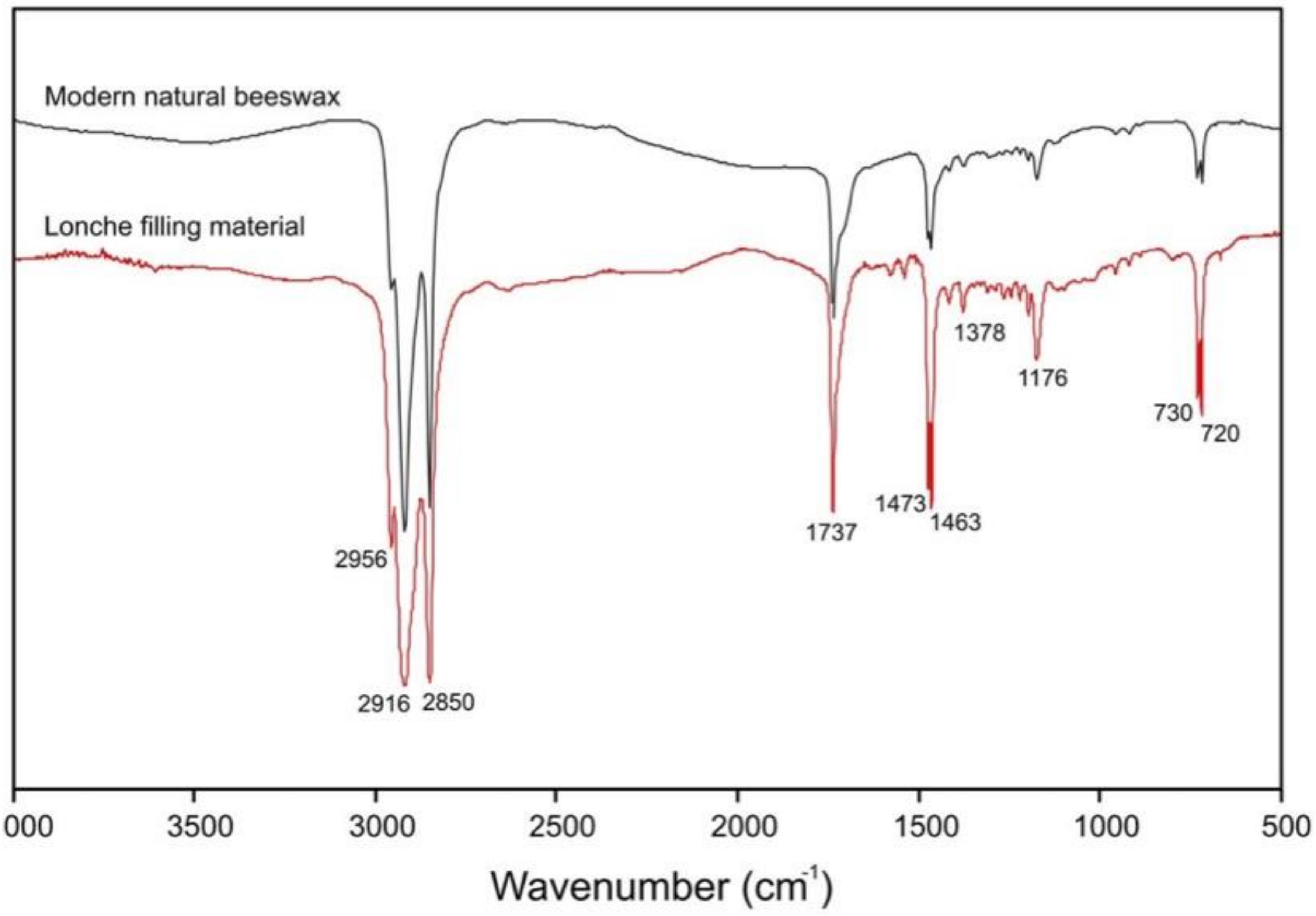
# Beeswax as Dental Filling on a Neolithic Human Tooth

**Federico Bernardini[1]*, Claudio Tuniz[1,2], Alfredo Coppa[3], Lucia Mancini[4], Diego Dreossi[4], Diane Eichert[4], Gianluca Turco[5], Matteo Biasotto[5], Filippo Terrasi[6], Nicola De Cesare[7], Quan Hua[8], Vladimir Levchenko[8]**

**1** Multidisciplinary Laboratory, "Abdus Salam" International Centre for Theoretical Physics, Trieste, Italy, **2** Centre for Archaeological Science, University of Wollongong, Wollongong, New South Wales, Australia, **3** Department of Environmental Biology, University "La Sapienza", Rome, Italy, **4** Sincrotrone Trieste S.C.p.A., AREA Science Park, Basovizza (Trieste), Italy, **5** Department of Medical Sciences, University of Trieste, Trieste, Italy, **6** CIRCE, INNOVA and Department of Environmental Sciences, 2nd University of Naples, Caserta, Italy, **7** CIRCE, INNOVA and Department of Life Sciences, 2nd University of Naples, Caserta, Italy, **8** Australian Nuclear Science and Technology Organisation, Lucas Heights, New South Wales, Australia



