



X-ray Absorption Spectroscopy: a versatile technique

Laura Simonelli
CLÆSS beamline responsible



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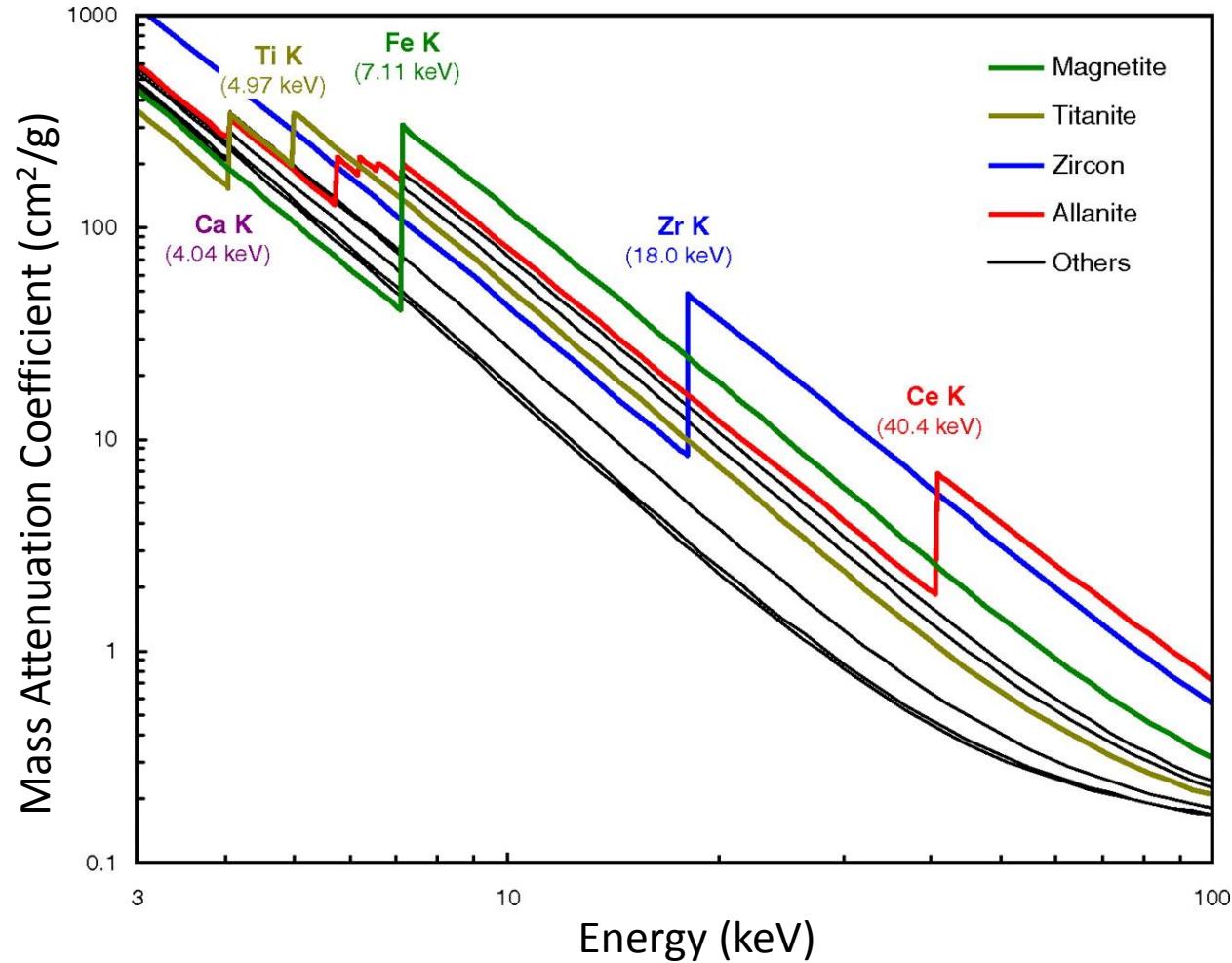
Photon absorption



