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

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Lecture 07

Let us start with the basic definition of fracture or basic description of fracture in ceramic materials. Now ceramics are widely known as brittle materials. Ceramics have covalent bonding. Ceramics have covalent bonding that is the inter atomic bonding and also ionic bonding. That why the ceramics are brittle that lies primarily in the characteristic bonding structure of this particular materials. Now, bonding in a very fundamental level can be described by this kind of spring model.

Now hypothetically if you see that if you load this ceramic under tension with sigma, these springs they get extended along the direction of the tensile forces. This is the inter atomic distance. A prime or A naught that is the original interatomic distance or initial interatomic distance. Now as these springs get stretched so it so happens the interatomic bond distance also increases with time.

At a critical stress level what happens at a critical stress level what we call as a fracture stress. This particularly, this springs like structures what I have described in the beginning that get broken and once they are broken then cracks can pass through go through the materials leading to the total fracture of the materials. Ceramics are very tricky materials this fracture is very important. to understand. Now if you look at in a more detailed manner that why this fracture takes place.

Now if you have edge crack right, if you have edge crack around the crack tip there is a stress concentration. What is the meaning of stress concentration? That you know if you apply the stress sigma everywhere remotely from the crack it is all sigma. But if you go here, it is not sigma, right. The stress at this red circle region is certainly not sigma. It is elevated.

Sigma is not sigma. If the stress is not sigma, it is elevated. And this elevated stress level if you can describe it as sigma max. right. And then sigma max is essentially related to the crack length that is C and also related to radius of curvature , sigma into C and comma rho, rho is the radius of the crack So and typically sigma max is equal to sigma

square root C upon rho that means longer the crack length and finer the crack tip radius more is the stress concentration.

So compressive strength I have mentioned that is typically load divided by area in one lecture I have clarified to all of you that ceramics are much better. under compression compared to tension and it really outperforms metals in terms of the compressive properties, compression properties. And one of the thing that I want you to recall that I have mentioned very categorically the compression strength of ceramics is around 8 times larger than that of the tensile strength. Now if you look at this what happens during tension if there are cracks which are distributed very randomly in ceramic then this crack which are most favorably oriented perpendicular to the stress axis tensile stress axis those cracks will essentially propagate leading to the complete fracture of the materials. Under compression, The cracks which are oriented most favorably towards the compression directions or very near in terms of the angle to the compression directions, those cracks will essentially propagate leading to the fracture.

So, for example, these are like secondary cracks which will be generated. Now, these secondary cracks will lead to the fracture of the materials. So, this is like compressive failure. Third one is the flexural strength that is the three-point bending. In the three-point bending what it means that for example, this particular flexural sample, this is resting on two support rolls, right.

On the top through this one that you apply the force. So if it is P then 2 reaction force is the P by 2 P by 2 right. According to Newton's law every action there is an equal and opposite reaction. And if you do simple mechanics of materials like if you consider this is a flexural bar and this is a neutral axis in the flexural bar and this is under 3 point configuration right. Flexural configuration P and P by 2, P by 2 the stress at any plane which is at a distance from the neutral axis can be described by this Bernoulli's equation sigma by Y is equal to M by I is equal to E by R.

So sigma is the stress at a distance Y from the neutral axis, M is the bending moment I is the moment of inertia, second moment of inertia, so it depends whether it is a rectangular cross section sample or cylindrical or circular cross section samples accordingly you know that I value is different. E is the elastic modulus of the material and R is the radius of curvature. So under the flexure the material bends and then this particular curvature is typically R. So first set of equations sigma by z m by R is used to arrive at fracture strength is 3PL divided by so L is your length between 2 rolls okay. b by d square, b and d are essentially dimensions of this rectangular cross section samples.

So, this is like you know how it is done. So, there is a two type of testing configuration is

possible 3 point and 4 point testing. So, this 3 point and 4 point testing their bending moment diagram also if you look at this bending moment diagram here, so it is like this it goes to increase and here bending moment diagram is some are like this. So, it is a uniform bending moment where this So this is your bending moment M and this is the distance okay. Now mechanical properties of polymers.

