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NEW GENERATION X-RAY IMAGING
FOR MULTIPHASE SYSTEMS

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ABSTRACT

Pulsed X-ray imaging has been pivotal to develop knowledge on bubble formation, bubble coalescence and splitting, to enable the study of the division of gas between bubble and interstitial phases in fluidized beds of fine powders, as well as the effect of high temperature and pressure on gas fluidized beds. This paper provides a brief history on X-ray imaging applied to investigate bubbling fluidized beds and presents a technical description of the new high power pulsed X-ray facility recently installed at UCL. A new X-ray generation system has been recently developed to provide X-ray pulses down to 200µs width with an intensity of up to 450mA at a voltage variable from 50kV to 150kV. The new system enables to capture details with high resolution, visualizing objects smaller than 100 microns.

INTRODUCTION

Multiphase systems are employed in physical, chemical, pharmaceuticals, petrochemical, electrochemical, biological and energy applications. Fluidized bed reactors are core technologies of the chemical process industries and are recognized as a key technology to optimize the potential of generating clean energy from renewable resources. Multiphase flows are often extremely complex in nature and many of the relationships used to describe multiphase systems are of an essentially empirical nature, of limited applicability, and reflect the poor physical understanding of many two-phase flow phenomena.

Non-invasive experimental techniques are invaluable for providing a detailed insight into the flow patterns and general hydrodynamic characteristics of multiphase systems. One such technique involves the use of X-rays and is based on the same principles as are used in diagnostic medicine to record the structures of body parts that are opaque to visible light. X-rays can give quantitative real time information about the internal flow pattern of a rapidly changing three dimensional system, provided that a powerful enough X-ray source is used.

The first recorded use of X-rays to study gas-solid fluidized beds dates from the mid-1950’s and over the subsequent fifty or so years the technique has been applied to a wide range of two- and three-phase fluidized systems. The technique has enabled gas and solids flow patterns to be observed and quantified and has led to the improved design of internal structures such as gas distributors and heat transfer surfaces in industrial units.
A brief history of X-ray imaging applied to bubbling fluidized beds

Grohse (1) first reported the use of X-rays to determine the instantaneous and time-averaged bulk density of a finely-divided powder fluidized at a range of gas velocities and with three different designs of gas distributor, a perforated plate, a mesh screen and a porous plate. The X-ray source consisted of standard components of an X-ray diffraction unit with a tungsten-target X-ray tube. The detection equipment consisted of a modification of the rate meter in a standard X-ray detector fed by a scintillation probe. The rate meter response was logarithmic over most of its range of sensitivity and in accordance with Eq. (1) below the reading varied approximately linearly with the local bed density being measured.

\[ I = I_0 \exp(-\mu_m \rho l) \]  

Eq. (1)

where \( \mu_m \) is the mass attenuation coefficient of the bed material, \( \rho \) is its density and \( I \) is the bed thickness. In practice polychromatic radiation is normally employed but Eq. (1) is generally considered an adequate approximation. The term \( \mu_m \rho l \) is called the optical density of the absorbing material and for two different materials A and B producing identical X-ray attenuation the two optical densities are equal.

Romero and Smith (2) used flash X-ray radiography to study the internal structure of fluidized beds and obtained data not only on bed density distribution but also on the shape, size and velocity of gas bubbles in an air-fluidized bed of sand. Their unit consisted of two flash tubes one operating at 300 kV the other at 600 kV each producing a square pulse of energy at 1,000 amp for a duration of about 0.2 \( \mu \)s. The tubes were mounted on opposite walls of a lead-lined room which housed a 7.6 cm square plexiglass column containing a fluidized bed of sand. After passing through the bed the X-ray beam was recorded on 20 x 25 cm sheets of film the 300 kV tube illuminating the lower part of the bed and the 600 kV tube the upper part. To measure bubble velocity the two units were fired in sequence a few tenths of a second apart. To determine bed density Romero and Smith measured the darkness of their films using an optical densitometer but in order to compensate for variations in X-ray operating voltages and variations in the quality and processing of the films each photograph was compared with a standard consisting of sand-filled wedges made from plexiglass. These were mounted on the side of the fluidization column so that an X-ray photograph of them was obtained each time the X-ray was fired. The wedge gave a film darkness gradation that could be related to sand thickness and by comparing film darkness in the wedge with darkness in the bed the local bed density could be obtained.

