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## **DEVELOPING TRACER PARTICLES FOR X-RAY PARTICLE TRACKING VELOCIMETRY**

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### **ABSTRACT**

X-ray imaging, as a noninvasive flow visualization technique, has been shown to be a useful method for observing and characterizing multiphase flows. One type of X-ray flow visualization technique, called X-ray Particle Tracking Velocimetry (XPTV), tracks an X-ray attenuating particle in an opaque fluid flow. A significant challenge with XPTV is identifying tracer particles with the desired fluid flow characteristics (e.g., small and neutrally buoyant) but yet differentially attenuate X-rays, which is based primarily on density differences. This paper describes the manufacturing of XPTV tracer particles that satisfy specific particle characteristics including high X-ray attenuation, uniform shape, specified effective density, and desired diameter. An example use of these particles as an intruder particle in a fluidized bed (to simulate biomass injection) is then demonstrated using X-ray stereographic imaging to determine intruder particle position as a function of time in a three-dimensional opaque system.

**Keywords:** imaging, particle tracking, tracer particles, X-ray flow visualization

### **INTRODUCTION**

Multiphase flows, composed of gas-liquid, gas-solid, liquid-solid, or gas-liquid-solid mixtures, are commonly found in many process industries such as petroleum-based fuel production, energy generation, commodity and specialty chemical production, mineral processing, textile processing, pulp and paper processing, wastewater treatment, food processing, and biological organism and pharmaceutical

production. Although the uses of these systems are extensive, their operation is very complex and an improved understanding of the fundamental hydrodynamic and transport processes are necessary to develop process improvements and optimization, as well as to develop and validate fundamental models of their operation.

The principle difficulty in characterizing and quantifying multiphase flows is the fact that the systems are typically opaque; even an air-water system becomes opaque at fairly low volumetric gas fractions. This necessitates either the use of invasive measurement probes when determining internal flow and transport characteristics or noninvasive (nondestructive) methods. The challenge with invasive probes is that they can alter the internal flow of the multiphase system interfering with realistic process measurements. Noninvasive measurement techniques for multiphase flows circumvents this issue.

Multiple noninvasive flow visualization techniques have been reviewed in the literature [1-4]. Some of these methods involve particle tracking, but when the system has limited optical access, additional complications arise. Noninvasive techniques such as X-ray absorption,  $\gamma$ -ray emission, or positron emission methods utilize the ability to penetrate and/or pass radiation through dense and/or opaque materials, alleviating the problems with optical access. Though similar to visible light, these techniques have the ability to visualize flow patterns through the opaque materials found in many multiphase flows. Specific particle tracking methods for opaque fluid flows that have appeared in the literature include radioactive emitting tracer particles [5-8], X-ray absorbing particles [9-12], or neutron absorbing particles [13].

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X-ray Particle Tracking Velocimetry (XPTV) is an X-ray imaging technique where several X-ray absorbing objects (particles) are tracked simultaneously as a function of time. As stated by Seeger et al. [14], the advantages of XPTV are many and include: (i) can track velocities in opaque flows or in flows with high gas fractions, (ii) can measure many points simultaneously, (iii) can record 3D velocity components, and (iv) is noninvasive. The disadvantages include (i) low image frequency, (ii) large tracer particles are typically required, and (iii) the method utilizes X-rays. A significant challenge with XPTV is identifying tracer particles with the desired fluid flow characteristics (e.g., neutrally buoyant) but yet differentially attenuate X-rays, which is based primarily on density differences.

By tracking neutrally buoyant X-ray absorbing particles, Seeger and co-workers [9, 10, 15] were able to record the three-dimensional liquid velocity field in a slurry bubble column. The solid particles were polymethylmethacrylate cubes with dimensions of 2 mm on a side. Similar cubic X-ray tracer particles were fabricated from polyurethane foam with tin alloy cylindrical inserts. In this case, the tin alloy absorbed the X-ray energy and the foam provided buoyancy to produce manufactured particles with an effective density and dimensions similar to the bulk solid material. Seeger et al. [10] concluded this method can simultaneously provide 3D velocity fields and solid holdup but, as designed, was limited. They further concluded that improvements could be made with smaller X-ray absorbing seeding particles and faster camera and image intensifier pairs. Kertzscher et al. [15] improved this system to track intruder particles in a gas-liquid system. Improvements included better mapping between the two image intensifiers in terms of an isocenter correction and matching of whole particle trajectories.