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



Modern natural beeswax
Lonche filling material
2956
2916
2850
1737
1473
1463
1378
1176
730
720
000
3500
3000
2500
2000
1500
1000
500
Wavenumber (cm-1)

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*

F. Bernardini et al., PLoS ONE 7 (2012)

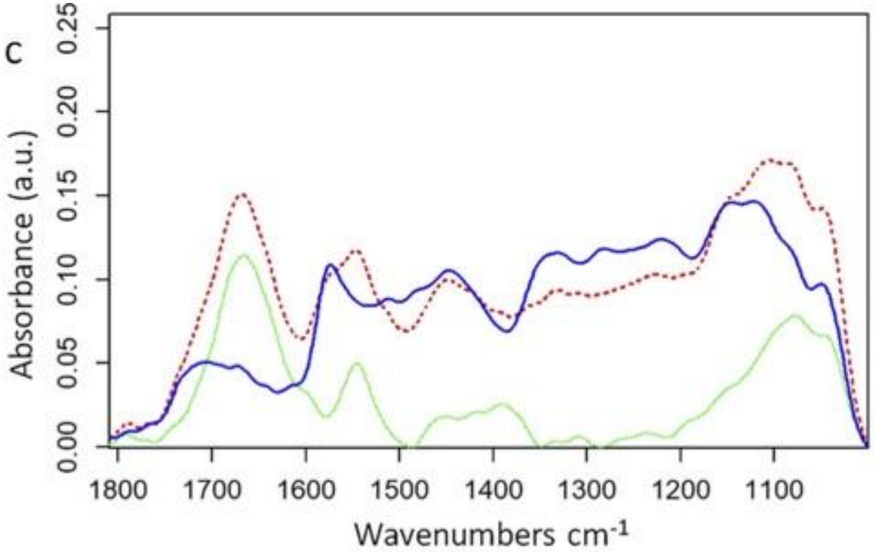
SCIENTIFIC REPORTS
natureresearch

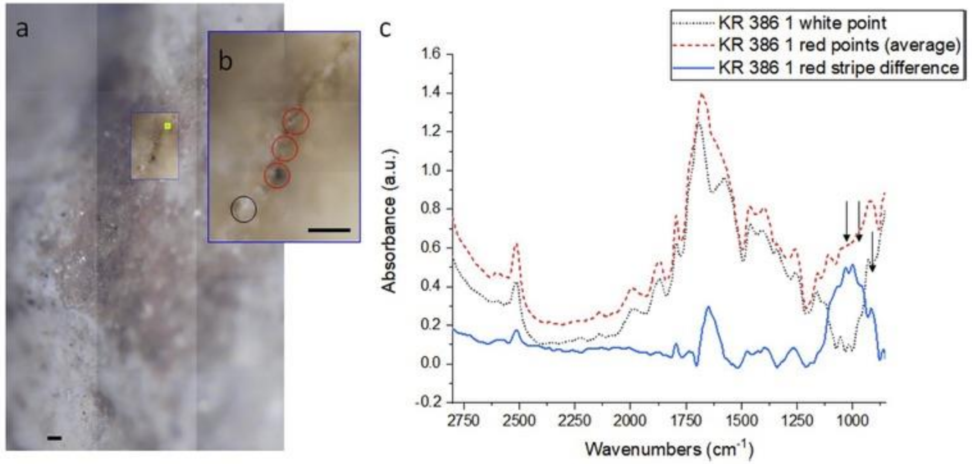
OPEN

# Surface analysis of an eagle talon from Krapina

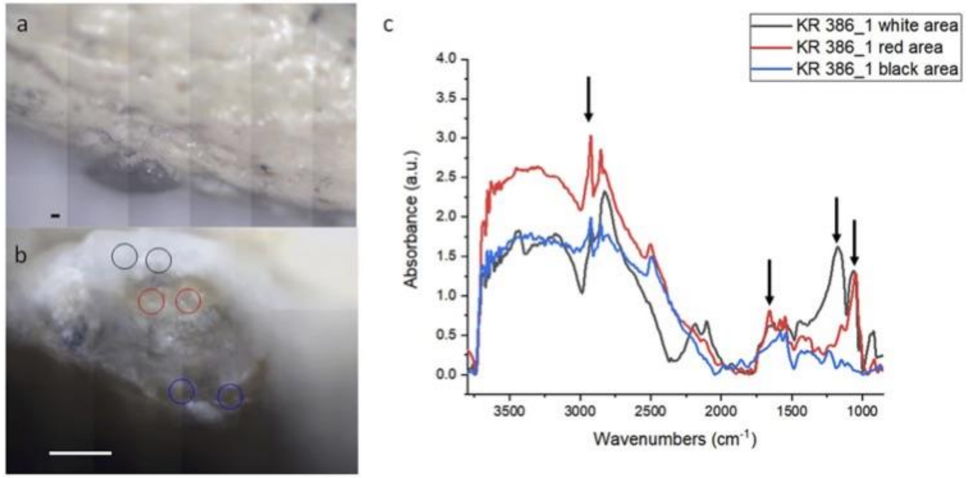
Davorka Radovčić[1], Giovanni Birarda[2], Ankica Oros Sršen[3], Lisa Vaccari[2], Jakov Radovčić[1] & David W. Frayer[4*]


