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Fundamentals and recent advances in X-ray micro computed tomography (microCT) applied on thermal-fluid dynamics and multiphase flows

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Abstract. X-ray computed tomography (CT) is a well-known technique nowadays, since its first practical application by Sir. G. Hounsfield (Nobel price for medicine 1979) has continually benefited from optimising improvements, especially in medical applications. Indeed, also application of CT in various engineering research fields provides fundamental informations on a wide range of applications, considering that the technique is not destructive, allowing 3D visualization without perturbation of the analysed material. Nowadays, it is technologically possible to design and realize an equipment that achieve a micrometric resolution and even improve the sensibility in revealing differences in materials having very radiotransparency, allowing i.e. to distinguish between different fluids (with different density) or states of matter (like with two-phase flows). At the University of Bergamo, a prototype of an X-ray microCT system was developed since 2008, so being fully operative from 2012, with specific customizations for investigations in thermal-fluid dynamics and multiphase flow researches. A technical session held at the UIT International Conference in L'Aquila (Italy), at which this paper is referring, has presented some microCT fundamentals, to allow the audience to gain basics to follow the "fil-rouge" that links all the instrumentation developments, till the recent applications. Hereinafter are reported some applications currently developed at Bergamo University at the X-ray computed micro-tomography laboratory.

1. X-ray computed micro-tomography (microCT)

The pervasive use of NDT (non-destructive technologies) in research and industrial applications are currently in their best development opportunities, especially the high resolution X-ray applications. A comprehensive recent review by T.J. Heindel [1] reports the applications on flow visualization and characterization of multiphase flows.

At the *Department of Engineering and Applied Science* of the University of Bergamo, in 2012, began a new research focused on the investigation of interfaces and multiphase systems at the microscale. A first prototype of an X-ray computed micro-tomography (microCT) was designed and built that overcomes the limitations of the very few currently available semi-commercial devices, generally unsuitable for laboratory researches, with low density materials and at micrometric scale.

Technically, the microCT is a sophisticated equipment that allows to obtain the digitized three-dimensional scan of an irradiated sample. In fact, it can be described as a non-destructive 3D microscope, whose spatial resolution is a few micrometers (i.e. consider that a human hair is circa 100 microns and a human red blood cell is almost 8 microns). The currently available prototype is fully

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operational at the laboratories of the University of Bergamo, and it is rather unique nationwide and competes with the few synchrotrons equipped in the world. The microCT setup is used, but not limited at, in research in the subject area of Technical Physics; its particularities have allowed to promote publications of scientific interest (here, in part, cited) and activates contracts for industrial research and technology transfer that have actually co-financed the construction. The research fields are multidisciplinary, so the technical session held at the UIT International Conference in L'Aquila (Italy), at which this paper is referring, reports only some of the fields of application among many others that are under development.

2. MicroCT applied to diagnostics of cavitation in fuel injection systems

Cavitation plays an important role in fuel injector systems as it alters the injector's internal flow structure, discharge coefficient and can contribute to injector damage and component wear. Increasing injection pressure and decreasing nozzle hole sizes and injection duration cause an acceleration of the fuel as it travels from the sac through the restricting orifice and can lead to rapid drop of the static pressure and as a consequence to local cavitation in the entrance of the nozzle hole. Cavitation has been extensively studied using transparent large scale [2] and real size injectors [3, 4]. Flows with cavitation can be extremely three-dimensional and requires experimental approaches capable of measuring such structures. Visible light is scattered at the vapor-liquid interface and so that X-ray diagnostics have been used over the past few years to obtain quantitative information of the void fraction [5, 6]. Radiography and fluorescence diagnostics are reported and provide time-averaged vapor distributions from raster-scanning approach. X-ray phase contrast technique allows higher temporal resolution in respect to both previous techniques but is still showing a projected two dimensional cavitation images. CT measurements are presented by Bauer et al. [6] using a clinical scanner and reported three-dimensional time-averaged void-fraction distribution in quasi-steady pipe flow with the limited resolution of clinical scanners. At the University of Bergamo, in collaboration with the International Institute for cavitation Research (ICR) for Prof. M. Gavaises CITY University London (UK), the microCT facilty is applied to cavitation diagnostics can deliver three-dimensional (time-averaged) information with noteworthy higher resolution. Figure 1 shows the time-averaged vapor distribution in high-resolution.