Owens et al. [16] also manufactured neutrally buoyant, X-ray absorbing particles to track fluid flow with a focus on packed columns. In this case, they used a 2.88 mm diameter high-density polyurethane foam cylinder 2.88 mm in height with a 0.81 mm diameter lead solder core as tracking particles. They then used a single X-ray source-detector pair to track injected particles as they traveled through a packed bed. Their biggest challenge was identifying the particles in the packed bed because the particles were moving too fast relative to the frame rate of the recording device (30 frames/s).

Shimada et al. [17] followed the techniques of Seeger and co-workers [9, 10, 15] to develop an XPTV technique for the visualization and characterization of highly viscous opaque slurry flows. The application Shimada et al. [17] was addressing was the casting of solid propellants. By using 1 mm diameter lead balls as tracers, they were able to develop velocity maps in the highly viscous slurry flows.

Lee and Kim [11] utilized a coherent X-ray source from the synchrotron radiation source at the Pohang Accelerator Laboratory (PAL; Pohang, Korea) to develop X-ray particle image velocimetry (PIV) for opaque fluids that exploit phase contrast imaging. With this technique, they were able to track 3

$\mu\text{m}$  alumina microspheres in a 750  $\mu\text{m}$  diameter opaque Teflon tube and reproduce the expected velocity profile. Kim and Lee [18] extended this technique to produce a highly coherent X-ray beam; the resulting beam induced a classic Fresnel edge diffraction in the radiographs which was used to identify the edge of particles in the opaque fluid.

X-ray phase-contrast imaging was also used by Im et al. [19] for particle tracking in polydispersed particle-laden flows in optically opaque systems. The authors used a monochromatic X-ray beam from the Advanced Proton Source (APS) at Argonne National Laboratory to probe a glycerin flow in a 860  $\mu\text{m}$  ID Teflon tube seeded with 10  $\mu\text{m}$  diameter silver-coated hollow glass spheres. They were able to measure the velocity profile in the opaque tube using this technique.

Kakimoto et al. [20] used a single X-ray source and image intensifier to visualize tracer particle movement in molten silicon. Their challenge was in creating a tracer particle that was thermally and chemically stable and allowed the observation of convection patterns in the molten silicon. They determined that a tungsten particle encased in silica ( $\text{SiO}_2$ ) and then coated with carbon was the best tracer particle. The effective tracer particle density was close to that of molten silicon and the carbon coating provided good wettability in the molten silicon. A similar process was used by Munakata and Tanasawa [21] to visualize silicon melt convection under radio frequency heating. The authors used 0.5 mm diameter zirconia particles covered with a 0.25 mm layer of quartz glass, and then coated with carbon to avoid wettability problems of the quartz and molten silicon.

X-ray stereography imaging was used by Morgan and Heindel [22, 23] to visualize the Brazil nut effect, where a dense object migrates to the top of a bed of granular media when exposed to vibration. One of the dense objects in this study was a wood sphere saturated with potassium iodide to enhance the X-ray absorption characteristics of the particle.

Tracking intruder particles in a fluidized bed using X-ray stereographic imaging has been completed by Drake et al. [12, 24]. They showed that they can successfully track large intruder particles ( $\sim 9$  mm in diameter) in a fluidized bed composed of 500-600  $\mu\text{m}$  glass beads. Drake and Heindel [12] summarized potential system improvements to enhance the tracking abilities, including enhanced image analysis techniques, better intruder particle manufacturing capabilities, and faster camera systems.

This paper focuses on the manufacturing process to fabricate particles for X-ray particle tracking velocimetry that satisfy specific particle characteristics including high X-ray attenuation, uniform shape, specified effective density, and desired diameter. The X-ray system for XPTV imaging will first be briefly discussed. Particle manufacturing for XPTV will then be outlined. Some of the manufacturing challenges will then be summarized. Tracking sample particles in a fluidized bed will then be demonstrated. Finally, general conclusions of this work will be presented.

