Dynamic multi-modal x-ray imaging (DM-MX) for additive manufacturing and beyond

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ABSTRACT

In this project we propose the use of a beam tracking implementation to perform time resolved multi-modal X-ray imaging. As a first application we targeted melting processes occurring during laser additive manufacturing, since we believe this research field will benefit from improved visualization of density gradients provided by phase imaging as well as by detection of sub-pixel inhomogeneities allowed by X-ray small angle scattering (also referred to as dark-field imaging). Our pilot experiment was successful, providing for the first time a full multimodal, complementary set of images with micron and millisecond spatial and time resolution, respectively.

Multi-modal X-ray imaging; laser additive manufacturing; dynamic imaging; dark field imaging.

1. INTRODUCTION

X-ray multi-modal imaging is based on the retrieval of phase changes and small angle scattering occurring when X-rays pass through a sample, in addition to conventional attenuation. The interest in these new contrast channels is increasing since phase imaging provides superior contrast on low-attenuating materials (e.g. soft tissues), while ultra-small angle scatter (or dark field (DF)) imaging highlights microscopic structures of objects below the system's resolution [1,2]. Concurrently, the availability of high-flux synchrotron beamlines is enabling dynamic X-ray imaging with sub-millisecond temporal resolution to investigate rapidly changing phenomena such as those occurring during phase transitions. However, so far dynamic imaging has been mostly restricted to conventional attenuation [3,4]. In this project we developed a method that enables performing multimodal dynamic imaging with millisecond time resolution, simultaneously providing fully quantitative transmission and phase images as well as dark field ones. The method is based on the continuous translation of an optical element (an absorbing mask) during the acquisition of an image sequence. The mask shapes the x-ray beam into "beamlets" which are (dynamically) dampened, deflected and broadened by the sample, the analysis of which effects yields attenuation, differential phase and DF images, respectively.

The successful completion of this project introduces a new technological capability that was not previously available. As a first demonstrator, we targeted the imaging of molten pool dynamics in additive manufacturing, where we expect the new technique could enable a quantitative determination of density gradients through the differential phase contrast (DPC) channel, and detect the presence of additional features (e.g. defects, solid/liquid phase, grain structure, pores) through a combination of DPC (for resolvable features) and DF (for unresolvable ones). However, once the technology is established, other research fields where dynamic imaging is needed would benefit from it, such as pre-clinical imaging.

In a first pilot experiment, we collected dynamic, multi-modal images of laser-induced melting processes in metal layers and metallic powders used in additive manufacturing. We achieved spatial and temporal resolution in the range of microns and milliseconds, respectively. We improved the visibility of microstructures forming during oxidation process thanks to DFC and DF, and demonstrated the method's ability to detect density changes occurring in the early stages of the melting process. Notably, time resolution was limited by the motor used to translate the optical element, which can be very easily improved: sub-milliseconds time resolution should be easily within reach in the next experiments.

2. STATE OF THE ART

For over a century, X-ray imaging has been based on attenuation contrast. However, X-rays experience a small change in propagation speed while travelling through different materials. This effect is at the basis of "phase" contrast, and is typically orders of magnitude stronger than changes in attenuation. As a result, it has been widely investigated for medical applications, where it has the potential to enable the detection of almost "x-ray transparent" features [1]. The same principle leads to multiple microscopic changes in the direction of x-rays as they travel through heterogeneous materials; this generates ultra-small angle scatter or DF contrast, which has the potential to quantitatively reveal sub-pixel inhomogeneity [5,6]. The retrieval of these three signals is usually referred as multi-modal X-ray imaging. In this project, we implemented multimodal imaging through a modification of the beam tracking (BT) approach [7]. BT is based on strongly structuring the x-ray beam by subdividing it into beamlets before it hits the sample. If a detector with a spatial resolution sufficient to resolve the individual beamlets is used, their decrease in intensity, lateral displacement and broadening can be directly extracted from individual image frames, yielding attenuation, DPC and DF images, respectively. An additional feature of our approach is the ability to provide tuneable spatial resolution, allowing for multiscale investigations [8]. So far, multimodal imaging has been restricted to static applications. However, 3rd generation synchrotron radiation facilities provide sufficient flux to enable dynamic imaging with microsecond resolution [3]. So far, most applications of dynamic x-ray imaging have been based on attenuation contrast [3,4], with exceptions based mostly on propagation-based phase contrast imaging (PCI). Propagation-based PCI is the simplest implementation of phase imaging [9]; however, it is not quantitative, and does not provide access to scattering or to a pure phase signal [10].