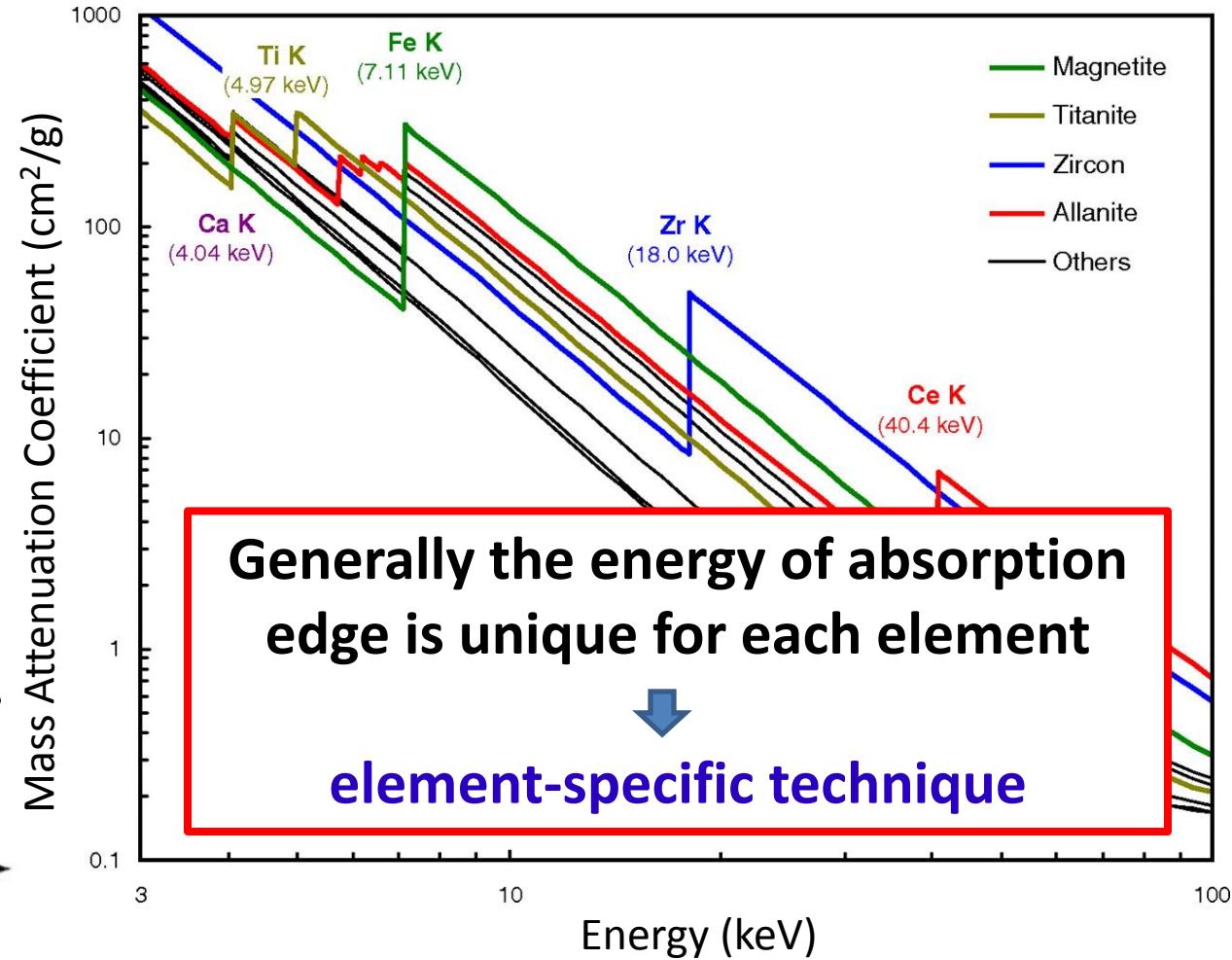
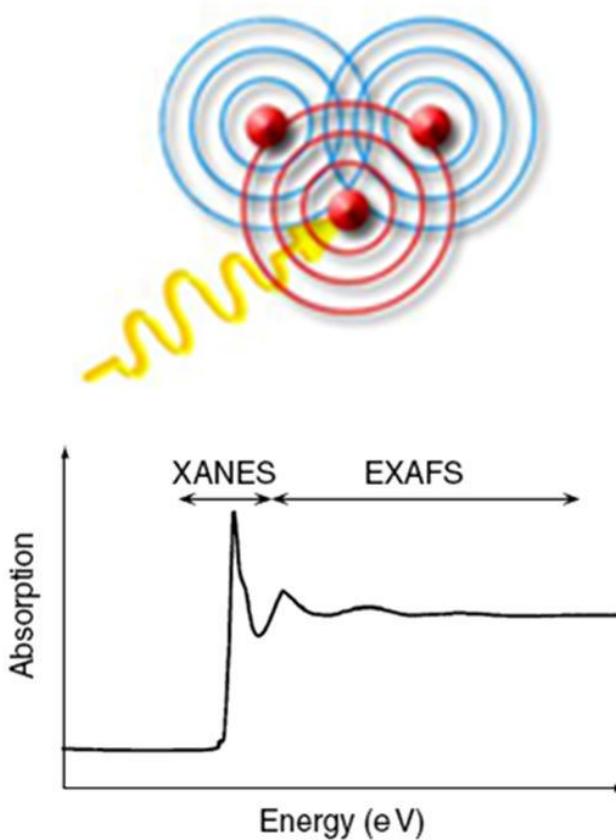
The synchrotron radiation interacts with the electrons bound in an atom.

