LABORATORY: CNR ISPC - XRAYLab

NOME STRUMENTO

XRD imaging scanner developed by XRAYLab

GENERAL DESCRIPTION

X-ray diffraction (XRD) allows to determine the mineralogical phases characterizing the materials under study. Unlike elemental analytical techniques such as the XRF technique, the XRD technique provides information on the nature of chemical compounds (even in complex mixtures) and their structure. Given the crystalline nature of different types of ancient materials, the XRD technique is particularly suitable for the analysis of archaeological and historical-artistic samples and their degradation products. The main limitation of the XRD technique generally applied in situ or in the laboratory is to be punctual and to provide only local information. ISPC's XRAYlab laboratory recently developed an innovative mobile XRD imaging system capable of providing images of the distribution of crystalline phases on macroscopic samples. This information is crucial for a better knowledge of materials and manufacturing techniques and for the study of degradation processes.
The mobile XRD mapping system developed by ISPC's XRAYLab operates the diffraction of polycrystalline samples in grazing geometry and parallel rays with the advantage of keeping the X source, detector and sample fixed and without the need for angular scanning of the Bragg angles. This experimental setup allows to obtain accurate XRD patterns, less affected by angular shifts and a higher resolution. The technique is non-invasive, and no sample preparation is required.

A brief guide to choosing the ISPC XRD technique
Samples: polycrystalline materials
Optimal cases of application: paints on any substrate, metals, patinas of corrosion and decay.
Sample placement: vertical, horizontal
Type of application: non-destructive and in situ (also on scaffolding with high stability).
Measurement times: Measurement times 8h for a 10x10cm area with 1s steps.
Characteristics and parameters of the X source: Cu anode, 50kV and 0.6mA (power 30W)
Beam size at the measurement point: rectangular shape of 0.2x1mm
Angular resolution of diffraction patterns: about 0.2 degrees
Detector features: Si-strip (1280 strips) with angular coverage in 2theta 15-60 degrees.
Other techniques present in the instrument: simultaneous acquisition of phase images (XRD) and elemental images (XRF) in the same measurement point.

TECHNICAL DETAILS

The XRD imaging system developed by ISPC's XRAYLab laboratory is a mobile system optimized to operate in situ. The system consists of 3 XYZ linear axes with a stroke of 50x50x20cm on which the XRD / XRF measurement is mounted. This is equipped with a 30W X microfocus source with Cu anode coupled to a polycapillary optic collimated with a rectangular slide. The dimensions of

the beam at the measuring point are 0.2x1mm. The detection of the diffraction pattern diffused by the samples takes place using a Si-strip detector sensitive to position and energy that covers an angular range from 15 to 60 degrees in 2theta. During the measurements it is possible to simultaneously acquire the XRD pattern and the XRF spectrum for the same measurement point. This mode is obtained by means of an ancillary SDD detector for XRF always positioned on the measuring head and which operates in parallel, by means of dedicated electronics, to the Si-strip diffraction detector. Finally, on the measuring head there is a laser sensor that monitors the distance of the beam from the sample moment by moment and dynamically corrects the position following the morphology of the surface under examination. XRD / XRF measurements take place through a ste-by-step scan (maximum air 50x50cm) with an acquisition time per step equal to 1s. The analysis of XRD and XRF data and the production of images of the distribution of the phases (XRD) and of the elements (XRF) takes place using proprietary software programmed on the system.

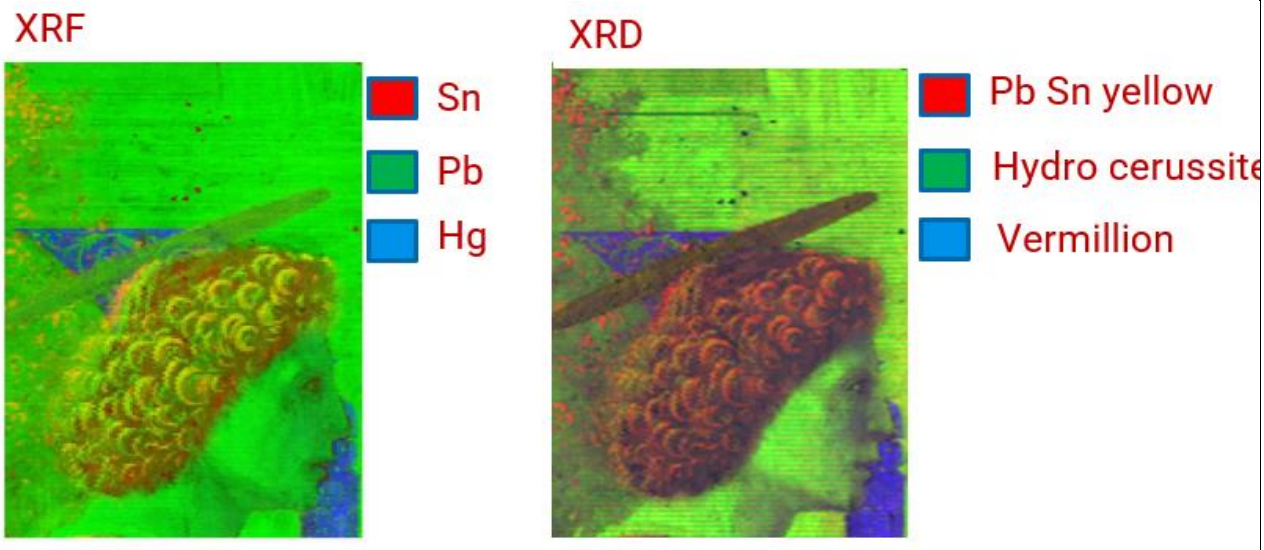

Figure: The scanner for XRD mapping in place at the National Gallery of Umbria in Perugia in the analysis of a painting by Piero della Francesca. The results of simultaneous XRD and XRF mapping are shown below

FURTHER INFORMATION

- F.P. Romano, et al., The new version of the portable XRD system of the LANDIS laboratory, Il Nuovo Cimento 121 (2005) 881-885.
- G. Gatto Rotondo, et al., Non-destructive characterization of fifty various species of pigments of archaeological and artistic interest by using the portable X-ray diffraction system of the LANDIS laboratory of Catania (Italy), Microchemical Journal 96 (2010) 252–258.
- F.P. Romano et al., The compositional and mineralogical analysis of fired pigments in Nasca pottery from Cahuachi (Peru) by the combined use of the portable PIXE-alpha and portable XRD techniques, Microchemical Journal 99 (2011) 449–453.
- L. Pappalardo, et al., The complementary use of PIXE-alpha and XRD non-destructive portable systems for the quantitative analysis of painted surfaces, X-Ray Spectrometry, 37 (2008) 370–375.

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