Now polymers they can be either crystalline or amorphous. Now typically for metals What happens that I have shown you that in the description in the very beginning of the lecture, I think when one of the first previous lectures, I have mentioned these atoms are organized in 3-dimension in regular periodic manner, right. The concept of crystallinity in case of metals and concept of crystallinity in case of polymers are very different. So in metals, crystallinity essentially refers to the way the atoms are organized in 3D space In case of polymers, Christianity essentially refers to the way that polymeric chains are organized in a local region in a very regular and periodic manner because polymers essentially means you have a mar units and you have a many mar units, okay. Now, crystalline materials have molecules arranged in repeating patterns and amorphous materials have molecules organized random manner and in most cases amorphous materials have no long range order but may have limited short range order and crystalline materials have both long range and short range order.

and morphology of the most polymers are essentially semi-crystalline. what it means they have amorphous domain and they have crystalline domain. these polymers for example if you consider this top part this particular polymeric chain is organized in a parallel manner. essentially this is more crystalline polymer but this part is amorphous region right this H part. In amorphous polymers all these chains are essentially in a very disorganized manner.

there may be in limited manner some short range order in some part or very microstructurally small parts these chains are organized in a parallel manner or organized manner but otherwise this polymer is essentially amorphous in nature. This is a classic example of semi-crystalline polymer the way this schematic is mentioned here. which is the crystalline part? The way I am trying to enclose this particular area with a dotted circle. where all the chains are parallel to each other. this is like a crystalline part.

except this part you know except this some of this marked regions everywhere else this polymers this materials have very disordered region. these are like amorphous region. this particular amorphous solid is formed when chains have little orientation throughout the bulk polymer. The glass transition temperature is the point at which the polymer hardens into an amorphous solid and this term is used because the amorphous solid has properties similar to that of the glass, Now, although this has been mentioned to you in a very very

briefly in one of the earlier lectures that polymers they have a unique property that is called viscoelastic deformation I will come back to this viscoelasticity also when I will be explaining to you some part of the rheology. it is a viscous liquid and elastic solid.

it has a linear elastic region. the slope of this linear elastic region is the elastic modulus and it is followed by yield strength. and then extended deformation at a constant load and that followed by some increase then leading to failure. this what we call plastic deformation it is more essentially described by viscoelastic deformation. this is the typical characteristics of this polymer deformation.

Mechanical behavior of polymers, a tensile dog bone type of samples where polymer chains are distributed along this backbone chain. Now, what I am trying to tell you is that if it is pulled in tension, what you notice from this to this position, some chains are being organized along the direction of the force. And subsequently if you see this change this is essentially all parallel to the direction of the tensile stress direction and therefore this deformation behavior allows the polymer to crystallize because this crystallization here is essentially referred to the localized organization or reorganization of the polymer chains to give rise to a more regular arrangement of these chains in a macroscopic domain and in this microscopic domain this is here what we call is the neck region and this neck essentially extended slowly along the entire length of this polymer sample and this gives rise to the time dependent deformation or an elasticity in the polymers. And this also explains why polymers undergo kind of extended deformation at the constant load or constant stress. this is more about this I think with this we will complete that kind of mechanical behaviour of materials and now I am going to start with this material structure how you can determine the material structure.

in the beginning of the last lecture I have mentioned that structure property correlation is the central theme in this entire materials or mechanical in the inter material science. I have described I think to a significant extent on the properties. Now, let me discuss about this materials structure. Let me change my file. As i mentioned that we will start with this material structure characterization and this structure characterization is very important because structure property correlation is the hallmark in material science.

before I go on describing that different structure characterization, this is the conceptual tetrahedron processing structure property performance tetrahedron. Processing means to provide desired shape using mechanical or micro mechanical, thermal and mechanical or chemical route. Structure means it is more macro and micro structure of the materials. Properties is more of the mechanical and functional properties and performance is more on the efficacy of a material for engineering applications that is how performance is determined. Packing of atoms if you look at this how this in 3 dimensional space this

atoms are packed together.

Now, you have this first layer of atoms for all in this particular plane all the atoms are packed together. Now, you will find that when you do this there are some void space here. This void space actually this red circled atoms are being placed. Now when you do this red circled atoms then you will find there is some void space here at where 3 red atoms are present and that is the space when yellow atoms are being placed. this is like one of the unique ways of organizing atoms in three dimensional space.

But there are several such unique ways, unique distinguishable ways these atoms are packed in three dimensional space and this particular atomic arrangement or organization leads to lattice structure and also this is defined by the unit cells and so on. Now, how you can characterize the structure? One of the techniques that has been used for decades is X-ray diffraction. you take monochromatic X-rays. These monochromatic X-rays are essentially diffracted by these atoms and these atoms will diffract And then several such monochromatic X-rays comes with a particular wavelength lambda and then if you have this angle of diffraction theta and if this is that interplanet distance dhkl then Bragg is the scientist who solved this basic theory and then he has come up with n lambda is equal to 2d sin theta. So what is d? d is the interplanar distance.