Scattered radiation

Absorbed radiation



X-ray absorption spectroscopy (XAS)



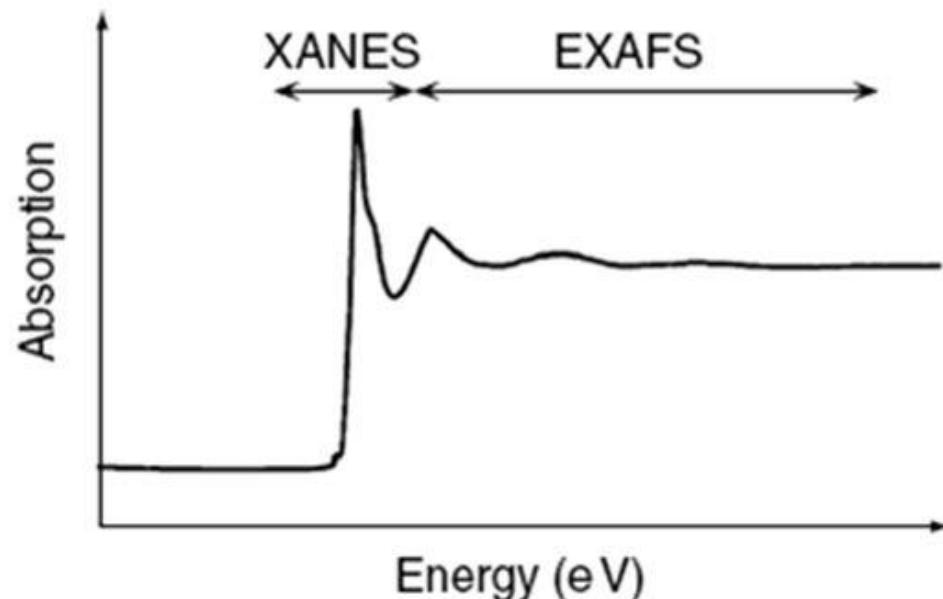
XAS: complementary information

XANES

- Oxidation state
- Unoccupied electronic states
 - Spin state
 - Local structure
- direct information about bond angles.

EXAFS

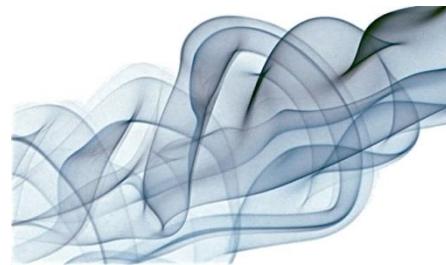
- Bond distances
- Coordination number
- Static and dynamic disorder



Which materials can be investigated by XAS?



**XAS is an element sensitive local probe that can be applied to any kind of materials:
Crystals, glass, liquids, etc...**





CLÆSS energy range: 2.4 – 62.3 keV

hydrogen 1 H 1.0079	
lithium 3 Li 6.941	beryllium 4 Be 9.0122
sodium 11 Na 22.990	magnesium 12 Mg 24.305
potassium 19 K 39.098	calcium 20 Ca 40.078
rubidium 37 Rb 85.468	strontium 38 Sr 87.62
caesium 55 Cs 132.91	barium 56 Ba 137.33
francium 87 Fr [223]	radium 88 Ra [226]
	57-70
	*
	103
	*
	262

k-edges
k-edges
L-edges



helium 2 He 4.0026	
neon 10 Ne 20.180	
boron 5 B 10.811	carbon 6 C 12.011
aluminium 13 Al 26.982	nitrogen 7 N 14.007
silicon 14 Si 28.086	oxygen 8 O 15.999
phosphorus 15 P 30.974	fluorine 9 F 18.998
sulfur 16 S 32.065	chlorine 17 Cl 35.453
argon 18 Ar 39.948	
gallium 31 Ga 69.723	germanium 32 Ge 72.61
indium 49 In 114.82	arsenic 33 As 74.922
cadmium 48 Cd 112.41	tin 50 Tin 118.71
tin 51 Tin 121.76	antimony 52 Sb 127.60
tin 53 Tin 126.90	tellurium 54 Te 131.29
iodine 54 I 131.29	iodine 54 I 131.29
xenon 54 Xe 131.29	
bromine 35 Br 83.80	
krypton 36 Kr 83.80	
seleium 34 Se 78.96	
brone 35 Br 79.904	
polonium 84 Po 126.90	
astatine 85 At 126.90	
radon 86 Rn 126.90	
ununoctium 114 Uuo [289]	