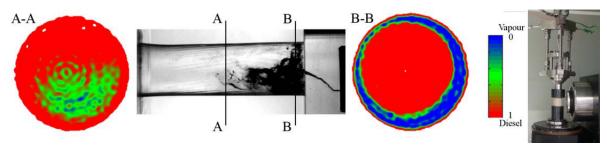


Figure 1. Vapor distribution within an Diesel injector at two different axial positions; the color represents the vapor fraction; blue corresponds to 100% vapor and red to 100% Diesel (liquid).

3. MicroCT applied to inspection and measurements of fuel injectors

MicroCT allows three-dimensionally profiling of the inner of complex injector parts and comparing this profile with nominal CAD geometries with a resolution of few microns. By doing this, CAD overlays tolerance bands could be generated and/or CAD overlays can be aligned with the inspected part to allow best fit analysis. Figure 2 show such comparison between nominal CAD drawings and fabricated new (not used) injector. Producer companies of microCT scanners often commercially offer such metrology analysis, and literature data are sparsely regarding fabrication tolerances of fuel injectors.

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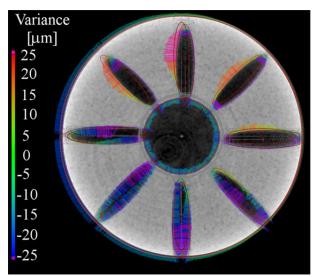


Figure 2. MicroCT volume of a multiple-hole injector with nominal CAD geometry (red lines) best-fitted on sac and resulting tolerance band.

Instead, several investigations on deposit formation and erosion in fuel injectors can be found due to two motivations: (a) continued legislative pressure to reduce exhaust emissions and as a consequence the development of advanced injection systems using higher temperatures and pressures at the injector tip, where deposit formation and erosion is initiated; and (b) the use of renewable and fuels with additives which are capable of providing good engine performance in short term engine operation but during long term operation degradation of engine performance, excessive carbon and lacquer deposits and actual damage to the engine were observed [7]. The formation of deposition and erosion within the holes of the injector affects the injection pattern and fuel flow rate and may have an adverse effect on the overall system performance [8]. Inspections of deposition were done with scanning electron microscopy, field emission scanning electron microscopy and energy dispersive X-ray spectroscopy [7, 9]. MicroCT allows a full three-dimensional inspection of such deposition layer and separation of this layer from the nozzle material is possible due to their differences in X-rays attenuation (see figure 3). Such a carbon layer after segmentation is shown figure 4 for the first time.

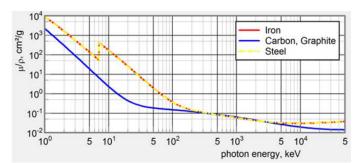


Figure 3. X-ray attenuation profile for carbon, steel and iron, a suitable energy range allows to reveal fouled surfaces.

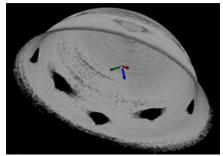


Figure 4. 3D carbon (fouled) deposition layer of injector tip after segmentation

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4. MicroCT applied to the estimation of phase transition rate of arbitrary shaped droplets

The experimental investigation on fluid evaporation or sublimation are generally complex, even more in highly deformed droplets, nevertheless available models needs to be improved in some extent. In general, the evaporation rate is a key parameter directly linked to the feasibility to measure the droplet volume variations (eventually in 3D) under non-stationary boundary conditions. The effect of particle deformation on phase transition was investigated by [10, 11]. These previous studies concluded that the transition rates (per unit area) are higher for deformed particles. The dependency of transition rate from the local curvature (an arbitrary shaped particle) can be studied starting from microCT investigations. 3D volume variation due to sublimation is presented here for the first time. Figure 5 shows the local change of the radius of a camphor sphere in 24 hours for different side views. These first results demonstrating microCT as a very promising technique for estimating phase change rates with adequate 3D accuracy and high sensitivity and accuracy. MicroCT allows studying any shaped geometry. Further, this technique can even used with imposed and oriented external convective flux, laminar or turbulent.