The talons of Krapina's white-tailed eagle represent a kind of jewelry worn by some members of a Neanderthal community about 130,000 years ago. Confirming this, a re-inspection revealed a fiber, sealed by a thin silicate coating, adhering to the surface within a large cut mark, as well as concentrated traces of red and yellow pigment spots and some black spots. We non-destructively analyzed the fiber and small portions of pigmented areas using infrared synchrotron light.






Several areas were analyzed, revealing the protein nature of the fiber, identified as being of animal origin. Targeted areas revealed intra- and inter-row aggregation suggesting the presence of collagen whose original triple α-helix conformation is in a state of degradation. This further confirms the diagenetic decay of the original collagen structure and the antiquity of the fiber.




It is possible that the fiber is a remnant of a leather cord or sinew used to bind the claws together. Spectroscopic analysis of the pigments in two isolated areas confirmed the presence of two types of ocher and that the dark spots are charcoal remnants. The application of new non-invasive technologies offers new possibilities to further test the hypothesis of using prehistoric objects for symbolic purposes.

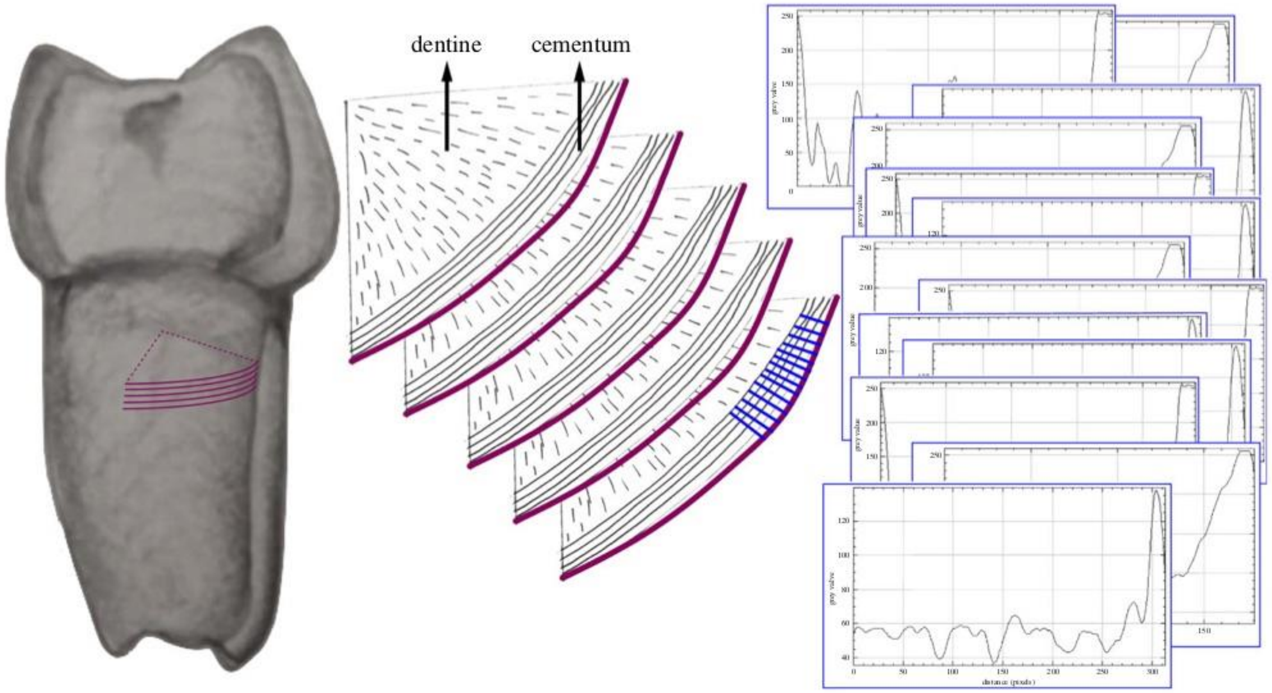
INTERFACE

royalsocietypublishing.org/journal/rsif

Research

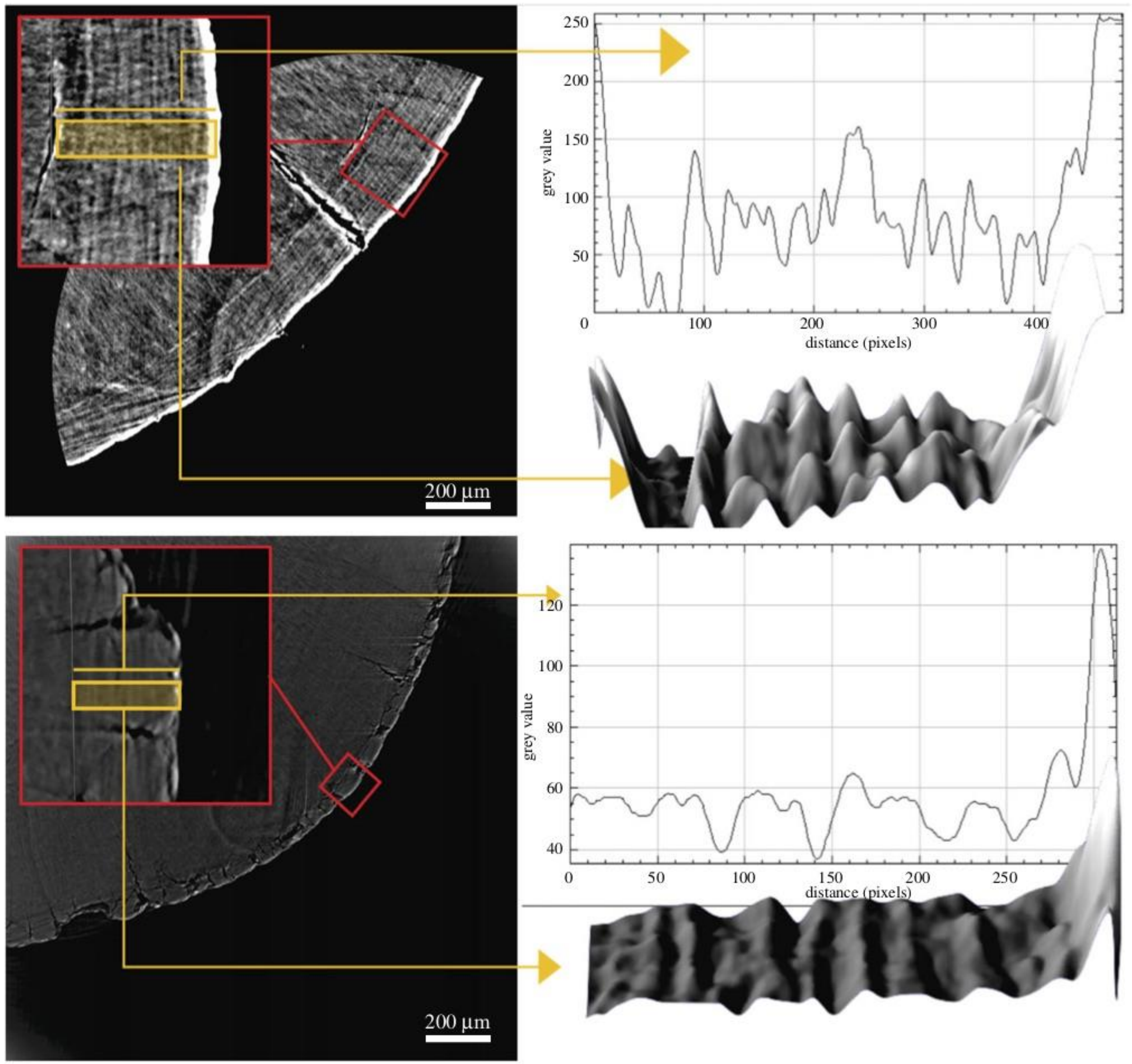
# Dental cementum virtual histology of Neanderthal teeth from Krapina (Croatia, 130–120 kyr): an informed estimate of age, sex and adult stressors

Paola Cerrito[1,2,3], Alessia Nava[4], Davorka Radovčić[5], Dušan Borić[6], Leonardo Cerrito[7], Tricia Basdeo[8], Guido Ruggiero[9,10], David W. Frayer[11], Alexander P. Kao[12], Luca Bondioli[13], Lucia Mancini[12] and Timothy G. Bromage[1,2,3]




Life-history variables (LHVs) are those variables that directly record the timing of the several major life-history events, such as gestation length, age at weaning, age at sexual maturity, age at first birth, interbirth intervals, age at menopause and longevity. Nonetheless, tracking the evolution of LHVs in the fossil record has proven to be a significant challenge as it is limited to what can be gleaned and inferred from teeth and bones.