## EXPERIMENTAL PROCEDURES

### X-ray Visualization System for XPTV

The X-ray flow visualization facility at Iowa State University has been described in detail elsewhere [25-27]; only a summary is described here. The X-ray system consists of two LORAD LPX200 X-ray sources mounted at 90 degrees to each other as shown in Figure 1. Across from each source is a Precise Optics PS164X image intensifier and a DVC-1412 Monochrome Digital Camera with a resolution of 1388(H)  $\times$  1024(V). However, to balance temporal and spatial resolution trade-offs, the cameras are typically set for  $2 \times 2$  binning to obtain a maximum theoretical frame rate of 20 frames per second (fps), lowering the resolution to 640(H)  $\times$  512(V). The cameras are connected to a dual Intel Xeon quad-core with a processor speed of 2.66 GHz per core and 16 GB of RAM; the camera drivers limit the available RAM to 3.8 GB while imaging is in progress. Custom software is used to trigger the DVC cameras every 55 ms. This provides a slightly lower frame rate (18 frames per second) than the theoretical maximum, but also generates a more consistent frame rate. The exact time at which each frame is acquired is also recorded, accurate to 1 ms, to account for slight variations that might occur.

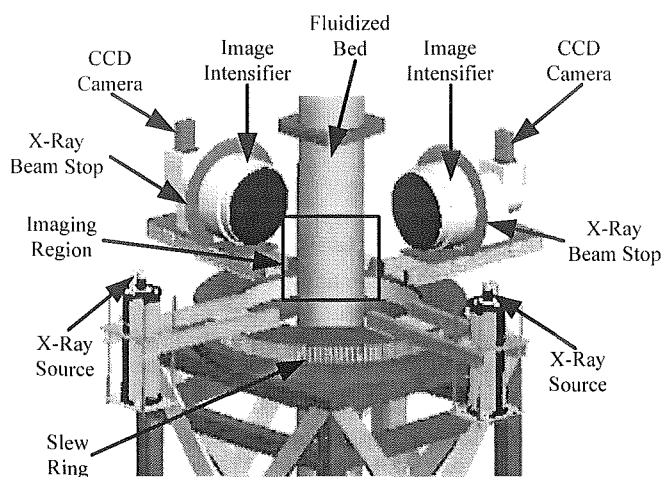


FIGURE 1: SCHEMATIC OF THE IOWA STATE X-RAY FLOW VISUALIZATION FACILITY.

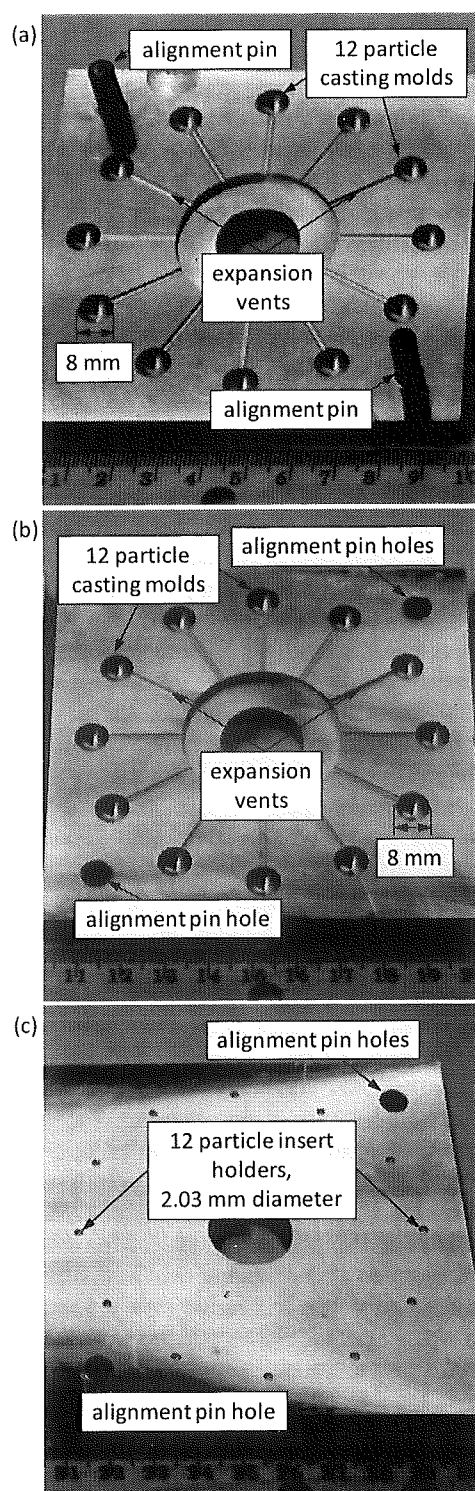
The images acquired directly from the camera are sequentially numbered 12-bit per pixel grayscale images. The same software that is used to acquire the images is also used to convert them from the native 12-bit format of the cameras (a format which is incompatible with most computers) to a standardized 16-bit per pixel grayscale format. The software also applies a correction for the pincushion distortion caused by the image intensifiers. A particle tracking algorithm can also be employed to track the location of the particle.