3. BREAKTHROUGH CHARACTER OF THE PROJECT

This project aimed at incorporating for the first time dynamic capabilities into X-ray multimodal imaging, simultaneously providing quantitative transmission, DPC and DF signals with micrometric spatial and millisecond time resolution. This is a significant step forward over previous attempts based on either pure attenuation or propagation-based PCI: while the latter exploits phase effects, it yields a single image, which normally does not allow to quantitatively determine the phase shift, and provides no access to the DF channel.

While we believe several fields will benefits of applications of dynamic multi modal imaging, we decided to initially focus on the study of molten pool dynamics in laser additive manufacturing (LAM), where we believe the multimodal capability can provide significant advantages. LAM enables the 3D printing of metal components with previously unattainable structural complexity. LAM has the potential to become the new standard in manufacturing, but defect formation due to our still limited knowledge of the underpinning physical processes can lead to sub-optimal products. X-ray dynamic imaging has already demonstrated the ability to allow a better understanding of the physics behind LAM [3], and we believe that the increased information provided by multimodal imaging can lead to new insights into melting processes. In particular, on top of the information made available by conventional x-ray imaging, we expect our multimodal dynamic imaging method to improve the visualization of faint density gradients in melting volumes through DPC, detection of defects, grains and unfused powder through DF, and quantitative assessment of material homogeneity via a combination of both. Looking forward, applications will not be limited to additive manufacturing; once the concept has been demonstrated, it will find applicability in a wide variety of other application areas posing similar requirements, from energy to healthcare [4,9].

4. PROJECT RESULTS

The experimental setup exploited the high flux available at beamline I13-2 at the Diamond Light Source, and used a single X-ray absorbing mask to enable for multimodal imaging based on the BT method [7].

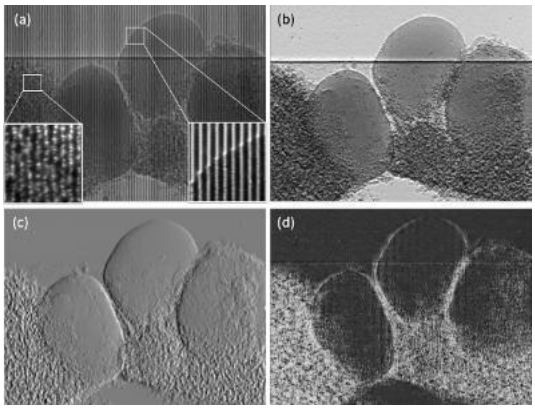

Fig. 1. Panel (a) shows an unprocessed frame image of a melting powder sample, with zooms on powder (left) and molten region (right). The attenuating lines of the x-ray mask are clearly visible. Panels (b) to (d) show the retrieved transmission, DPC and dark field contrast channels, respectively.

In our case, the pre-sample mask was scanned at a constant speed in the direction orthogonal to the mask lines, while the camera acquired a sequence of frames.

The aperture size is equal to the ultimate spatial resolution that can be achieved, and the ratio between aperture size and period (open fraction) determines the available flux, and therefore the temporal resolution, although in this experiment we were limited by motor speed and the available flux could not be fully exploited.

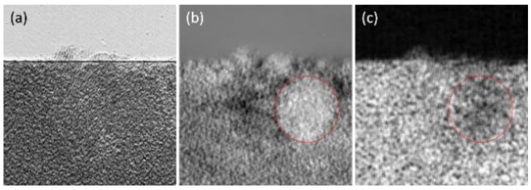

Fig. 2. Early stage of powder melting for each contrast channel. Panels (a) to (c) show the transmission, DPC and dark field, respectively.