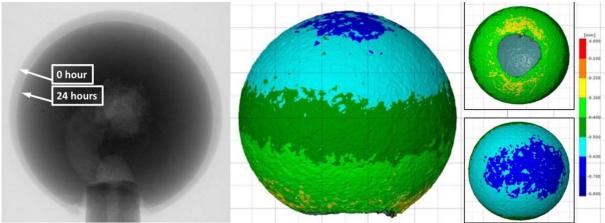


Figure 5. Sublimation regression in 24 hours of a camphor sphere. The resolution corresponds to a voxel of 7 microns, on the left 2 overlapped radiography, on the right the 3D contour plot of local ΔR .

5. MicroCT applied on wetting behaviour

Wetting of ideal surfaces is characterized by the equilibrium contact angle given by the well-known Young Equation [12]. Instead, wetting of rough surfaces is characterized by an apparent contact angle (APCA), which is defined as an equilibrium contact angle measured macroscopically [13], since the detailed microscopic topography cannot be viewed using classical optical methods. Therefore, APCA is the angle between the tangent to the liquid-vapor interface and the apparent solid surface. Two important models for describing the wettability behavior of rough surface are commonly used to explain the effect of roughness on the APCA: the Wenzel [14] model for homogeneous and the Cassie-Baxter [15] model for heterogeneous wetting. Further wetting states are reported by [16-19].

The APCA is governed by the wetting regime occurring in the vicinity of the triple line [14]; hence, visualization of this area is crucial and classical optical methods cannot be applied. Several advanced techniques were used for visualization the triple line such as reflection interference contrast microscopy [20], laser scanning confocal microscopy [21] or microCT [22-25] as shown in figure 6 and figure 7.

MicroCT allows analyzing the wetting behavior in terms of drop shape, wetting state, real and apparent wetted area; both enabling to estimate the Wenzel ratio, real and apparent contact angles, and the amount of drop liquid penetrating in texture fibers [22-25]. There, the drop and the substrate volumes can be extracted from the total scanned volume by means of volume segmentation on the basis of the X-ray attenuation of the different substances. The analysis of the drop and surface contact

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area gives the information about the contact angle applying of the axisymmetric drop shape analysis (ADSA) [26]. With respect to the commonly used ADSA; using microCT the drop/surface side pictures are replaced with real slices of the drop and substrate volumes in the chosen direction and at the chosen location [22-25]. Thus local information about both the real and the apparent contact angle along the triple line can be obtained, overcoming the limitation of the conventional optical approach based on side projections. For further discussion of applying microCT to wetting analysis, it is referred to [22,25].

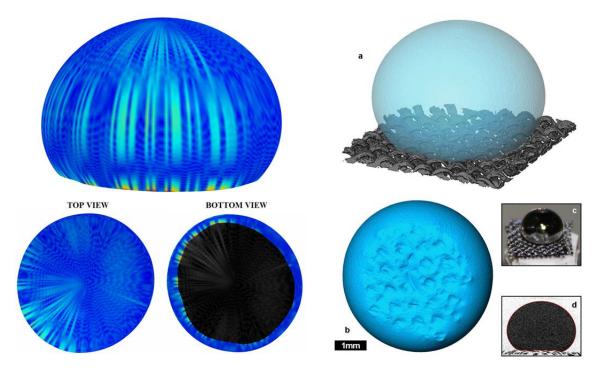


Figure 6. 3D orthographic, top and bottom view of the microCT droplet after the reconstruction based on the Laplace-Young fitting of 240 half slices of the droplet. Color is proportional to the dimensionless error $\Delta R/R_{max}$ between the Laplace-Young fitted surface and the original surface (maximum error 3.85%) [22].

Figure 7. MicroCT of complex artificial substrate and a sessile water drop: segmented and rendered volume [23].

6. MicroCT applied to biofilm morphologies

Power output limitation is one of the main concerns that need to be addressed for full-scale applications of the microbial fuel cell technology. Fouling and biofilm growth on the electrodes in single chamber microbial fuel cells (MCF), the most likely design of microbial fuel cells, affects their performance in long-term operation with wastewater. These performances losses are caused by the direct exposure of the electrodes to the solution enriched with the inorganic/organic substrate and bacteria which induces the growth of an electroactive biofilm [27-30].