### XPTV Particle Manufacturing

The tracer particle manufacturing process outlined in this paper is designed to fabricate tracer particles with specified criteria, including a specific effective tracer particle density, diameter, and shape, while still being opaque to X-rays. In this example, the effective density chosen for the tracer particle is similar to softwood biomass, such as poplar, which has an approximate density of 0.3-0.9 g/cm<sup>3</sup>. A diameter of 0.8-0.9 cm is used for the composite particle due to the constraints of the multiphase flow system (fluidized bed) into which the particle is injected, and the desire to simulate a large biomass particle injection. In order to make the particle opaque to X-rays, a composite particle is manufactured in which one component is a high density material (e.g., lead). By combining the high density material with a much larger volume of low density material, the desired effective density can be tailored for the specific conditions. The low density material used in this work was a two-part closed cell polyurethane expandable foam (US Composites) "spiked" with bismuth powder.

The manufacturing of the tracer particles with the desired characteristics was accomplished through a three-part particle casting process in which three individual molds were fabricated. If it is desired to produce tracer particles with different characteristics (e.g., a different effective density, particle size, or lead insert shape), new molds would have to be machined, but the particle manufacturing process should remain the same.

The first part of the particle mold is composed of an aluminum block with twelve milled 8 mm diameter hemispheres arranged in a circular pattern (Figure 2a). From each hemisphere, a channel is milled to a central location to allow for the expansion of the two-part closed cell polyurethane foam that is used as the low density material. Two holes for alignment pins are also drilled in the aluminum block. The second part of the mold is identical to the first (Figure 2b). The third part of the mold is an aluminum block with twelve 2.03 mm diameter dimples in a circular pattern in which the lead insert is placed (Figure 2c); this part is used to make certain the lead insert is placed in the center of the low density expandable foam. Note that it is ideal to have the lead insert located in the center of the foam so when it is tracked with X-rays, the center of the effective tracer particle can be determined. However, if other attributes are desired, like particle orientation, they can be designed into the manufactured particle. Each mold also has a large hole drilled in the center to allow air to circulate and the foam to freely expand and cure properly.



**FIGURE 2: PICTURE OF THE CASTS USED TO MANUFACTURE TRACER PARTICLES.**

The procedure to manufacture X-ray tracer particles is a multiple step process. Through trial-and-error, the following steps were developed:

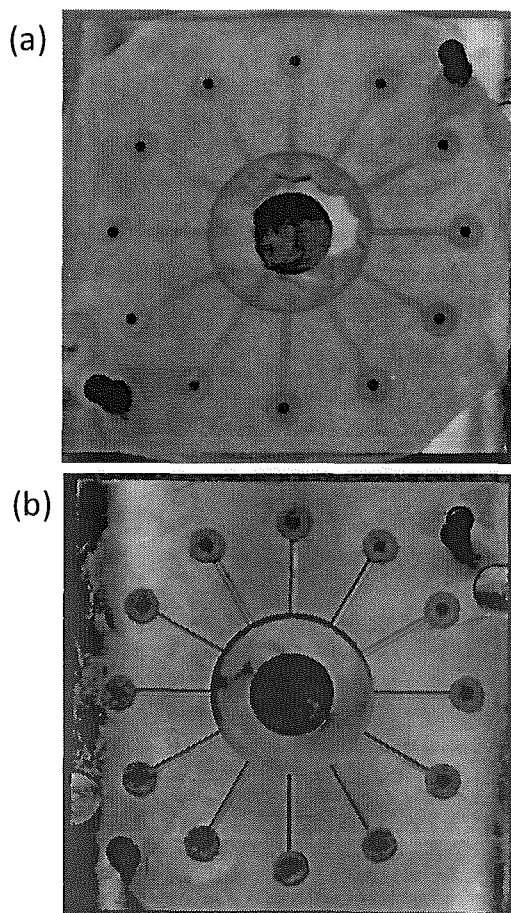
1. Before initiating the casting process, each cast must be sprayed with Ease Release 200 to allow the tracer particles to be easily removed from the cast.
2. Place the lead inserts into the mold dimples (Figure 2c).
3. Mix 1.03 g of part A and 1.44 g of part B of the two-part expandable foam with 0.15 g of bismuth and immediately pour into each hemisphere of mold piece containing the alignment pins (Figure 2a).
4. Clamp the cast with the foam and the cast with the lead inserts together, making sure the lead insert is on the bottom and laying horizontal. Allow the foam to set and cure for approximately 30 minutes.
5. When the set time is complete, carefully separate the two mold pieces and remove the excess foam from around the particle halves with an exact-o knife (see Figure 3).
6. Repeat steps 3-4 using the unused hemisphere mold (Figure 2b) and the completed hemispheres (Figure 3b).
7. When the set time is complete, separate the two pieces and again remove excess foam from around the particle.
8. Carefully remove each particle from the mold and apply a thin layer of fingernail polish to produce a hardened outer shell.

A completed tracer particle and hemisphere with lead insert is shown in Figure 4.

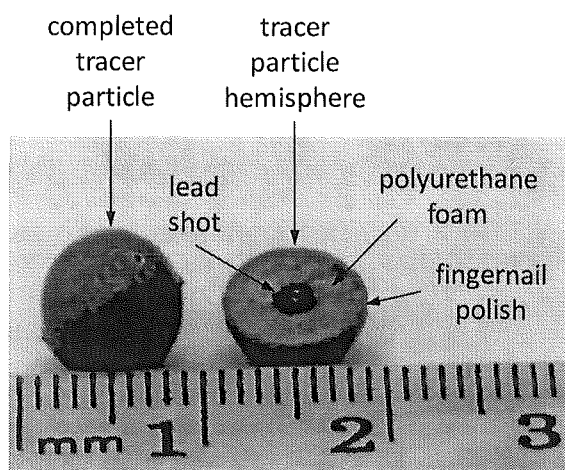
## RESULTS AND DISCUSSION

### Initial Particle Fabrication Challenges

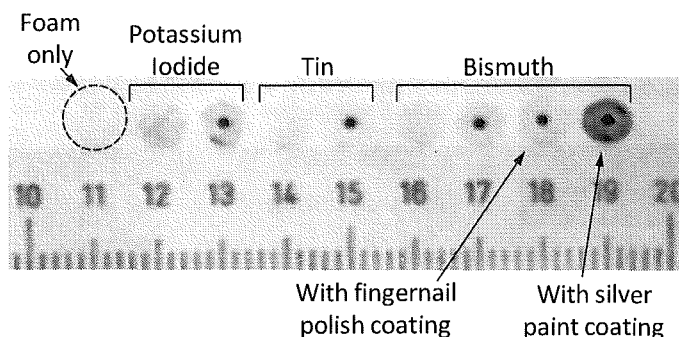
Several different foam additives were explored to enhance the uniform X-ray absorption in the foam region. Figure 5 shows various tracer particle candidates containing a range of powdered metals. The tracer particle on the far left is a foam-only particle and is almost transparent to X-rays because of the low attenuation characteristics of the foam. The next four particle images are two pairs and each pair has one particle with and without a lead insert. The first pair is foam loaded with potassium iodide salt, while the other is foam loaded with tin powder. The last four tracer particle images are foam loaded with bismuth powder. Again, there is one with and without the lead shot insert, along with one coated with fingernail polish and the last coated with silver paint. Though the silver paint attenuates X-rays very well, it has an acetone base which erodes the foam surface, so it is not usable.