While flux considerations would suggest a larger open fraction is preferable, there is a need to keep a reasonable separation between beamlets at the detector, to enable the reliable retrieval of the various contrast channels. After defining a reasonable parameter space through simulations, we designed and built a mask with various combinations of aperture size/periods, which were tested experimentally. 5/20 and 9/26 (aperture/pitch, both in micron) provided the best compromise between photon statistic, beamlet separation at the detector and time/spatial resolution. The mentioned motor limitations meant these arrangements provided temporal resolutions of 22 ms and 40 ms (respectively) at the maximum spatial resolution equal to the aperture size. Since both masks provided good images for all three signals, we focus here on the fastest, higher-resolution results obtained with the 5/20 mask. The first investigated sample was aluminium powder with an average grain size of 45 microns, melted by a laser with variable power, ranging from 20 W to 75 W. In Fig.1a, a pre-retrieval experimental image is shown. The different behaviour of the X-ray beamlets can be observed in the zoomed regions. Where a bubble has formed making the material homogeneous, the beamlets appear straight, as negligible refraction or scattering occur. A decrease in intensity compared to air can be seen, due to absorption. Conversely in the region where powder is unfused, large refraction/scatter occur and the shape of beamlets is significantly distorted. The assessment of variations in beamlet amplitude, centre, and width, with and without the sample, leads to the retrieved transmission, differential phase and DF images shown in Fig.(b) to (d). A large difference between un-melted and melted powder is observed, in particular in the DF image, where no signal is observed within the bubble demonstrating its homogeneity on the sub-micron scale. The separation of the three signals leads to interesting details being observed at the early stages of powder melting, as shown in Fig.2. Panel (b) shows the phase shift obtained by integrating the differential signal along the image rows. Phase provides higher sensitivity to fine density changes occurring during the melting process. In particular, the formation of a bubble is clearly visible in the phase image (see red circle) at a time when it is still invisible in the conventional transmission image of Fig.2(a). The formation of this bubble is also visible as a decrease in the scatter signal (red circle in Fig.2(c)). The average of a 5x5 region is shown to improve visibility.

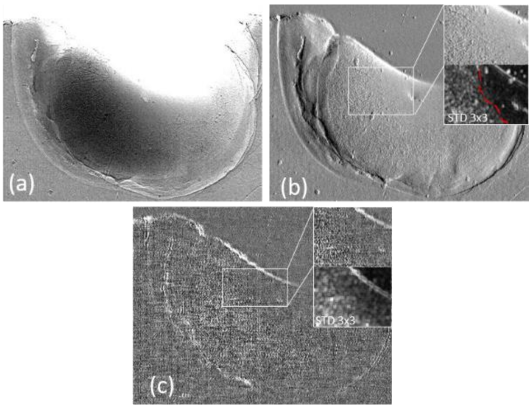

Fig. 3. Panels (a) to (c) show the transmission, DPC and dark field channels for a titanium melting experiment. The insets show a zoom in and the corresponding standard deviation in a 3x3 ROI

The second investigated process was the melting of a titanium slab in air using laser powers from 20W to 75W. Also in this case, the 5/20 micron mask was used, achieving the same spatial and temporal resolution as in the previous case. An example of the images for each contrast channel is shown in Fig.3. Panel (a) shows the transmission image. Panel (b) shows the differential phase, in which the presence of a “rough” region can be appreciated as shown in the inset, below which the standard deviation in a 3x3 region highlights the boundary between the rough and a homogenously flat region. We interpret this as the separation between the melted titanium pool (flat region) and solidified titanium oxide, characterized by micron and sub-micron size structures. The presence of sub-pixel resolution structures is confirmed by the signal in the DF channel, shown in Fig.3(e). This appears as a high-frequency intensity variation, well highlighted by the standard deviation reported in the inset.

5. FUTURE PROJECT VISION

One aspect of our vision is to develop the technology as an enabler for new synchrotron studies. Phase 1 has shown that the application of our technology to LAM provides access to information that was previously undetectable; systematic studies are needed to understand in detail the benefits its use can bring. The DF signal can be calibrated, including in a dynamic context [11], and quantitative links can be made between DF signal and the size of the sub-resolution objects causing it [12].