What is hkl? hkl is a plane. In typical material science, you can essentially define different planes by Miller indices and this hkl is essentially Miller indices. what are X-rays? These are beams of electromagnetic radiation, this is short wavelength and high energy and it is what is wave, it is like sinusoidal oscillating electric field with a right angles to it in a magnetic field. it is described essentially lambda nu is the frequency. it is photon energies typically E is equal to h nu, these are like very basic fundamentals that you remember from the physics. And what is X-ray used for? Determination of crystal structure, also unit cell dimensions, determination of grain size and strain and typical metals, typical samples which are used like metals, ceramics, glass and various materials.

diffraction is essentially x-rays to experience positive interference in crystal that is called diffraction and this is in a more textbook type of definitions that when a periodic array of objects if I go back to this particular drawing schematic drawing a periodic array of objects like this is essentially atomic plane these planes of atoms when the periodic array of objects. scatter radiation coherently. that means each of these atoms essentially will scatter this extra radiation in a coherent manner. The concerted constructive interference at specific angles is called diffractions. I underline specific angles, I underline constructive interference and diffraction in crystalline materials is best described by Bragg's law which I have just introduced you in the last to last slide is n lambda is equal to 2d sin theta.

this positive interference too takes place the path difference. path difference must be equal to one wavelength or multiple wavelength. n is the order of reflection in this and here it is clearly shown that X-rays in phase. n is essentially order of reflection.

this is a typical X-ray. if you have the Y along the Y axis, it is the intensity and along the X axis, it is 2 theta. What is theta? Theta is the angle of diffraction. you will see that when it is recorded in a machine not is as fast as you see here but it is fast enough depending on what is the scan speed and what is the step size and so on and so forth. You get this is called background intensity. Now, then at 2 theta is 20 degree or so on, so there are certain characteristic peaks are coming And these peaks are essentially determine some of the crystalline phases in the material.

What is the intensity of the crystalline intensity of the X-ray? This is called characteristic peak. an X-ray diffraction essentially described by 2 things. One is the background intensity, one is the characteristic peak, their position and also their intensity. And you will see there is a multiple such peaks are there, And these peaks are to be indexed and thereafter there are ways that how to assign them to a specific materials or metals and these materials can be either metallic materials or ceramic materials or composite materials and so on. This is the background intensity and then second one is the peaks at certain angle with certain intensity as I mentioned resulting from positive interference of X-rays with crystal structure of one or more minerals.

Minerals can be either materials, different materials. As I said that X-rays also can be used for measuring strain. one such example has been shown here for a particular crystal lattice when it is unstrained conditions it will give a characteristic peak. Now, when the material undergoes strain, this can be essentially shifted towards the more on the left or there is a strain induced peak broadening. what I am saying, one is a shifting of peak and one is a broadening of peak.

both essentially may signify that the material has under constraint. I use the word may carefully because it can have another reasons that why this peak broadening can take place and that is because of the particle size. But if you know the condition of the samples that you are testing using extra diffraction and if you know the material has undergone deformation and then suddenly you see that the material that particular peak has been shifted or peak has been broadened then you can attribute that kind of peak broadening, peak shifting to deformation of the material. This is one such examples in the annealed brass. in case of annealed brass you know if it is rolled and then subsequently after that it has undergone heat treatment 200, 250, 300 up to 450 degree Celsius.

In the annealed brass if you see that since it has undergone deformation there is a very clear peak broadening. 200 also it is peak burden is there and then suddenly at 250 onwards that you know that when strain is getting removed this is for the all this slowly the strains get removed then you get very characteristic peaks of this brass. X-ray diffraction this summary is that you know it is a non-destructive technique that is used to identify the crystalline structure and composition of a material. This X-ray beam sample it is a transmitted beam and diffracted beam and Bragg's law of diffraction that you can get from this figure that CB essentially C and B is essentially is equal to BD is equal to D sin theta. and part difference between ray 1 and ray 2 that is essentially delta.

