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Application of an innovative laboratory-based phase contrast X-ray imaging system for the study and characterisation of cultural heritage materials

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Abstract: X-ray imaging is a standard tool for the non-invasive inspection of the internal structure of materials. Conventional X-ray absorption imaging is effective for characterising medium density samples, but presents some limitations with weakly absorbing material. Phase contrast imaging addresses this limitation by exploiting interference patterns generated by spatial variations in the

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refractive index, enhancing the detectability of fine structural details. The PITCH (Phase-contrast X-ray Imaging setup with a Talbot interferometer and a liquid anode source for Cultural Heritage application) project, funded by MUR in the framework of PRIN2022, aims at designing, developing and characterising a laboratory grating interferometry phase contrast imaging setup based on a liquid-anode X-ray source, for the study and characterisation of cultural heritage materials and artifacts, both for radiographic and tomographic analyses. This paper illustrates the new setup and presents the preliminary results of its characterisation that show a simulated visibility of about 3%. More tests and modifications on the setup will be implemented.

Keywords: Computerized Tomography (CT) and Computed Radiography (CR); X-ray radiography and digital radiography (DR)

2026 JINST 21 C06016

Contents

2026 JINST 21 C06016

1 Introduction

The use of X-rays has been a standard tool for studying non-invasively the internal structure of objects since W.C. Röntgen's discovery in 1895. In the past decades, X-ray Imaging, both Digital Radiography (DR) and Computed Tomography (CT), has found application in different fields, from medicine, biology and engineering disciplines, to palaeontology, anthropology and cultural heritage [1]. Its fundamental working principle is based on the differential absorption of X-rays passing through the sample, which generates contrast due to differences in the X-ray attenuation coefficients of the materials. Conventional absorption imaging techniques follow this principle; they provide excellent results when relatively high attenuation exists, but lead to poor image quality with weakly absorbing samples, such as materials composed of light elements or soft tissues [2]. Being based on a different physical principle, X-ray Phase Contrast Imaging (PCI) can overcome the limitations of conventional methods when low-Z or weakly absorbing materials are analysed. The physical difference is related to the complex refractive index ($n = 1 - \delta + i\beta$), whose imaginary (β) and real (δ) parts are related to the attenuation and the phase shift of X-rays, respectively. β and δ have very different dependences upon X-ray energy: for most materials and over the range of energies commonly employed in X-ray imaging, δ is much larger than β — typically up to 1000 times larger. This means that, if properly exploited, phase effects can lead to a dramatic increase in image contrast [3]. X-ray PCI allows the visualisation of low-Z and soft materials by relying on the phase shift or the refraction of X-rays caused by the sample.

The University of Torino together with the University of Milano-Bicocca, the Istituto Nazionale di Fisica Nucleare (INFN) of Torino and the Centro Conservazione e Restauro La Venaria Reale, designed and developed a laboratory setup for X-ray PCI with a Talbot-Laue interferometer and a liquid anode X-ray source for the analysis of Cultural Heritage (CH) materials. This research is part of the PITCH and Vi_Hi projects, funded by MUR in the framework of PRIN2022 and by INFN respectively.

This paper presents the new system installed at University of Torino and the first results of the preliminary characterisation on test samples that are part of the PITCH project.

1.1 The PITCH project

There are different ways of exploiting X-ray PCI, using both synchrotron [4] and laboratory [5] sources. Many synchrotron-based methods take advantage of the unique properties of the primary beam and also make use of specialized X-ray optical elements to generate X-ray beams with a high degree of both spatial and chromatic coherence, a crucial property for PCI [6].

Among the different PCI methods [2], X-ray Grating Interferometry (GI), which provides simultaneously differential phase and scattering images in addition to the conventional absorption-based image, allows the design of systems that could be implemented in a laboratory setup. By using a liquid-metal-jet source [7] such a system allows shortening the scan times and/or increasing the signal-to-noise ratio, thereby opening extremely interesting perspectives for the analysis of CH materials.

The INFN of Torino from 2019 to 2021 designed and developed a prototype of an X-ray grating-based phase-contrast imaging scanner in a laboratory setting for the 4D GRAPH-X project [8]. The goal was implementing a prototype of a dynamic X-ray Talbot-Lau imaging system suitable for a range of applications in medicine with major focus on lung imaging. The 4D GRAPH-X was the first step leading to the design of the new laboratory system developed in the framework of the PITCH project. PITCH aims at 1) designing, building and validating the first Italian fully operational, beyond the state of the art, phase-contrast X-ray grating interferometer based on a liquid-anode source; 2) investigating and developing different data acquisition methods and algorithms for signal extraction and tomographic reconstruction; 3) optimising the setup for the analysis of different CH materials to both characterise and understand their related issues; 4) analysing real artifacts coming from museums prioritising samples relevant to cultural heritage preservation projects.