The current knowledge regarding LHVs in the hominin fossil record is very scant, being limited to estimates of age at death, gestation length, onset of weaning and physiological stress occurrence. The present work aims to improve and enrich our understanding of some of the more elusive LHVs using a non-destructive cutting-edge methodology.




Human teeth permanently record physiologically impactful events during their formation, enabling the collection of data about stressors experienced during development in addition to the estimation of the age at death. All recording structures are periodically layered, and each layer is the visible result of changing micromorphology, which corresponds to changing physiological states of the organism.

The authors were able to correctly detect and time all reproductive (menarche, parturition, menopause) and other physiologically impactful events in the modern sample. For the fossil specimens, age at death and age at occurrence of biologically significant events were estimated.

scientific reports

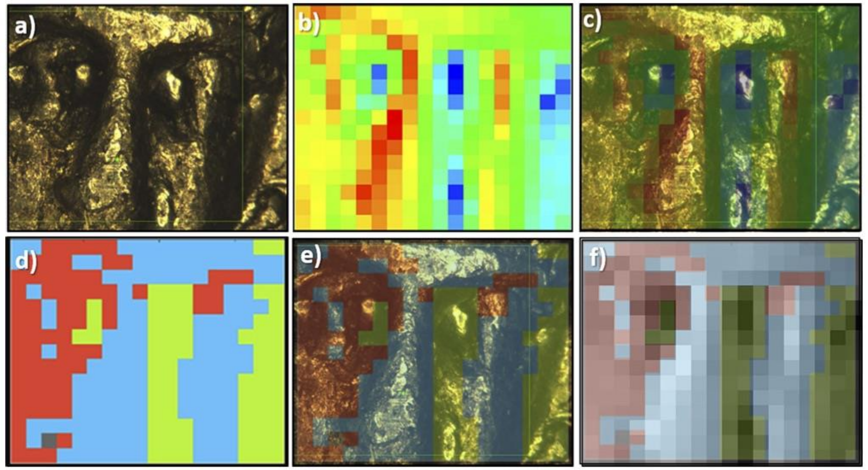
OPEN

# Combining synchrotron radiation techniques for the analysis of gold coins from the Roman Empire

I. Carlomagno[1✉], P. Zeller[1,4,5], M. Amati[1], G. Aquilanti[1], E. Prenesti[2], G. Marussi[3], M. Crosera[3] & G. Adami[3]

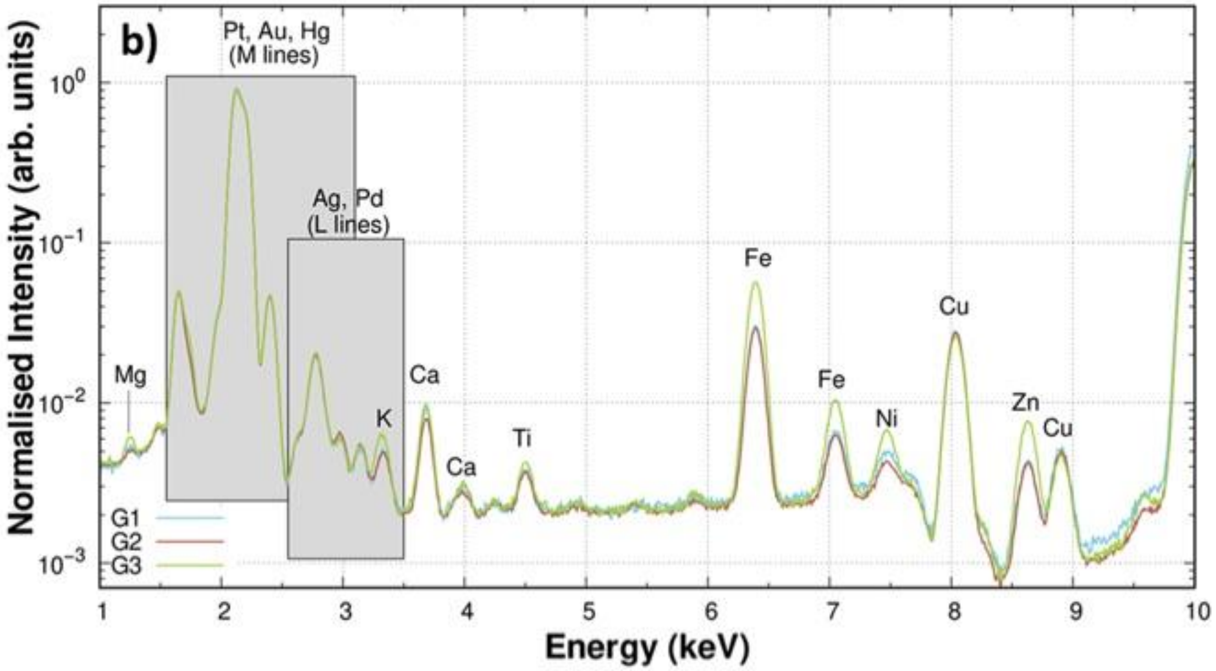
The aim of this work is to describe a methodological strategy based on advanced analytical synchrotron techniques coupled with a statistical data analysis approach to find minor differences and give useful information to the historians on several aspects of ancient civilizations.

