Advances in Additive Manufacturing of Materials: Current status and emerging opportunities

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Lecture 11

In last few lectures, I have provided an overview of different additive manufacturing processes, particularly for metals, because we have not discussed so far on the 3D printing of hydrogels of soft materials, it was more for hard materials like metals or ceramics. And, in that context I have covered selected laser sintering, selected laser melting, directed energy deposition, lens processes, binder jet 3D printing and electron beam melting. I have also promised you guys that I will cover more on the process science of some of the selected additive manufacturing processes. And in this context in this lecture I will be discussing more on binder jet 3D printing processes in particular relevance to the kind of underlying process science. This binderjet printing has been investigated for quite some time and there are several researchers who have also provided some understanding on this subject. For example, Denesuk, Popovich and Charles Williams, they have used droplets of 2 mm diameter which is fairly large in size compared to the components that we are interested in and also have good used larger drops of 6 millimetre in diameter.

Typically in today's additive binder jet 3D printing, our inkjet printing, our drop sizes vary in 10 to 80 micrometer. And what is very important for you to realize and that I am going to cover in this lecture is that how to do dynamic monitoring to grab or capture real time ink powder interactions and quantitatively analyzing drop spreading and infiltration. that is what is much less explored in the binder jet 3D printing community. what we have used? We have used essentially X-ray imaging techniques.

this is that experimental setup that you can see. you have a droplet generator here and this droplet generator will generate drop and demand. This drops essentially through this printhead and you may control this ink reservoir. These drops will be essentially impinging on the powder bed. This is porous powder bed.

and this is the synchrotron X-ray source which will be focused very carefully and then images will be acquired here in the 2D slice manner okay. And acquisition speed is 500 frames per second. So, each second 500 frames will be continuously acquired, they are

recorded. So, you can imagine that the quantum of data that would generate in the entire experimental time which will spread over several seconds and this print head once it is released, it releases kind of 1000 to 6000 drops essentially. And this powder bed is essentially porous, we have used in our experiments alumina powder bed.

So, ink what we have used this ink is distilled water or ethylene glycol in different ratio. So, why different ratio? Essentially by changing the ratio of the distilled ethylene glycol, you can essentially change the viscosity and surface tension of this particular binder fluid. What are the printing parameters of relevance? For us the nozzle die is 60 micron and this is the frequency and dwell time and fall time and this frames per second that I have mentioned. This is the experiments were conducted in collaboration with University of Manchester, Professor Brian Derby's group at Diamond Synchrotron Centre in UK. So, where this 10 to 30 kilo electron volt X-ray was focused on this particular chamber, experimental chamber, where alumina powders were kept in a polymeric cuvette.

And, then water H2O and ethylene glycol this mixture in different ratio is being fed into that which is being forced in a drop by drop manner. So, this is the microfiber tape filter head and as I said this is the simulated ink. And each second 500 frames are being recorded and it shows that one particular frame and then you can see that what is the 2D projected X-ray radiograph of dynamic wetting contour okay. And this is the beamline stage and then beamline stage is being rotated each time. what is the experimental steps to be involved? what we have done we have first 3D printed cuvette of 2 millimetre and 4 millimetre cavity and which is SLA printed this is polymeric vessels and which is filled up with this alumina powders.

And then after that we have used high speed X-ray 2D shadow graphs and then also in the second stage we have used 3D tomography. In the X-ray tomography we have used the exposure time of the 8 milliseconds. Synchrotron experiments is one thing but data analysis is far more extensive which has been mentioned here. And what you can see in the synchrotron experiments and data collection, so the first part of the top part has been mentioned that you have alumina powders for the simulated bed, then powder holder cuvettes and then simulated ink composition, then ink loaded in microfibre printed 16 micron orifice. Then, DoD inkjet printed, these are 90 volt driving voltage, 3 microsecond rise and fall time, 30 microsecond dwelling time, printing frequency is 3000 hertz and up to 5000 droplets were printed.