* Lanthanide series

lanthanum 57 La 138.91	cerium 58 Ce 140.12	praseodymium 59 Pr 140.91	neodymium 60 Nd 144.24	promethium 61 Pm [145]	samarium 62 Sm 150.36	europerium 63 Eu 151.96	gadolinium 64 Gd 157.25	terbium 65 Tb 158.93	dysprosium 66 Dy 162.50	holmium 67 Ho 164.93	erbium 68 Er 167.26	thulium 69 Tm 168.93	yterbium 70 Yb 173.04
actinium 89 Ac [227]	thorium 90 Th 232.04	protactinium 91 Pa 234.04	uranium 92 U 238.03	neptunium 93 Np [237]	plutonium 94 Pu [244]	americium 95 Am [243]	curium 96 Cm [247]	berkelium 97 Bk [247]	californium 98 Cf [251]	einsteinium 99 Es [252]	fermium 100 Fm [252]	mendelevium 101 Md [258]	nobelium 102 No [259]

** Actinide series



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k-edges
k-edges
L-edges



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neon 10 Ne 20.180	
chlorine 17 Cl 39.948	
argon 18 Ar 39.453	
bromine 35 Br 83.80	
krypton 36 Kr 83.80	
antimony 51 Sb 131.29	
tellurium 52 Te 126.90	
iodine 53 I 131.29	
xenon 54 Xe 126.90	
arsenic 33 As 108.88	
germanium 32 Ge 108.88	
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tin 50 Sn 118.71	
lead 82 Pb 208.98	
bismuth 83 Bi 208.98	
polonium 84 Po [209]	
astatine 85 At [210]	
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carbon 6 C 12.011	
nitrogen 7 N 14.007	
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Article
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Anal. Chem. 2012, 84, 10221–10228

Combined use of Synchrotron Radiation Based Micro-X-ray Fluorescence, Micro-X-ray Diffraction, Micro-X-ray Absorption Near-Edge, and Micro-Fourier Transform Infrared Spectroscopies for Revealing an Alternative Degradation Pathway of the Pigment Cadmium Yellow in a Painting by Van Gogh

Geert Van der Snickt,^{*,†} Koen Janssens,[†] Joris Dik,[‡] Wout De Nolf,[†] Frederik Vanmeert,[†] Jacob Jaroszewicz,[†] Marine Cotte,^{§,||} Gerald Falkenberg,[⊥] and Luuk Van der Loeff[¶]

[†]Antwerp X-ray Instrumentation and Imaging Laboratory, Department of Chemistry, University of Antwerp, Universiteitsplein 1, B-2610 Wilrijk, Belgium

[‡]Department of Materials Science and Engineering, Delft University of Technology, Mekelweg 2, NL-2628CD Delft, The Netherlands

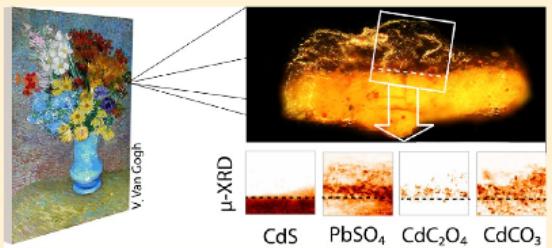
[§]Laboratoire du Centre de Recherche et de Restauration des Musées de France, CNRS UMR 171, Palais du Louvre, Porte des Lions, 14, Quai François Mitterrand, F-75001 Paris, France

[¶]European Synchrotron Radiation Facility, Polygone Scientifique Louis Néel, 6, rue Jules Horowitz, F-38000 Grenoble, France

[⊥]Deutsches Elektronen-Synchrotron, P06 Beamline, PETRA-III, Notkestrasse 85, D-22607 Hamburg, Germany

^{||}Conservation Department, Kröller-Müller Museum, Houtkampweg 6, NL-6731AW Otterlo, The Netherlands

ABSTRACT: Over the past years a number of studies have described the instability of the pigment cadmium yellow (CdS). In a previous paper we have shown how cadmium sulfide on paintings by James Ensor oxidizes to CdSO₄·H₂O. The degradation process gives rise to the fading of the bright yellow color and the formation of disfiguring white crystals that are present on the paint surface in approximately 50 μm sized globular agglomerations. Here, we study cadmium yellow in the painting "Flowers in a blue vase" by Vincent van Gogh. This painting differs from the Ensor case in the fact that (a)



Time instability of the pigment cadmium yellow (CdS)