Applying the t-SNE algorithm to spatially resolved XRF data sets, we could identify the elements used in the minting of each piece, disentangling the fluorescence emission of the dirt accumulated on the surface from the signal of the elements making up the original coin.




A semi-quantitative fitting of the XRF spectra relative to the metallic alloy evaluated the Au fineness and the presence of minority alloy elements. Our data show a variation in the alloy purity of about 2% among the pieces analysed, which is in agreement with the available literature.

The lower fineness of some of the coins can be explained with the use of Au alloys probably coming from tax collection as heterogeneous source material of minting. The fine evaluation of the trace elements highlighted a different Pt content in one of the coin with respect to the others. This suggests that a different gold source was used to struck this coin with respect to the other samples.



Analyst

ROYAL SOCIETY OF CHEMISTRY

PAPER

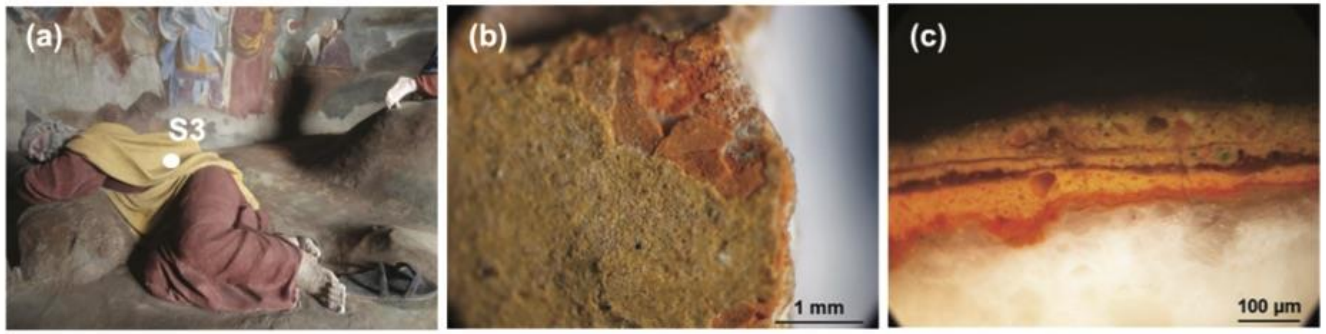




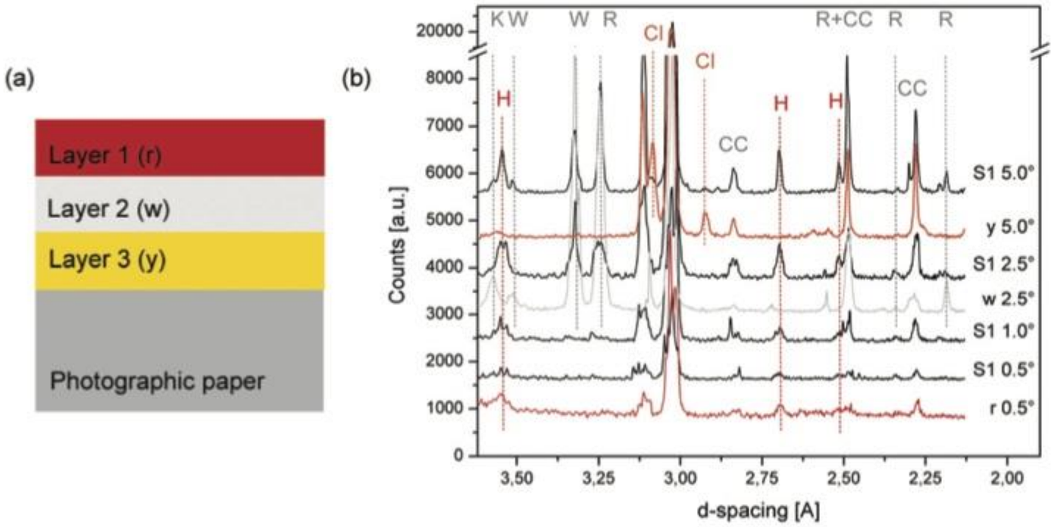
Cite this: *Analyst*, 2018, **143**, 4290

## What's underneath? A non-destructive depth profile of painted stratigraphies by synchrotron grazing incidence X-ray diffraction

Elena Possenti, *[a,b] Chiara Colombo, [b] Claudia Conti, [b] Lara Gigli, [c] Marco Merlini, [a] Jasper Rikkert Plaisier, [c] Marco Realini [b] and G. Diego Gatta [a]