2 Materials and methods

2.1 Experimental setup

The new laboratory system is installed at the University of Torino, at the HiBriX Lab laboratory. The system shown in figure 1 consists of a source, a detector and a set of three gratings (G0, G1, G2) in a symmetric configuration with a total length of 1.5 meters and at the third Talbot order (T.O.). The system is installed on an anti-vibration table. A railway system helps the movements of the different components during the alignment. The current setup configuration works with a design energy of 45 keV. This design energy matches the 5.25 µm pitch of the gratings.

The detector is a Flat panel CMOS, scintillator: CsI, with a pixel size: $49.5 \times 49.5\ \mu m^2$ and pixel number: 2304×2940, already employed on the 4D GRAPH-X setup. The X-ray source Excillum MetalJet D2+ is based on a liquid metal-jet anode (Ga-In-Sn alloy), has a focal spot size of 5–30 µm range, a liquid anode's jet diameter of ~ 70 µm, max power 250 W and it operates in Cone-Beam configuration. This type of source allows to have 10× brightness compared to a solid anode, higher flux and the possibility to exploit an energy range of 40–160 keV. In this range X-ray PCI provides improved sensitivity for weakly absorbing materials compared to conventional (i.e. absorption-based) imaging.

At the moment, no filters are applied to the MetalJet source; in the future filters of different materials and thicknesses (e.g. 1 mm Ti, 2 mm Al) will be implemented.

The Talbot-Lau interferometer has a three-grating setup (figure 2a) each with different functions: a gold source grating G0 (90 µm thickness), giving spatial coherence to the X-ray beam; a silicon phase grating G1 (58 µm), creating the interference pattern; a gold analyser grating G2 (90 µm), sampling

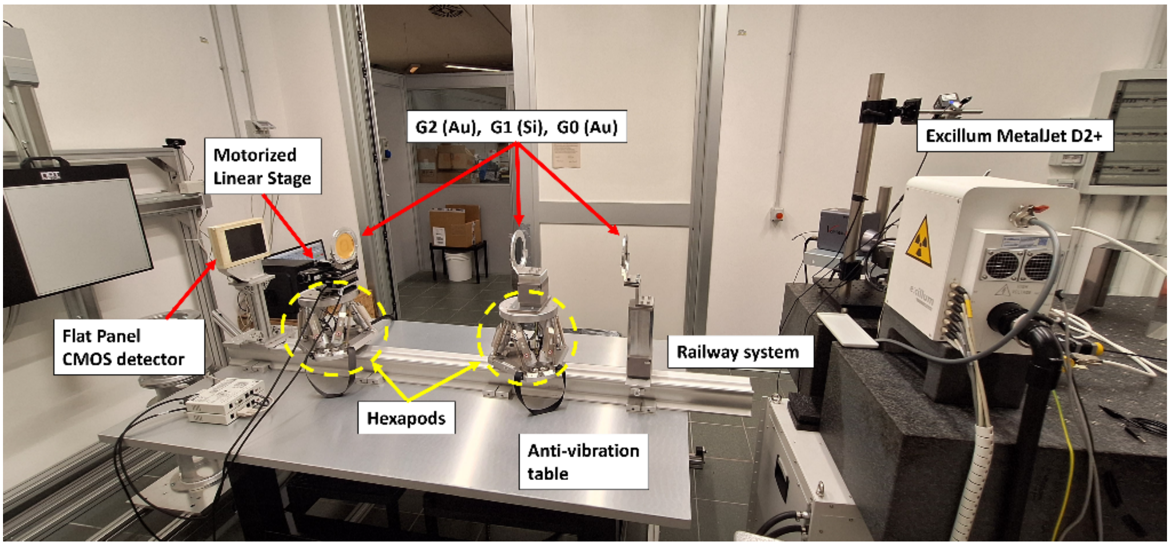

Figure 1. The laboratory system installed at the HiBriX Lab at University of Torino. The geometrical distances between the elements are: X-Ray source – G0 = 370 mm; G0 – G1 = G1 – G2 = 750 mm; G2 – detector = 144.7 mm.

the modulated wavefield and enabling extraction of absorption (ABS), differential phase contrast (DPC) and dark-field (DF) images (figure 2b). G1 and G2 sit on hexapods (*Hexapods HXP100P by Newport*) mounted on the railway system, moreover G2 is equipped with a motorised goniometer (*Motorized Linear Stage VP-25XA by Newport*) which allows the variation of its angle for more precise alignment. The gratings' pitch is 5.25 µm and the field of view (FoV) size is 7×7 cm^2. The sample holder is currently positioned between G0 and G1, in the next future it will be implemented with a rotatory stage for CT acquisitions. All motors in the setup are controlled via an in-house developed LabView 2019 SP1 software, which also controls the acquisition of the X-ray detector. The code allows for the remote alignment of different components and the automatic execution of different acquisition sequences for both radiographic and tomographic modalities.