Experimental approach

μ-XRD

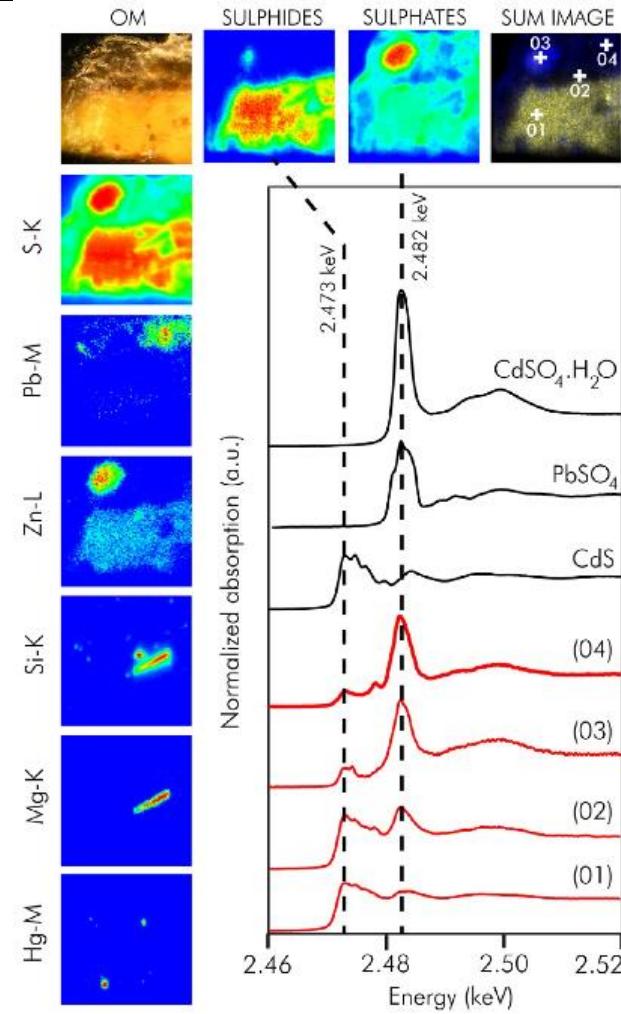
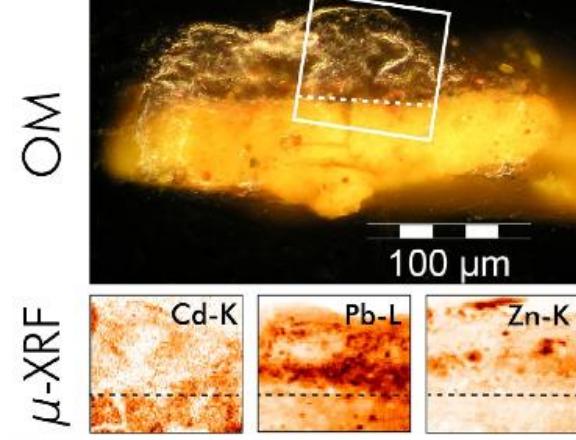
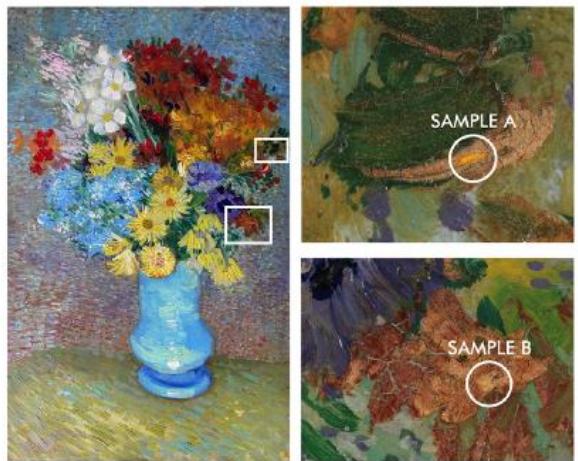
μ-XANES

μ-XRF

μ-FT-IR



No crystalline CdSO_4 compounds were identified on the Van Gogh paint samples → the observed degradation was initially caused by oxidation of the original CdS Pigment



The resulting opaque anglesite compound in the varnish, in combination with the underlying CdC_2O_4 layer at the paint/varnish interface, account for the orange-gray crust that is disfiguring the painting on a macroscopic level.