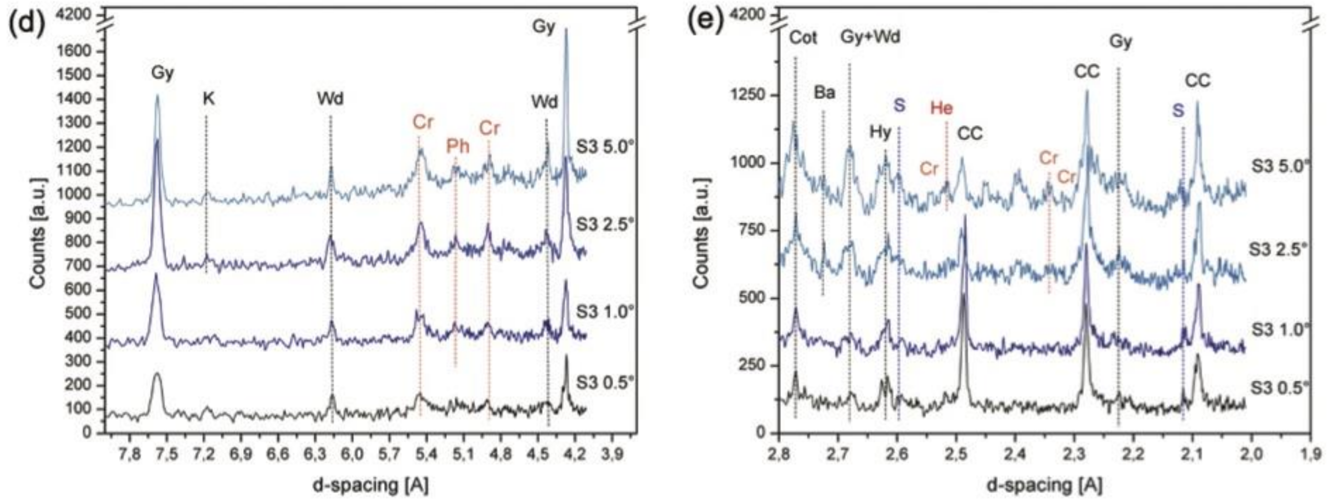



Many works of art are complex systems consisting of a core completed by the overlapping of several painted layers. In this work, we apply an innovative method based on grazing incidence X-ray diffraction (GIXRD) with synchrotron radiation (SR) to investigate polychrome stratigraphies with a completely non-destructive approach. The SR-GIXRD measurements provided direct and unambiguous compositional and stratigraphic information of the crystalline species lying in different layers.




(a) Scheme of the layer succession and (b) GIXRD patterns of the mock-up sample S1 collected at different incidence angles (0.5°, 1.0°, 2.5°, 5.0°) in comparison with the reference patterns of red (r), yellow (y) and white (w) varnishes, collected at Φ of 0.5°, 2.5° and 5.0° respectively. It is possible to observe the XRD peaks ascribable to hematite (H), rutile (R), wollastonite (W), clinobisvanite (Cl), calcite (CC), and kaolinite (K) of the photographic support.

GIXRD patterns of sample S3 collected at different Φ in the ranges 8–3.9 Å (d) and 2.8–1.9 Å (e). XRD peaks of gypsum (Gy), kaolinite (K), weddellite (Wd), crocoite (Cr), phoenicochroite (Ph), sodalite (S), barite (Ba), cotunnite (Cot), hematite (He), calcite (CC) and hydrocerussite (Hy).