Rowe and Everett (3, 4, 5) described the design and operation of a fluidized bed X-ray system in which the attenuated beam on passing through the bed was registered on a 0.22 m diameter Mullard image intensifier. The X-ray tube used was a Machlett Super Dynamax 125 with a rotating anode and a fine or broad focal spot of 1 mm or 2 mm diameter. It had a rating of 200 mA at 120 kV_p for \( 10^2 \) s on fine focus or 500 mA at the same voltage and duration on broad focus. The image intensifier augmented the beam intensity by a factor of 3x10^3 and was filmed by a 70 mm cine camera at a rate of 6 or 8 frames/s, the X-ray beam being synchronised with the camera and pulsed for 2.5 ms. A series of rectangular section fluidized beds were studied with thicknesses of from 1.4 cm to 29.5 cm. Bubble sizes, their number and
velocity were measured at various heights up to 70 cm in beds of alumina, carbon, quartz, glass ballotini and crushed glass powder fluidized by ambient air.

A similar system to that of Rowe and Everett (3, 4, 5) was used by Rowe et al. (6) to make detailed measurements of the emulsion-phase voidage in fluidized beds and its variation with the particle size distribution of the bed material. As described above for Romero and Smith (2) a calibrating wedge filled with bed material was used to quantify the observations. By comparing the optical density of a region on the film with that at a given level of the wedge of known dimensions the powder voidage in that region could be determined from:

\[ \varepsilon_{e} = 1 - (1 - \varepsilon_{w})(\Delta W/\Delta B) \]  

(2)

where \( \varepsilon_{e} \) and \( \varepsilon_{w} \) are the powder voidages in the emulsion phase and the wedge respectively, \( \Delta W \) is the wedge thickness with the same optical density as the bed, and \( \Delta B \) is the bed thickness.

Since the '70s, using X-rays, a great deal of fundamental and applied research has been undertaken at UCL to study the highly complex flow phenomena and fluid-particle interactions involved in dense multiphase systems and how the operating conditions influence the reactor performance, efficiency and scale-up. Hoffman and Yates (7) used X-ray images to study the effect of pressure on fluidized beds of powders in Groups A and B of the Geldart classification (8). Other X-ray studies of the effects of system pressure on fluidization were reported by Rowe et al. (9) and Barreto et al. (10).

More recently, X-rays were used to investigate the influence of high temperature on the fluidization behaviour of fresh and doped industrial catalysts (11, 12). This study was subsequently extended to investigate the combined effect of high temperature and adding different fines cuts on the fluidization behaviour of alumina powders (13, 14).

The X-ray facility has also enabled interdisciplinary research to investigate the fluidization of pyroclastic flows materials at high temperature. In this study, X-rays have been used for the first time to elucidate the fluid-mechanics governing the behaviour of dense pyroclastic flows generated during explosive volcanic eruptions (15, 16) and to explain their ability to maintain high gas pore pressure, and hence low friction, during run-out.

X-ray imaging has also been applied to study circulating fluidized beds and jet penetration, as described in detail by Yates et al. (17), and also to support the development of new industrial processes, as briefly explained by Newton (18).

**How does the technique work?**

In the X-ray technique, a high energy beam (from 40 to 150 kV) is produced from a rotating anode. The X-ray pulses are synchronised with an image capturing device and pass through the reactor vessel. When X-rays pass through a solid material they are attenuated by processes of absorption, reflection and scattering and the extent of the attenuation is a function of the chemical nature of the solid and of the
quantity of material in the path of the beam. The x-ray beam emerging from the vessel is amplified using an image intensifier. This converts the x-ray absorption pattern into a light image of sufficient brightness and contrast to be recorded on a camera synchronized with the x-ray generator. A detailed description of the x-ray image analysis and calibration technique is reported by Yates et al. (17). Figure 1 shows a sketch of the X-ray imaging technique.

A new generation high power pulsed X-ray imaging system at UCL

A new high power pulsed X-ray generation system has been recently developed at UCL to provide X-ray pulses down to 200µs width with an intensity of up to 450mA at a voltage variable from 50kV to 150kV. This specially designed generation system is coupled to a specialist rotating anode x-ray tube has a capacity of up to 1 million heat units and 68kW peak loading. This heat capacity allows for sustained x-ray pulse streams of several minutes. The microprocessor controller ensures that these limits are not exceeded. Two focal spots are selectable (0.6mm and 1.2mm) and the target can be rotated at either 50/60Hz or 180Hz, depending on the power required. X-rays are detected on the latest generation 30cm Industrial X-ray Image Intensifier, optically coupled to a 1024 x 1024 pixel high speed digital CCD camera. The camera is triggered by the control software, which itself is triggered by the x-ray generator at frame rates from 24 to 72 frames per second (fps). The generator has been designed to operate up to 500 fps, when coupled with an equally high speed camera. 8-bit digital images are then sent optically to a powerful workstation with dual Xeon® processors and a RAID disc array store. Both the x-ray tube and x-ray detector have motorised x-ray masking to ensure the best quality images with the minimum of scatter. The new system enables to capture details with high resolution, visualizing objects smaller than 100 microns.