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	57-70
	*
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	261

k-edges
k-edges
L-edges



scandium 21 Sc 44.96	titanium 22 Ti 47.867	vanadium 23 V 50.942	chromium 24 Cr 51.996	manganese 25 Mn 54.938	iron 26 Fe 55.845	cobalt 27 Co 58.933	nickel 28 Ni 58.693	copper 29 Cu 63.546	zinc 30 Zn 65.39	gallium 31 Ga 69.723	germanium 32 Ge 72.61	indium 33 As 74.922	tin 34 Se 78.96	antimony 35 Br 79.904	arsenic 36 Kr 83.80
yttrium 39 Y 88.906	zirconium 40 Zr 91.224	niobium 41 Nb 92.906	molybdenum 42 Mo 95.94	technetium 43 Tc [98]	ruthenium 44 Ru 101.07	rhodium 45 Rh 102.91	palladium 46 Pd 106.42	silver 47 Ag 107.87	gold 48 Pt 112.41	cadmium 49 Cd 114.82	indium 50 In 118.71	tin 51 Sn 121.76	antimony 52 Sb 127.60	tellurium 53 Te 126.90	iodine 54 Xe 131.29
lutetium 71 Lu 174.97	hafnium 72 Hf 178.49	tantalum 73 Ta 180.95	tungsten 74 W 183.84	rhenium 75 Re 186.21	osmium 76 Os 190.23	iridium 77 Ir 192.22	platinum 78 Pt 195.08	gold 79 Au 196.97	mercury 80 Hg 200.59	thallium 81 Tl 204.38	lead 82 Pb 207.2	bismuth 83 Bi 208.98	polonium 84 Po [209]	astatine 85 At [210]	radon 86 Rn [222]
lawrencium 103 Lr [262]	rutherfordium 104 Rf [261]	dubnium 105 Db [262]	seaborgium 106 Sg [266]	bohrium 107 Bh [264]	hassium 108 Hs [269]	meitnerium 109 Mt [268]	unnilium 110 Uun [271]	ununnilium 111 Uuu [272]	ununbium 112 Uub [277]	ununquadium 114 Uuq [289]					

* Lanthanide series

** Actinide series

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Material Evidence for Multiple Firings of Ancient Athenian Red-Figure Pottery

Marc Walton,^{‡,†} Karen Trentelman,[‡] Marvin Cummings,[‡] Giulia Poretti,[‡] Jeff Maish,[§] David Saunders,[§] Brendan Foran,[¶] Miles Brodie,[¶] and Apurva Mehta^{||}

[‡]Getty Conservation Institute, Los Angeles, California 90049

[§]J. Paul Getty Museum, Los Angeles, California 90049

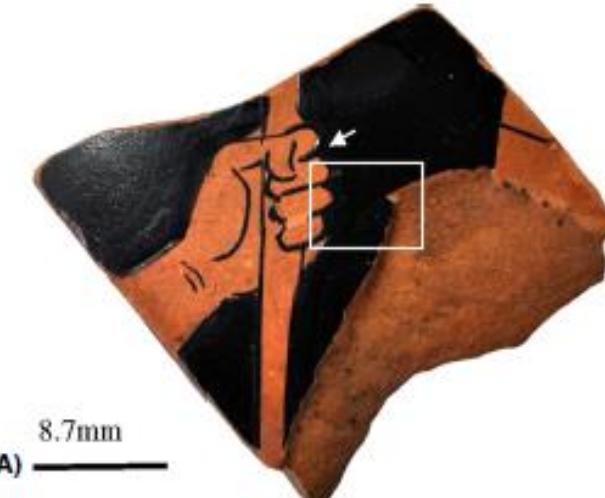
[¶]The Aerospace Corporation, El Segundo, California 90245