What we have used, it is a pink beam synchrotron X-ray 10 to 35 kilo volt and this is a refraction based on first order phase contrast imaging. we have used specific scintillator based detector with 2.2 into 2.2 millimeters per field of view, 500 hertz time frame and 1 millisecond exposure time. Once we have used that, now next part is data analysis.

this is the entire workflow which was used in the data analysis and in this data analysis workflow, what you see is that this X-ray image data which is 4 terabytes. So, 4 terabytes is a very large amount of data, then you import X-ray image data to mining the relevant data for post-processing. Then you can crop essentially this image sequence either time or space and extract the region of infiltration. Then you divide the image sequence with first image pre-infiltration to capture the fluid movement. Then wet phase like you know after the interactions of the binder with the powder, the wet phase is binarized, age detected and smoothened, then local and global kinetics of the wetting front and area were plotted against time, then some of the semi-empirical models were utilized to fit it and several parametric dependence of the wetting volume like bed porosity was measured.

as you can well realize from these kind of experiments and data analysis that synchrotron experiments is the first step but data analysis is far too time consuming. what you see in this particular slide some of the example of the acquired data. For example, in the first thing is that background is to be subtracted for the liquid solid interface 5 frame by frame and real time dynamic liquid front is embedded in the original porous powder bed. this is the porous powder bed as you can see and I will show you some more examples. in the powder bed with alumina particles are used, the porosity is 0.

7, particle diameter is 15 micron and contact angle is 70 degree. It is not purely hydrophilic and number of particles of this alumina particles you can see that majority of the particles is in the around 10 to 15 micron and there is a large extended tail. it is a kind of more log normal type of distribution. Ethylene glycol to deionized water, it is in various ratios. one of the ink formulation which has 3 is to 1 ratio.

It has dynamic viscosity 7 millipascal second, surface tension 52 millinewton per meter, Ohnesorge number is 0.12 and density is slightly above that of water that is 1080 kilogram per meter cube. this is what I was telling mentioning to you a few slides back that both this 2D images are to be segregated both in time and space domain and this is called region of importance. it is at the very start of the experiments then as it interacting that binder is penetrating into the powder bed. it is going deeper and deeper into the powder bed, it is like at 400 milliseconds, it goes to 600, 800, 1000 milliseconds, it goes more and more into details.

Now, when you threshold and bannerize this wetting contours, then you can see at 200, 400 to 1000 milliseconds how this wetting contour spreads. Edge detection was conducted. edge detection allowed you to find out that what is the penetration depth. and what is the spread. what is the penetration depth and spread that was important because as you measure this penetration depth and width as a function of time, then you can do

that color map for the time dependent contour evolution and as a result you can actually feed this data into some of the analytical models right.

this analytical model some of the examples I will be showing you in the next couple of slides. this is like a 2D image X-ray image and this is the infiltration study what you can see this is that liquid inkjet printed liquid interface right you know how this contour actually essentially penetrates into the powder bed. And at different time frame you can find out that how the depth also increases, spread also increases and then different coordinates you can see that you can measure. This is essentially done in the same experiments the porous alumina bed. at the beginning of the experiments now if you follow that how this video actually was recorded at 0 seconds.

Then if it comes at the 0.9 seconds, so you quantitatively measure that what is the surface spread and penetration depth and also what is the voiding contour area A. And this one that I have shown you before, this is after the edge corrections, right. Here you have shown that edge detection was done for penetration depth. Then if you look at this particular ratio of this ethylene glycol to deionized water 1 is to 3 to 1 is to 1 and if you look at this how the interface position has been changed.

You look at carefully this interface position was measured with respect to the baseline and here the qualitatively in both the cases the interface position. and wetting area both increases are not strictly in a linear manner but almost like a linear like manner. And this is for the link to ethylene glycol or ink that ethylene glycol to deionized water is 1 is to 1 ratio. If when it is 1 is to 3 ratio then it goes up then it goes through some kind of a pseudo steady state then it increases then it goes to kind of pseudo steady state then it increases. qualitatively although there is no distinct differences in terms of how the interface position and projected area increases with respect to time, but suddenly 1 is to 3, this is little bit more sluggish increase compared to more steady increase that we have observed in case of 1 is to 1 ratio.

The color map below essentially shows as a function of time like 0.1 second increases to 1.2 seconds. This green one is the wetting contours develop and then spread across the print bed.

that has been mentioned. there is certainly there is a when this binder droplet essentially penetrates or interacts this is more of an impact driven at the initial stage. But then when it goes into this print bed then there is a transition from impact driven to capillary driven phenomena that actually drives this wetting of the powder bed with the binder. what you see here, it is a penetration depth is being plotted against the post impact time and which is fraction of a second. from 0 to 0.