The X-ray equipment is housed in a radiation proof room which contains the high power X-ray generator see Fig. (2a), x-ray tube, see (2b), and an image intensifier with motorized shutters, see (2c). The X-ray tube and image intensifier are mounted on a twin column ceiling suspension unit, which allows the columns to be moved along the length of the room. The lateral movement of each column permits the
distance between the tube and image intensifier to be changed. Each of the columns can also be moved in a vertical plane either independently or synchronized as a pair. This motion is motorized and can be remotely controlled from outside the room.

Figure 2. (a) X-ray generator; (b) whole system including x-ray tube (left hand side), image intensifier and camera (right hand side); c) x-ray detector on manipulator arm.

The new system has just been recently installed and tested; preliminary images are presented in this paper to show the enhanced resolution of the new system. Figure 3 shows a comparison between (3a) the original X-ray image of a bubbling bed of glass ballottini taken by Rowe et al. (3) and (3b) the enhanced resolution obtained with the new system installed. In Figure 3b, bubble coalescence and splitting can be clearly observed, where the particles raining down through the bubble can also be clearly seen.

Figure 3. X-ray image of a bubbling fluidized bed: (a) original image (3); (b) image with the new X-ray system at UCL.

The high resolution of the new system was tested during a project in collaboration with UCL Biomedical Physics, where x-ray images of a set of 29 electrodes implanted in the skull of a rat were taken to visualize the geometry of the implantation. Each electrode was 500µm in diameter and positioned at about 200µm
distance between each other. Figure 4 clearly shows the array of electrodes implanted, indicating a resolution of at least 100µm.

![X-ray image of electrodes' array implanted in a rat's skull](image)

**Figure 4. X-ray image of electrodes’ array implanted in a rat’s skull**

In the new system, images are captured, displayed and stored using a custom version of the SPS ‘iX-Control’ software. The software handles acquisition, image processing/corrections, image display, image storage and playback up to 72 fps in either real-time or frame by frame. An extensive set of analysis tools allows for offline data analysis and for measurements to be made. Figure 5 shows as an example the x-ray image of a stable plume obtained by injecting gas through a single orifice nozzle; here the contrast has been adjusted in order to visualize clearly the solid distribution within the plum and at the boundary in order to quantify the solid mass transfer between the plum and the surrounding bed of particles.

The new X-ray facility is a central part of an on-going research programme with Advanced Plasma Power to enable the study of the interaction between gas-emitting fuel particles and freely bubbling beds during gasification of waste, and obtain fundamental data needed for the model validation and to assess the reactor performance. In parallel, a project in collaboration with NNL is on-going to investigate the flow behaviour in a scale-down TDN reactor. X-rays are complementary to other techniques such as PEPT (Positron Emission Particle Tracking) and MRI (Magnetic Resonance Imaging). A project is currently on-going in which all three techniques (X-rays, MRI and PEPT) are employed to investigate the behaviour of single and multiple jets at the distributor in a 50 mm and 150 mm diameter fluidized bed containing Group B poppy seeds having a particle diameter of 0.5 mm. Preliminary results on the lengths of the jets with a single orifice distributor plate using both X-ray and MRI were obtained via measurements of the voidage in the fluidized bed. Good agreement between these two techniques was obtained, confirming the accuracy of these advanced imaging modalities. The high temporal and spatial resolution of the X-ray apparatus was used to investigate the frequency of bubble detachment in the fluidized bed; whereas the capability of MRI to measure velocities directly was used to probe particle velocity fields in the same system. By combining X-rays with these complementary techniques, a complete
picture of the fluid dynamics and particle movement of complex multiphase systems can be provided, allowing new and exciting research in multiphase flows to be undertaken.

![Image of fluid dynamics and particle movement](image)

**Figure 5. ‘iX-Control’ software displaying an optimised, stable plume from test nozzle**

**CONCLUSIONS**

The X-ray technique is one of an increasing number of non-invasive methods currently being employed to study multiphase systems of industrial interest. The above examples demonstrate the versatility of the technique for real-time observation and analysis of the many hydrodynamic features of fluidized beds and it will continue to be used for this purpose. Combining pulsed X-rays with complementary techniques such as PEPT, MRI or ECT, makes it a powerful tool to study complex multiphase systems. Furthermore, the high degree of discrimination possible with X-rays makes it ideal for the much-needed validation of theoretical models for the CFD modelling of complex multiphase systems; the progress made in this direction is expected to be invaluable in future.

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**NOTATION**

- \( I \) transmitted intensity
- \( I_0 \) incident intensity
- \( l \) length (m)
\[ \Delta B \]  bed thickness  m
\[ \Delta W \]  wedge thickness  m
\[ \varepsilon_e \]  powder voidage in the emulsion phase
\[ \varepsilon_w \]  powder voidage in the wedge
\[ \mu_{m} \]  mass attenuation coefficient  m\(^{-1}\)
\[ \rho \]  density  kg m\(^{-3}\)

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