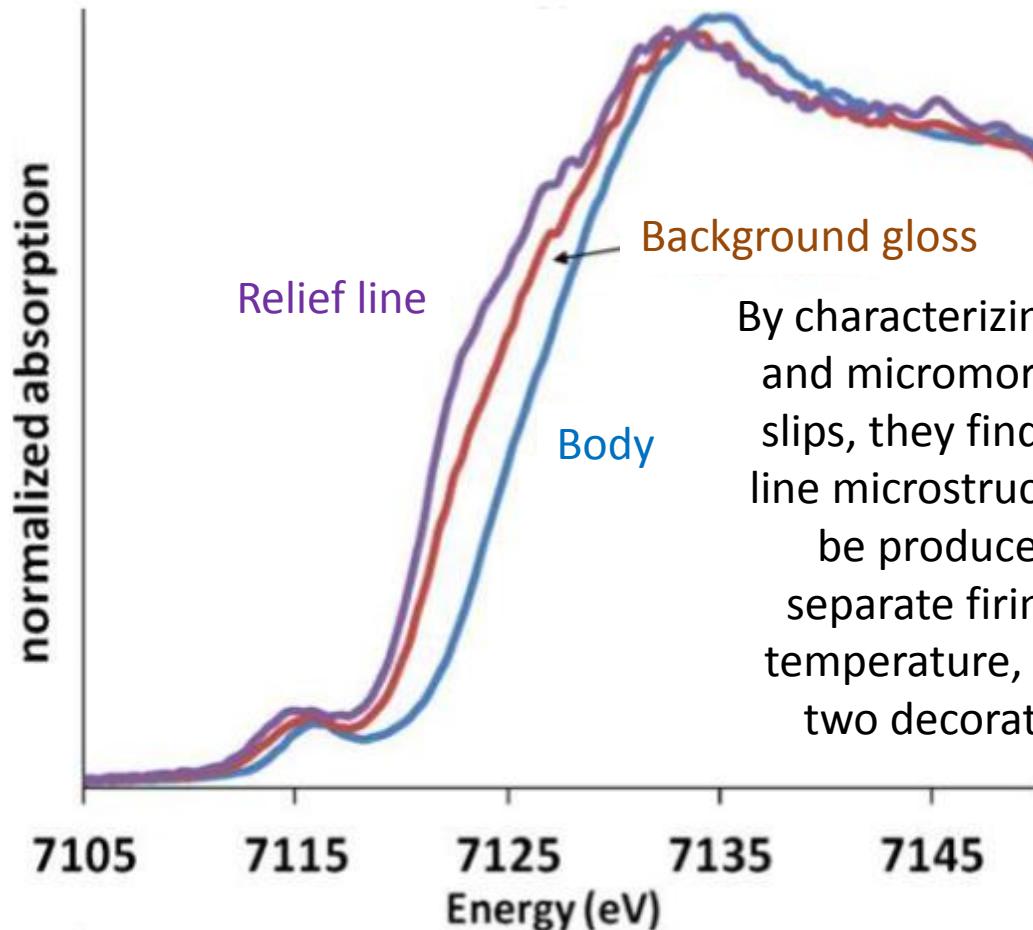
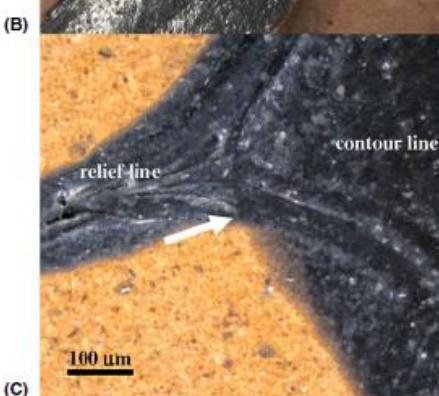
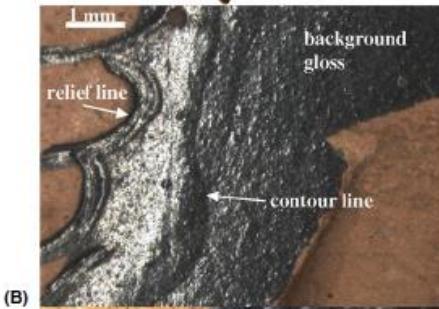
^{||}Stanford Synchrotron Radiation Lightsource, Menlo Park, California 94025

The production of Athenian fine ware pottery, produced between the 6th and 4th centuries B.C., required alternating the high-temperature kiln between oxidative and reductive environments during a single firing to create the iconic red and black decorative scenes. Here, we show that the production of this pottery was even more complex, with vessels subjected to two, or possibly more, firings in the kiln, with applications of slip between each firing. On a representative sherd, we compared three painted black decorative features—relief line, contour line, and background slip. Scanning transmission electron microscopy (STEM) of the slips revealed that the relief line had a more melted microstructure than either the contour line or background slip. By characterizing the chemistry and micromorphology of the slips, we find that the relief line microstructure could only be produced through a separate firing, at a hotter temperature, than the other two decorative features.

inclusions of the red colored mineral hematite (Fe_2O_3) in the slip are reduced to black colored magnetite (Fe_3O_4) as well as hercynite (FeAl_2O_4)^{10,11} and the clay platelets in the slip layer densify significantly,¹² forming a black colored layer that is impervious to reoxidation, whereas the coarser grain body remains porous. In the final, oxidizing, stage the slip remains black whereas the iron minerals in the porous body ceramic are reoxidized to Fe_2O_3 to produce a contrasting red color. Replication experiments based on this production model (summarized in Ref. [2]) have successfully mimicked the glossy black appearance of the slip that is commonly called black gloss and thus this process of firing has become the consensus view of how these slips were made.^{9,13–17} High-resolution mineralogical and morphological analysis of the slip material was undertaken to better understand this technology. Surprisingly, our results on the relative vitrification of the individual clay phyllosilicate units instead provide the

Several high temperature treatments between oxidative and reductive environments to create the red and black decorative scenes





By characterizing the chemistry and micromorphology of the slips, they find that the relief line microstructure could only be produced through a separate firing, at a hotter temperature, than the other two decorative features.