**FIGURE 3: EXPANDED FOAM HEMISPHERES (A) BEFORE EXCESS FOAM IS REMOVED AND (B) AFTER EXCESS FOAM IS REMOVED.**



**FIGURE 4: PICTURE OF THE COMPOSITE TRACER PARTICLE.**

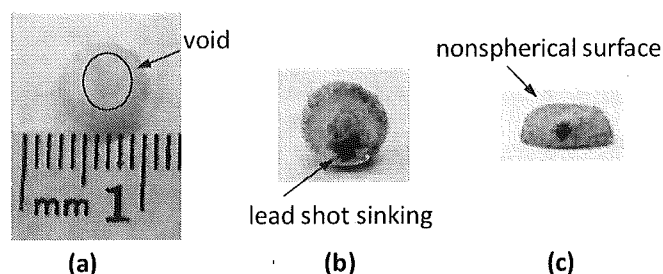


**FIGURE 5: X-RAY IMAGES OF POSSIBLE TRACER PARTICLE MIXTURE CANDIDATES, WITH AND WITHOUT LEAD INSERTS.**

Loading the two-part expandable foam with bismuth powder was chosen for the manufacturing of the tracer particles because bismuth is the heavier of the metals, which allowed the ability to increase the effective tracer particle mass in order to achieve the desired effective density. It also increases the X-ray absorption of the foam region. The two-part polyurethane foam mixture ratio was 40/60 by volume for parts A and B, respectively. To manufacture each half of a tracer particle, 1.03 g of part A and 1.44 g of part B was combined with 0.15 g of bismuth powder. The final mass of each manufactured tracer particle was 0.11 g with a volume of 0.2 cm<sup>3</sup>; which produced an effective density of 0.55 g/cm<sup>3</sup>, simulating a woody biomass particle.

Several challenges were also encountered when developing the tracer particle manufacturing process. One problem was eliminating any air bubbles in the casted foam (see, for example, Figure 6a). It was determined that by adding the bismuth powder, which provided enhanced X-ray absorption in the foam region, it prevented relatively large bubbles from forming during the foam expansion and curing process. The powdered bismuth was also found to help with the expansion of the foam which was determined by several trials in which the foam and loading concentrations were varied. The advertised expansion rate for the two-part polyurethane foam was approximately 25 to 30 times the liquid volume. Originally, the highest expansion rate was approximately 13 times the liquid volume. However, with a bismuth-loaded foam, an expansion rate of approximately 17 times the liquid volume was obtained. The foam may not be producing the advertised expansion rate due to the relatively small amounts of foam used in each casting.





**FIGURE 6: CASTING CHALLENGES CAN CREATE PARTICLES WITH (A) VOIDS ON THE SURFACE DUE TO LARGE AIR BUBBLES, (B) THE LEAD SHOT BEING DISPLACED FROM THE TRACER PARTICLE CENTER, OR (C) A NON-SPHERICAL PARTICLE SURFACE.**

Another problem encountered was the challenge in fabricating a homogenous foam region. If the powdered bismuth was not mixed thoroughly, it would sink to the bottom making the mixture non-uniform. To enhance the mixture uniformity, it was determined that the bismuth should first be combined with part A of the foam before adding part B, all the while continuously mixing the solution. Another problem was positioning the cast so the lead shot would set properly in the center of the tracer particle. If the initial hemisphere mold was not set such that the lead particles were on the bottom, the lead could dislodge from the holder and be displaced from the tracer particle center (Figure 6b). Finally, if the casted particles were removed from the mold before being completely cured, the particle's spherical shape may be distorted (Figure 6c).