Previously reported techniques for biofilm structural characterization include optical sectioning, fourier transform infrared spectroscopy analysis, and other microscopy methods, where the most challenging problem is to determine morphological features of thick biofilms (i.e., having a thickness of a few millimeters). Scanning electron microscopy (SEM) requires sample pretreatment, increases chances of artifacts and morphological distortions [31, 32]. The developed environmental SEM allows the imaging of the biofilm also in wet conditions reducing before mentioned problems [33]. But, this

technique is quite complex and requires a dedicated instrumentation with a special detector. Confocal laser scanning microscopy (CLSM) imaging is arguably the most common nondestructive method in use for imaging biofilms [31, 32]. However, CLSM cannot be used to image inorganic fouling layers such as the carbonate deposits commonly observed in MFC electrodes, as they are impenetrable to photons. Recently, microCT was successfully used for volumetric investigation of MFC electrodes without the above described limitations of other established techniques in this sector for the first time [34]. MicroCT was used to analyze both the surface and the deep layers of biofilms and inorganic fouling in their native state, without any pretreatment.

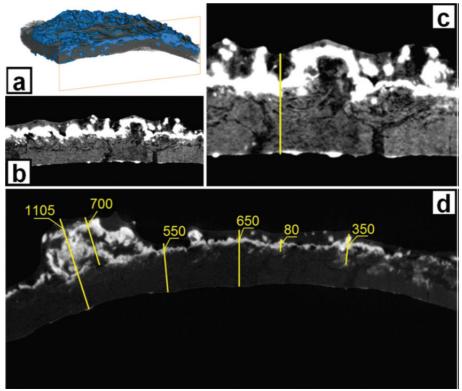


Figure 8. Orthogonal cross-section of the electrode sample extracted from the reconstructed microCT volume and post-processed to extract estimated thickness information (dimensions in micrometer); in (c) the line is equal to $650 \ \mu m \ [34]$.

In figure 8, such analysis is shown reporting also quantitative measure of distance (and volume) available due to proper calibration. Unveil details are reported like surface defects, density of the fouling layers, and the interface between biological and inorganic fouling, Finally, being a nondestructive method, microCT can be used *in-situ*, thus allowing experiments at different time on the same sample.

7. MicroCT applied to biological tissues: human heart valve disease visualizations in foetuses

The most frequent causes of fetal death are represented by chromosomal or genetic abnormalities, structural congenital malformations, and other vascular abnormalities. Currently in the prenatal period, the gold standard for clinical evaluation of fetal abnormalities is represented by ultrasound-based diagnoses. Numerous studies have shown that a nuchal scan during the first trimester screening performed by experienced operators can identify of major structural and cardiac abnormalities [35-39]. However in case of fetal death or miscarriage, the prenatal ultrasound information is unavailable in the majority of cases. Here, the traditional autopsy techniques provide only partial clinical responses,

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especially before 16 weeks of gestation due to the small size of fetal organs. Consequently in recent years, scientific research has focused on the study of alternative methods of investigation traditional postmortem autopsy, such as the use of magnetic resonance imaging (MRI) and post-mortem computed tomography (CT), which allow performing an virtual non-destructive autopsy and obtaining as result high resolution images of fetal organs in their entirety. The main limitations of these methods are represented by limited resolution. Recently, Lombardi et al. [40] presented a feasibility study on the use of microCT using a commercial system and this technique - also with quite low resolution - was able to more fully and accurately the presence and type of anatomic abnormality in comparison with the mentioned established methods.

Recently, the Author was authorized to start large post-mortem study in cooperation with Policlinico Milano, Clinica Mangiagalli on human foetuses with low gestational age (first and second trimester) affected by severe cardiac pathologies. The study is aimed to create the first 3D anatomical atlas for human heart foetuses for different gestational ages. Figure 9 shows the feasibility test on a murine heart (provided by Istituto di ricerche farmacologiche "Mario Negri", Dr. A. Remuzzi).



Figure 9. Visualization of a millimetric heart size from a reconstructed microCT volume, voxel 7 microns.

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