LABORATORY: CNR ISPC - XRAYLab

NAME OF THE INSTRUMENT

Mobile XRF confocal scanner developed within the XRAYLab

GENERAL DESCRIPTION:

Confocal XRF (CXRF) is a powerful method for stratigraphic investigations (1D) and elemental imagining in three dimensions (3D) of samples with complex depth profiles. The CXRF method is non-destructive, requires no sample preparation and can be performed in-situ. CXRF is particularly suited for the investigation of layered paints in paintings.
Confocal XRF is based on the detection of the sample X-ray fluorescence, induced by an X-ray beam focused on the micrometer scale by polycapillary optics. The direction of detection is defined by another polycapillary mounted in front of the detector window, with comparable focusing power. In this fashion, the point of origin of the X-ray fluorescence detected is well defined in the three dimensions, as the point where the direction of the beam meets the direction of detection, or, equivalently, where the foci of the two polycapillary lens. The investigated volume is therefore defined by the overlap of the primary beam focus with the focus of the detection polycapillary. By changing the sample position, and its distance from the scanner, its volume can be mapped. The scan can be performed along the sample depth only (1D measurement), or in all three dimensions (3D imaging).
In the first case, CXRF provides information on the chemical composition of the different layers while, in the last case, it provides 3D imaging of the chemical elements spatial distribution, in both cases with spatial resolution in the micrometer scale. The maximum depth that can
be analyzed by CXRO depends on the specific material investigated, since Xrays penetrability and absorption are both material specific, however the typical accessible thickness is in the order of 100-150 microns.

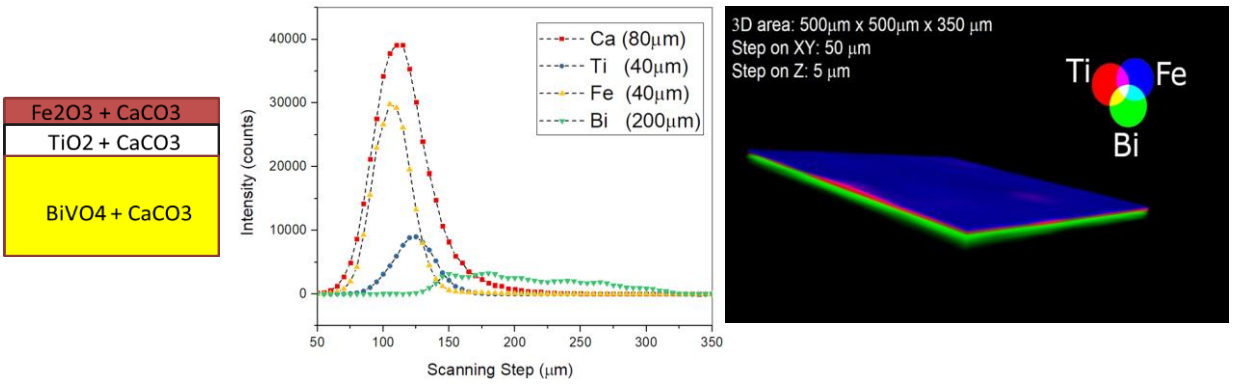
Practical guide for the choice of CXRF method of ISPC

Materials: all inorganic layered materials
Typical applications: paint pigments on any medium, enamels, glazed pottery, enrichment coatings, surface degradations and corrosions
Sample positioning: vertical and horizontal
Type of analysis: point by point or continuous, not-destructive, in-situ (also on scaffolding)
Measuring times: about 1000 seconds for 1D measurements, hours for 3D mapping
Size of the foci in the measuring position: about 10 microns (at the energy of Mo)
Spatial resolution: down to 3 microns
X-source characteristics and parameters: Mo anode, 50kV and 0,6 mA (30W power)
Additional method available: micro-XRF single spot and imaging
Analytical tools: CXRF analysis is video-guided by an high-resolution optical microscope; 1D stratigraphy and 3D mapping are output live during the measurements through deconvolution of the XRF spectra (in absence of artifacts); the analysis software allows to operate on-line; image-

processing, RGB elemental correlations, scatter plots of elemental correlations, statistical analyses PCA ICA and NMF, analysis in specific region of interest, maxima dispersion (to identify trace elements or localized inclusions).

TECHNICAL DESCRIPTION:

The CXRF scanner consists in a spectroscopic head equipped with a low power (30W) microfocus X-ray tube using a Mo anode coupled with highly focusing polycapillary optics. The focus of the primary beam that emerges (from both the tube and the polycapillary) is near 10 microns in size, at 3.5 mm from the end of the polycapillary. The detection system is oriented 90 degrees off from the primary beam direction and comprises an SDD detector (with 50 mm2 active area and 130 eV energy resolution at 5.9 KeV) coupled to a second polycapillary (half-lens) with a focus on the sample of also approximately 10 microns. The overlaps between the two foci defines the analytical volume with which the sample can be scanned, either along its depth Z (1D spot) or in all three dimensions XYZ (3D mapping).
On the spectrometer is mounted an optical microscope with large focal distance and high resolution to perform video guided CXRF investigations. In a single scan, the instrument can cover a volume of 20x20x30cm3, thanks to a 3-axes automated motorized system. Spatial resolution is in the order of 3-5 microns.
The CXRF system operates in a continuous mode with a maximum velocity of 50 mm/sec. To ensure sufficient statistics, measurements are typically acquired for a time that varies between 100msec to 3 sec, per pixel. To reduce the total acquisition time, it is recommended to limit the use of 3D mapping mode to small area of particular interest in the sample and perform systematic studies with the point-by-point stratigraphic method.
The system is equipped with a Central Unit (CU) that controls the measurements operating parameters and enforces security checks for the safeguard of the analyzed sample. Through the CU, scans can be analyzed in real time and 3-d images (or depth profiles) are made available to the user during the measurement. In this fashion, it is possible to define and adapt the investigation strategy on-line, including the individuation of spots with particular relevance to be investigated more attentively.



CXRF analysis of a pictorial sequence. The results of 1D stratigraphic analysis and 3D mapping are visible.

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