#### **Sample Particle Tracking in a Fluidized Bed**

Fluidized beds are found in many industrial applications, including chemical and fuel production, power generation, mineral and powder processing, and pharmaceutical production. Their operation is based on a gas or liquid moving through a granular bed at a sufficient velocity to suspend the particles. They are useful because they have good mixing, low pressure drop, and high heat and mass transfer rates. Gas-solid fluidized beds have been studied extensively, but they are difficult to visualize because the systems are opaque due to the dense particle-laden flow, limiting the choice of experimental techniques. One type of fluidized bed operation involves the gasification of injected biomass particles. X-ray particle tracking is an excellent experimental technique to track the simulation of particle injection into a fluidized bed [12, 24].

The tracer particles developed in this study were used as simulated biomass particles and tracked in an operating 15.2 cm diameter fluidized bed filled with 500-600  $\mu\text{m}$  glass beads. The single particle injection system of Drake et al. [28] was used with a gas injection flow rate of  $0.1Q_{mf}$ , where  $Q_{mf}$  is the minimum fluidization volumetric flow rate. The superficial gas velocity was specified at  $1.5U_{mf}$ , where  $U_{mf}$  is the superficial gas velocity at minimum fluidization conditions (i.e.,  $Q_{mf}/A_c$ , with  $A_c$  the cross-sectional area of the fluidized bed). Figure 7 shows five sequential stereographic images of a single tracer

particle in an operating fluidized bed. The time difference between each image pair is 55 ms. The white circles identify the tracer particle location within the fluidized bed and is tracked from frame-to-frame using custom software [23]. As shown in Figure 7, it is difficult to identify the tracer particle in each image when presented in the paper because the fluidized bed is 15.2 cm in diameter and the lead tracer, which would be dark in the image, is only  $\sim 2$  mm. However, assembling the sequential image pairs into a movie allows the human eye to easily track the intruder particle location.

Using custom software developed by Morgan and Heindel [23], particle position can be identified, a sample of which is shown in Figure 8. Time = 0 corresponds to the time when the injected particle first enters the fluidized bed through the injection port. In this example, the injected particle rises to the surface of the fluidized bed almost immediately and then bounces around the top of the bed due to large bubbles erupting from the bed interior. At  $\sim 23$  secs, the tracer particle begins to be entrained in the fluidized bed and slowly falls through the bed. The tracer particle is eventually abruptly pushed to the bed surface ( $t \sim 30$  secs) and then bounces on the surface again. Similar x- and y-location plots as a function of time can be generated, as well as 2D location projections (e.g., x-y plots).