The expected impact extends beyond LAM and into materials science in general, especially where phase

transitions are concerned. Applications exist in energy, e.g. for resolving rapid phenomena in batteries [4], and in the life sciences, e.g. by exploiting the technique's ability to enable significant dose reductions [13]. We also envisage extending the technology from 2D to 3D.
The other aspect of our vision is the technology's implementations with laboratory sources. While the ultra-high flux element will be lost, some novel source technologies deliver fluxes comparable to 2nd generation synchrotrons [14], in some cases with comparable brilliance [15]. The BT technique does not require micro-focal sources; if mask designs based on long slits are used, there are practically no restrictions on focal spot size along one direction. Laser-plasma sources are progressing rapidly, targeting higher fluxes by increasing the repetition rate: this is a perfect match to our technology, since the acquisition of the dynamic frames could be synchronised with the laser pulses.

5.1. Technology Scaling

This involves both optimisation for synchrotron use and lab translation. The synchrotron aspect is split in planar and CT. Planar requires setup optimisation, tailored to the characteristics of a specific facility/beamline. Designs will build on a common "module", but employ different masks/motors to ensure full exploitation of the available flux. CT translation will be based on our "cycloidal CT" innovation, which enables high resolution multi-modal imaging with fewer projections [16], and be facilitated by the upgrades underway at many synchrotron facilities, leading to higher fluxes.

Lab translation will be based on our collaboration with companies developing advanced source technology [14,15]. We will optimise the design to enable strong multi-modal signals in the fastest possible timescale. As well as by BT working with extended focal spots, this is facilitated by its ability to allow for compact designs [17], which reduce the source-to-detector distance.

5.2. Project Synergies and Outreach

We will enlarge our consortium by including source specialists and imaging system manufacturers (see 5.4), plus additional synchrotron facilities (e.g. the post-upgrade ESRF is demonstrating outstanding performance) and end users. Synergies exist with our other ATTRACT projects ML-CYCLO-CT and UTXμCT; discussions will be held with consortia on detectors (X-COL, where we are partners, and FASTPIX/ESSENCE) and CT reconstruction (QuIT).
Public engagement (PE) will be a key element. Our entire team received training from the PE unit created when UCL was selected as a centrally funded "Beacon of PE", and we regularly engage with non-specialist audiences; this will be scaled up to guarantee appropriate dissemination of our ATTRACT results.

5.3. Technology application and demonstration cases

We will target at least three demonstrations in collaboration with end users:

- *Health*, through ultra-low dose dynamic lung imaging. Excellence exist at UCL (Rachel Chambers/EMINENT consortium) plus we have links with EU leaders (Sam Bayat, CHU).
- *Secure, clean and efficient energy*, through dynamic studies in batteries with our collaborator Prof Shearing who runs the world-leading Electrochemical Innovation Lab.
- *Smart transport/resource efficiency* by expanding our activity in AM.

EU Research Infrastructure will benefit through deployment of the technology at synchrotron facilities. We have established collaborations with Diamond, the ESRF and Elettra at least two of which will be our partners in Phase 2, enabling installation and thorough testing of the technology and thus facilitating ensuing deployment in other facilities.

5.4. Technology commercialization

We collaborate with several x-ray companies [18], which puts us in the ideal position to target the exploitation of our research, be it through agreements with a single company, or creation of a consortium. Established mechanisms exist for both, as do collaboration agreements which both de-risk and reduce the bureaucratical burden of any step in this direction.

5.5. Envisioned risks

Mitigation against technical risks comes from the programme structure itself: having multiple targets with increasing level of complexity (2D at synchrotrons, 2D with lab sources, 3D at synchrotrons etc) guarantees that even if the most ambitious goals are not achieved in full, the less ambitious ones would still provide significant benefit. The key non-technical risk is the covid-19 pandemic, mitigation against which will be based on work-packages that can be completed by local teams (e.g. of synchrotron beamline scientists) with remote communication with the co-investigators.

5.6. Liaison with Student Teams and Socio-Economic Study

All our Departments run popular MSc programmes. In Phase 2 we will offer 10 projects/year, both to our cohorts and to our partner universities [19]. We are setting up a Doctoral Training Programme through [20], and we will promote cohort-building activities across MSc and PhD students. We will involve students in the design of teaching/training material on our technology, which will be used for both demonstrations to other institutes and (in an appropriately modified version) public engagement events. Not only will we offer constant availability for interviews, technology impact references, case studies, but we will actively pursue opportunities to include our technology in any such activity.

6. ACKNOWLEDGEMENT

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