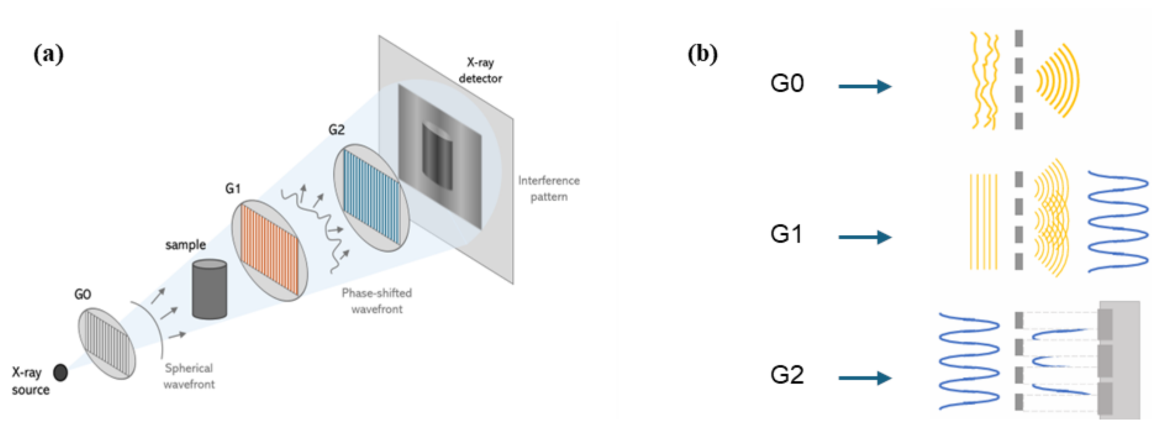

Figure 2. (a) Model of the interferometer with a symmetric configuration with three gratings (G0, G1, G2), 5.25 µm gratings pitch and 45 keV energy at the third T.O; the distance between the gratings is 750 mm; the sample is between G0 and G1. (b) Each grating has a specific function.

2.2 Cultural heritage materials

To achieve the PITCH project's aim to characterise a wide variety of CH materials, a selection of different original fragments and samples related to the CH field has been made. The creation of mock-ups will enable the optimisation and characterisation of the set-up. This selection has been made considering the request of restorers and museum conservators, the possible archaeological, scientific or restorative questions that can be answered by PCI and the availability of the materials. Both organic and inorganic materials were selected together with complex systems that reproduce the condition of real artifacts. Here are some examples: textile materials; leather; parchment; cartonnage; wood; shells and eggs from archaeological findings; mummified remains; pearls; pigment layers; metallic yarns. Different samples' geometries will be tested as well.

The analysis with the new setup will be carried out to characterise the materials and to understand possible related issues e.g. degradation byproducts, porosity, conservation state, manufacturing technology, differences among species. The preliminary analysis on mock-ups will lead to the study of real artifacts coming from museums and other cultural institutions.

2.3 The data acquisition system

The data acquisition system will implement two methods: Phase Stepping (PS) and Moiré imaging. As explained in [9] and [10], with both approaches the ABS, DPC and DF signals are extracted through Fast Fourier Transform (FFT) analysis of the acquired data. When performing a CT acquisition, where the procedure needs to be repeated for each CT projection, the Moiré imaging approach, being a single-shot method, significantly shortens the acquisition time, but slightly worsens the spatial resolution [11] with respect to the PS method which is longer, since at least five acquisitions are needed for each CT projection.

For the purposes of the PITCH project, custom-made software in Julia and Python code have been specifically developed for signal extraction and CT reconstruction, exploiting also open-source software such as the ASTRA-Toolbox [12] packages.

The data treatment consists in the FFT analysis implemented for both PS and Moiré imaging, moreover Sine function fitting will be also implemented for PS.

The reconstruction software will operate on raw data to obtain ABS, DPC and DF images [13].

3 Preliminary characterisation results

The optimisation of the GI setup is performed with a LabVIEW program specifically developed for the new system for the alignment of the gratings. In the initial phases, both G1 and G2 undergo translational motion provided by hexapods, while rotational adjustment is applied only to G2 via the motorised goniometer, to obtain the interference pattern without the sample, until the fringes are parallel with each other. To define the performance of a gratings system the visibility curve needs to be calculated [8]. The visibility is the normalised magnitude of the first Fourier component of the Phase Stepping Curve (PSC). For a harmonic oscillation pattern, the visibility (V) is defined as the oscillation amplitude normalised to the mean intensity.