coatings

MDPI

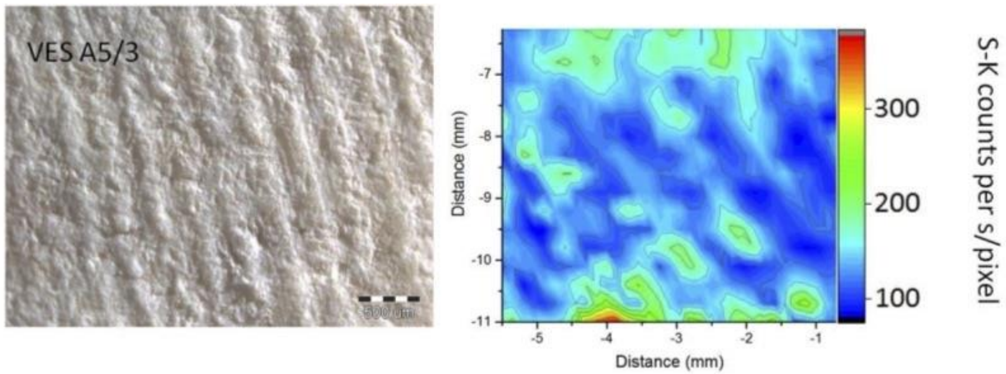
Article

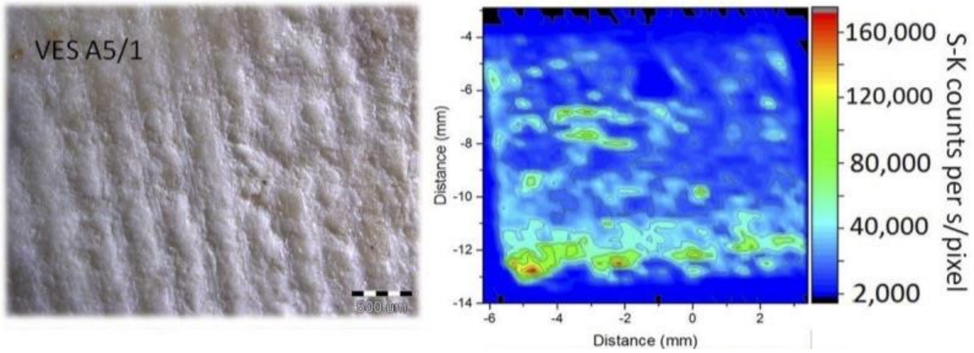
# Protection of Stone Monuments Using a Brushing Treatment with Ammonium Oxalate

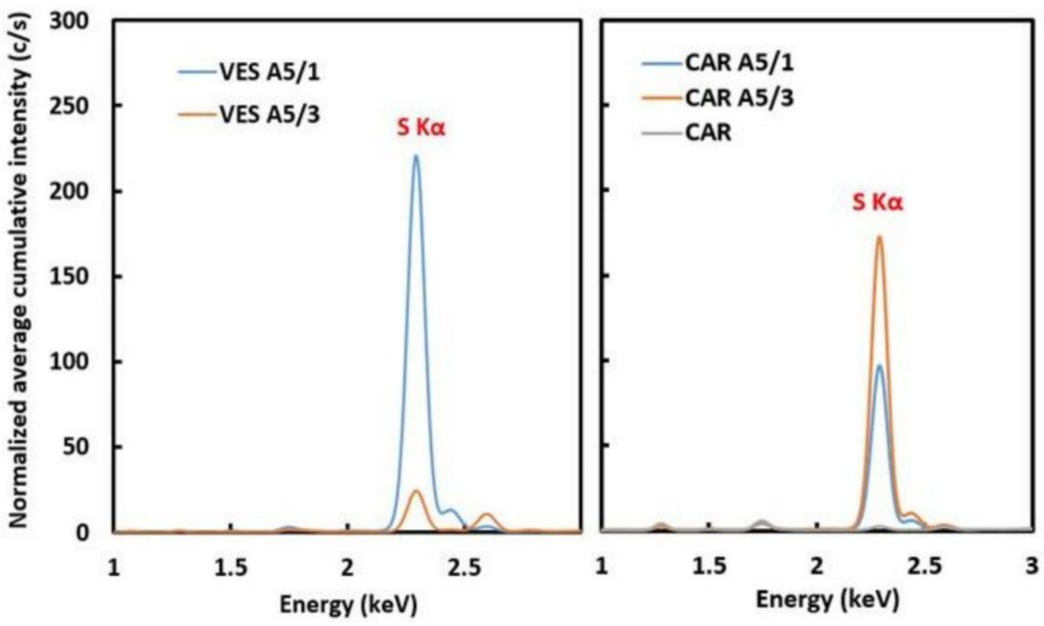
Domagoj Mudronja [1,*], Frederik Vanmeert [2], Stjepko Fazinic [3], Koen Janssens [2], Darko Tibljas [4] and Vladan Desnica [5]

Stone buildings are subject to atmospheric agents. Carbonate-based stones are particularly vulnerable to acidic environments, while magmatic acid stones are more susceptible to chemical atmospheric agents in basic environments.

To slow down the surface corrosion of limestone and marble works, protective coatings have been proposed in order to inhibit the dissolution of calcite. In this work, samples of two types of stone with different porosity were treated with ammonium oxalate ($AmO_x$) to create a protective layer of calcium oxalate ($CaO_x$).






Normalized cumulative XRF spectra were obtained for brushing treatments of both Veselje limestone and Carrara marble for one and three hours. In the case of Veselje limestone, the 3 hour brushing treatment showed a much lower cumulative sulfur signal rate than the 1 hour treatment.

This is in line with the conclusion drawn from the comparison of the 2D maps and confirms that on average the protective layer after 3 hours of treatment works more efficiently than the 1 hour treatment.

In the case of Carrara marble, however, an unexpected result was obtained. The 1 hour brushing treatment showed a much lower cumulative sulfur signal rate than the 3 hour treatment, indicating that less $CaSO_4 \cdot 2H_2O$ is formed after the shortest treatment time.

# Thanks for your attention