6 second it increases. The same thing it is that angle is around 82 degree. But it is that this penetration depth you know when it is shown for more initial impact driven to more capillary driven if you see this angle changes quite a bit. Angle is now from 82 degree to 54 degree. And this is essentially fraction of a second again this increases not exactly like a linear but almost like a pseudo linear like increase of the waiting area with respect to post impact time. kind of more analysis of the penetration depth, wetting area and volume and how they are being distributed globally with respect to the post impact time.

lateral spread it almost follows a non-linear like but if you see that b(t) is 2.86 into t plus 0.016 to the power 0.51. it is if the power is 1 then it is almost linear but it is not essentially linear.

Penetration depth with respect to post impact time again it is a non-linear because when we fitted this data we are getting the function is dt is equal to 1.34 multiplied by t plus 0.0044 to the power 0.33. it is not non-linear so power of exponent is T plus 0.

0038 to the power 0.7 and transformed wetting volume that what is the wetting volume this is like V is equal to 8 by 3 S square by pi d so this is the volume that you get. As I said before that there is a number of or a few researchers have made attempt to provide some theoretical understanding into the binder jet printing. we have utilized those analytical framework but at the same time we have modified those analytical framework based on the experiments which were performed at the X-ray synchrotron centre. essentially we are more interested that how wetting volume they evolve with time and if you look at this, this is the droplet impact plane and this particular droplet impact plane as I said that it is impact driven. to capillary driven that is a transition during the 3D binder jet printing processes.

droplet impact plane and impacted droplet in spreading surface and volume if you see these two dimensions rs and rv that rs is almost equal to rv. And, Denesuk actually first gave some quantitative estimates of that how the wetting volume increases with time. this kappa is some constant, b is that lateral width and t prime as any instantaneous time. Holman is another researcher who has provided wetting volume and this V dot and V dot wetting volume is essentially related to K is equal to pi P and gamma LV is equal to liquid volume, interfacial energy, cos theta is that wetting angle, r is the pore diameter, eta is the viscosity. this term which essentially is explained here kappa term.

what we have done? experimental data or synchrotron data and when we have plotted the synchrotron data wet particularly wetting volume with respect to the post impact time again as I mentioned that post impact time is essentially less than a second is up to 0.6

second. experimental data is this individual point. what is the semi-empirical best fit? At p is equal to 0.

5, you get this blue line. And semi-empirical 0.7 is this particular line. This is the p is equal to 0.

7. What is this p value? p value is here. Now, there are other semi-empirical values, this green one is P is equal to 0.

1. So, 0.1 to 0.7 varies and P is 0.5 is the best fit. by varying the porosity, so this is the porosity fraction, powder bed porosity. by varying the powder bed porosity what we are able to see that at point when powder bed porosity is 0.5, then our experimental data matches very, very closely with that of the modified washburn's model. And we are getting very close approximation or very close correlation between the experiments and analytically obtained these values.

this is the summary of this part of the process science. As I said that this is a complex set of experiments and detailed analysis of the synchrotron data. we have used synchrotron X-ray at diamond station, we have used this print head which essentially releases several thousand drops, we have used alumina powder bed. And from using the synchrotron X-ray we are able to capture several 2D films or 2D slices. Now this has been categorized both time and space and then we have done detailed analysis including edge detection to show to quantitatively find out that what is the depth and spread of this of the wetting volume or wetting contour.

We have also find out that what is the area and wetting volume. Then when you plotted about the how the interface position has been growing towards more and more to the powder bed that is the time increment with the 0.1 second resolution you can see the rate to this other spectrum of the colour. Then we can see that you know how the wetting volume increases with time and as explained in last few minutes is that there are some established theories which we had to modify to take into account our present experimental set up or take into account or while rationalizing our experimental results to see what is the powder bed porosity that we should consider to find out. whether the experimental results can be closely correlated with the theoretical predictions.

And what we have found that at powder bed porosity of 0.5 this correlates very well. Srimanta was the PhD student in my group at Indian Institute of science. He was the lead author of this paper which is published in ACS applied materials and interfaces and this was a collaboration with Brian Derby's group.

at University of Manchester. And Srimanta actually spent common wealth split site fellowship at University Manchester and then he have published few papers during this collaboration. In the next lecture, I will come back to you to present some of the scientific case study. Thank you.