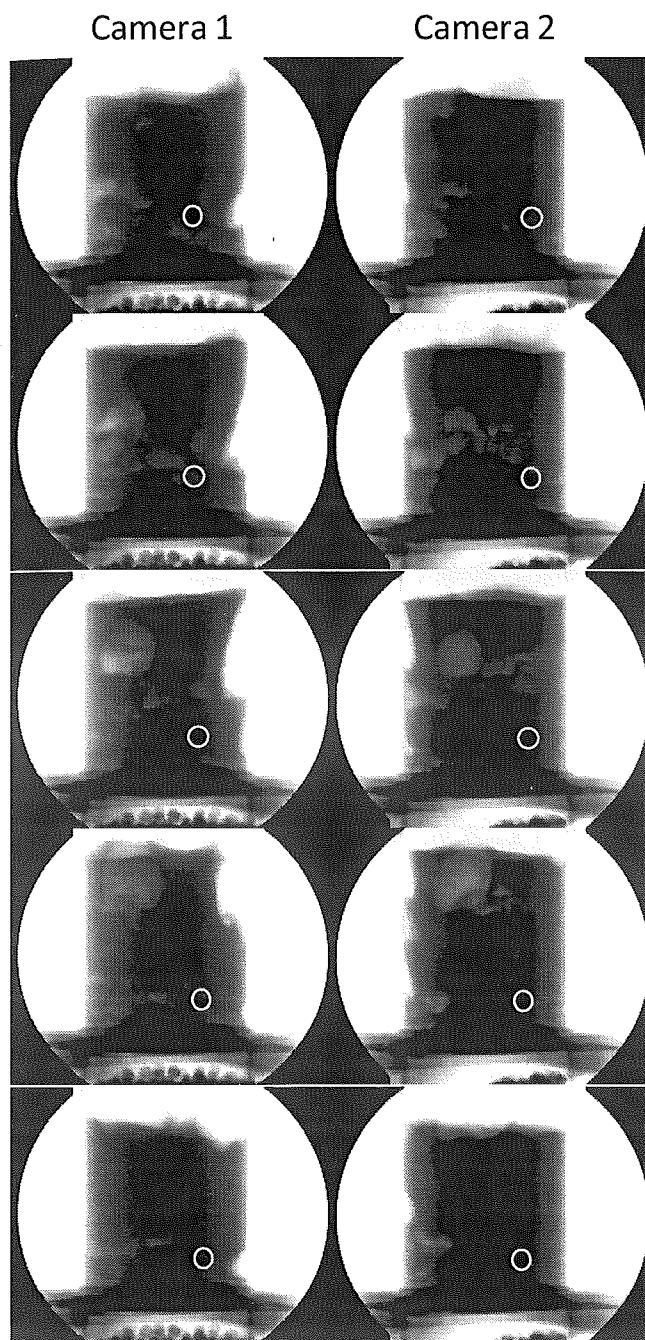
The sample particles manufactured to be fluidized bed intruder particles in this study were stable and resisted attrition when coated with fingernail polish. Overall, the manufacturing process was reproducible and provided the desired intruder particle characteristics.

#### **CONCLUSIONS**

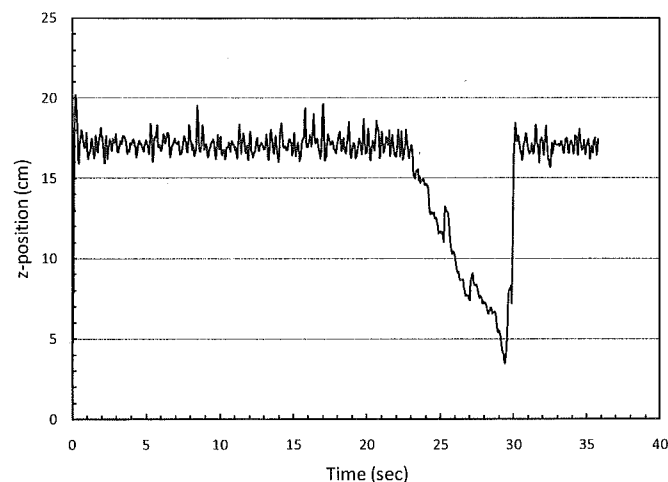
This paper has outlined a process to manufacture tracer (intruder) particles that can be used in X-ray Particle Tracking Velocimetry. Tracers with specific characteristics can be produced and used effectively in XPTV imaging, as shown in the sample fluidized bed study.

#### **ACKNOWLEDGMENTS**

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**FIGURE 7:** TRACER PARTICLE LOCATION IN AN OPERATING FLUIDIZED BED. THE TRACER PARTICLE IS IN THE CENTER OF THE WHITE CIRCLE. TIME BETWEEN EACH IMAGE SEQUENCE IS 55 ms.



**FIGURE 8:** VERTICAL (z-POSITION) TRACER PARTICLE LOCATION AS A FUNCTION OF TIME IN AN OPERATING FLUIDIZED BED.

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**VOLUME 1  
PART C**

FLUID-STRUCTURE INTERACTION AND  
FLOW-INDUCED NOISE IN INDUSTRIAL APPLICATIONS

LIQUID-SOLID FLOWS

GAS-LIQUID TWO-PHASE FLOWS

NON-INVASIVE MEASUREMENTS IN SINGLE AND MULTIPHASE FLOWS

FUNDAMENTAL ISSUES AND PERSPECTIVES IN FLUID MECHANICS

FLOW MANIPULATION AND ACTIVE CONTROL:  
THEORY, EXPERIMENTS AND IMPLEMENTATION

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