Two macros in ImageJ software were created and used to correct and visualise the acquired images, by subtracting the dark acquisition and normalising the region of interest of the gratings with the background area.

From the data calculated with the two macros the PSC and the visibility (equation (3.1)) are obtained.

$$V = \frac{I_{\max} - I_{\min}}{I_{\max} + I_{\min}} \tag{3.1}$$

Where $I_{\max}$ and $I_{\min}$ refers to the maximum and minimum intensities of the interference fringes respectively.

Different tests were done to align the setup, varying the acquisition parameters (e.g. voltage, current, focal spot size, power of the X-ray source and integration time of the X-ray detector) to find the optimal conditions in terms of visibility (expected to be 5–10% to ensure good image quality), initially without the presence of samples between G0 and G1. Different phase steps were considered for the various measurements: 7 steps of 1.5 µm and 21 steps of 0.5 µm. The measurement conditions were chosen considering how the visibility changed step by step: integration time 5 s; voltage 85 kV; focal spot size 36.7 × 11.9 µm; power 140 W. Maintaining the same parameters, the visibility values are almost the same comparing the acquisition with 7 and 21 steps (3.2% and 3.3%). The variation of the other selected parameters may result in a change of the visibility value. For example, by varying the integration time (2.5–15 s), keeping constant the other parameters (85 kV; 140 W; 36.7 × 11.9 µm; 21 steps), the visibility remains constant (figure 3a). On the contrary, if the voltage increases (65–145 kV) the visibility decreases (figure 3b); in this case the integration time range is adjusted (range 18–3.5 s) to keep the detector counts nearly constant, while the other parameters are calculated by the software each time the voltage varies except for the current that remains constant (1.7 mA). The decrease in visibility is expected since the design energy is 45 keV.

The integration time is selected from the Sapera CamExpert software. The X-ray parameters are selected from the MetalJet D2+ software.

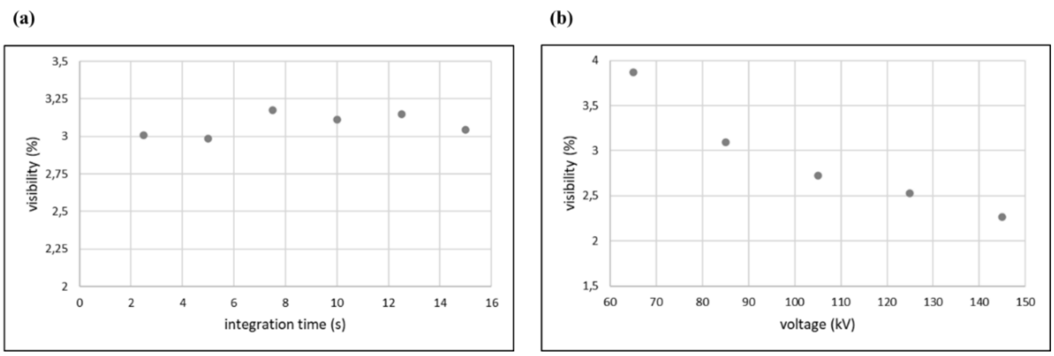

Figure 3. Visibility vs. (a) integration time and (b) voltage. (a) By varying the integration time, the visibility is constant around 3%. (b) The visibility decreases from 3.9% to 2.3% as the tension increases.

Preliminary 2D acquisitions were performed on selected test samples [13]. These measurements were intended as an initial qualitative assessment of the system response. A comprehensive quantitative characterisation requires dedicated optimisation of the acquisition parameters and will be the subject of future works.

The next steps will be continuing the optimisation by varying the focal spot size and trying different beam filters to study the changes in the visibility value. Other configurations will be implemented, and more test objects will be created and characterised as well.

2026 JINST 21 C06016

4 Conclusions

The present paper illustrates the new laboratory setup and the preliminary characterisation results obtained. The system works with a symmetric configuration with 5.25 µm gratings pitch and 45 keV energy, at the third Talbot order, that allows the analysis of small and medium-size objects related to CH field. The simulated visibility of 5–10% expected with this configuration wasn't achieved in this first phase, but more tests and modifications on the setup will be implemented to obtain better results, as noise in PCI is highly dependent on the visibility.

The high contrast provided by the X-ray PCI technology, coupled with the high resolution that the laboratory setup, developed in the framework of the PITCH project, will implement, could greatly facilitate the collection of detailed structural information on a significantly larger number of samples. With the new system, laboratory analysis of artworks and archaeological finds using X-ray PCI, currently limited to a few synchrotron facilities, will become possible, rendering this analytical approach more feasible and accessible.

The outcomes of the PITCH project would help archaeologists, anthropologists, art historians and restorers in better understanding the production techniques, the materials and the state of conservation of the objects under analysis.

Acknowledgments

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2026 JINST 21 C06016

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