Confocal XANES and the Attic Black Glaze: The Three-Stage Firing Process through Modern Reproduction

Lars Lühl,^{*†‡§,L} Bernhard Hesse,^{†,l} Ioanna Mantouvalou,[†] Max Wilke,[‡] Sammia Mahlkow,[†] Eleni Aloupi-Siotis,^{§,L} and Birgit Kanngiesser,[†]

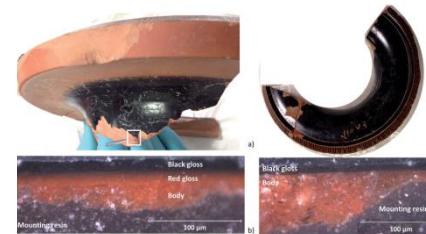
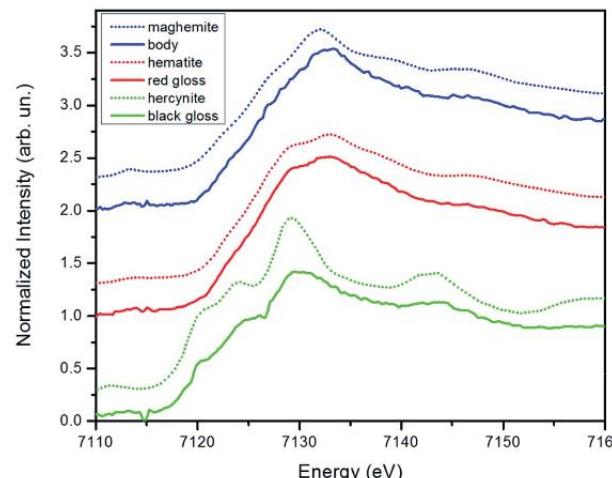
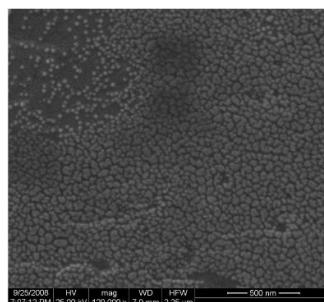
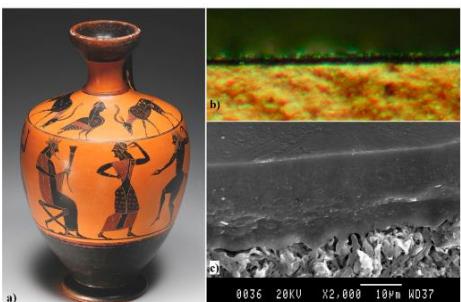
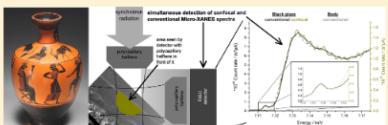
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Supporting Information

ABSTRACT: The decorated black- and red-figured Athenian vases (sixth and fifth century BC) and the plain black-glazed ware represent a milestone in our material culture due to their aesthetic and technological value; the Attic black glaze is of particular interest since it is a highly resistant potash-alumino-silicate glass, colored by magnetite nanocrystals (<200 nm). This study presents a new methodological approach for correlating the iron oxidation state in the black glaze layer with the manufacturing process by means of conventional and confocal X-ray absorption near edge spectroscopy (XANES). The enhanced surface sensitivity of confocal XANES is combined with conventional XANES resulting in higher counting rates to reliably evaluate the iron oxidation state (Fe^{3+}/Fe) of the surface layer. A detailed description of the new evaluation procedure is presented. The three-stage firing process was retraced by correlating selected attic black-glazed (BG) specimens from different periods (Archaic, Classical, Hellenistic) with laboratory reproductions. The modern BG specimens serving as reference samples were produced by following the three-stage firing process (i.e., under oxidizing-reducing-oxidizing (ORO) conditions) at different top temperatures, using clay suspensions of different particle size produced with treatment of raw illitic clays from Attica.



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Evidence for an unorthodox firing sequence employed by the Berlin Painter: deciphering ancient ceramic firing conditions through high-resolution material characterization and replication

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Chemical, electronic, and structural information (**2.4 - 68 keV**)

CLÆSS beamline performance:

- Beam size up to $100 \times 200 \mu\text{m}^2$
- Continuous scan (5-10 min scan)
- XAS in transmission and fluorescence detection mode
- XES – *from 6.4 to 9.3 keV (K β and K α of most of the transition metal)*
- Stable beam-position and beam-size (200-500 μm) in the full energy range
- Temperature control from 80 K to 800 K; or from 4 K to 300 K
- In-situ cell for solid-gas reactions (80 K – 700 K)