delta is equal to CB plus BD right and if CB is d sin this 2d sin theta. part difference delta is equal to n lambda where n is equal to 1, 2, 3, 4 and this is the 2d is equal to n sin theta. this essentially shows that you know how this Bragg reflection takes place for every diffracted beam there exists a set of crystal lattice planes such that diffracted beam appears to be reflected from that set of planes Then move on to the electron microscopy of materials, these are used to image the microstructure of the materials, whether it is metals or polymers or ceramics, all the different materials can be imaged by different electron microscopy techniques. first and foremost things is that you need to know that what are the different characterization techniques and what information you can extract from this characterization techniques. There are certain abbreviation has been used in this particular slide that is TEM stands for transmission electron microscope, SEM stands for scanning electron microscope, FIB stands for focused ion beam.

for all this additive manufacturing courses I think perhaps if you know more of scanning electron microscope and little bit of transmission electron microscope that is good enough and I have already mentioned about your X-ray diffraction. these 3 techniques more or less are useful to characterize that additively manufactured course samples or components or parts. Now what is the detection range in this additively these different techniques and what is the analytical spot size. in X-ray if you see the spot size is fairly high it is more than 100 micron. Now if you go to SEM and so on the spot size is relatively low that is kind of almost like 100 nanometer and so on.

And detection range like atoms per centimeter volume of this atoms that you can detect and this you can see this is that very high that 5 $X10^{21}$ atoms per centimeter cube this you can essentially detect. it is almost like 10 atom percentage that can be detected in this one. This is like different characterization techniques and what kind of information that you can obtain from this characterization techniques that has been mentioned. you can see this scanning electron microscope it is used for up to 1 nanometer resolution and elemental mapping down to few micron thick. Then there is a transmission at a microscope, this morphology elemental mapping up to atomic scale resolution.

Then you have X-ray diffraction that is a crystal structure and material modification of phase diagram of the solids that has been also. you know if you look at this particular 2-3 different techniques, this should be enough particularly in the context of additive manufacturing of materials. Now scanning electron microscope as the name suggest you use electron beam and this electron beam essentially, so this is a accelerated electron beam and it is typically accelerated around 20-25 kilo volt. this is a stage and here your samples are kept. you can see this is the door through which actually you can push these samples inside microscope chamber.

this chamber is kept under tight vacuum right and this is your electron beam line. This electron gun is here and you have either tungsten filament or lanthanum boride filament lap 6. and you have a Gun alignment control and pneumatic airlock valve. At different path of this electron beam, you have a condenser lens, you have objective lens and so on. And as these particular lenses are magnetic in nature, so these are like magnetic lenses.

It is not like a simple lenses that are used in the optical microscope. And then it is a motorized stage. This motorized stage actually is used to have a control of the sample movement. You can do in the mostly x, y and z movement. z movement is restricted because that is the working distance between the beam and then objective lens and then sample.

that is typically kept at some specific value like 10 mm and so on. from scientific standpoint, it is important to understand the electron beam material interactions which are essentially very important to get to know that what are the information we can get and what is the typical interaction volume. if you have electron beam, E beam. And when the electron beam interacts then it is having a Pear shaped interaction volume. And this Pear shaped interaction volume in the beginning they release the secondary electrons and then there is also primary backscattered electrons that will give you atomic number contrast and some information but secondary electrons mostly topographical information, your X-rays will give you through thickness composition information.

But here there is a catch this excess that will be emitted it will certainly not give you very reliable information if for the low atomic weight element like oxygen, nitrogen, carbon and so on. But for larger atomic weight element this will give you quite good reliable values. Now this interaction volume if you see this incident electron the interaction volume this is called pear shaped interaction volume. And this pear sphere shaped interaction volume that extends quite a few microns into the depth. if it is a 10 nanometer, this is a principal beam size, this interaction volume, this is a width of around 1 micron and there is also depth to which this will operate.

And this is the secondary emission range, secondary electrons and backscattered electrons and X-rays which I have shown you before. transmission electron microscope again it is a high energy electron beam. it can go to 200 kV up to 300 kV or even some of the most modern microscope 300 kV electron beam that is used. this is the typical electron path how it goes and you can see that constantly this electron beam goes and they are being guided. using a different kind of magnetic lenses like condenser lens, intermediate lens, project lens.

you have a condenser lens, you have objective lens and you have intermediate lens. And there are different image planes that are also being mentioned like object plane, the first intermediate image plane, this is secondary, second intermediate image and so on. And finally, you see that image here, this is the viewing screen. this is you are talking about this viewing screen, through this viewing screen you can look at this image very very carefully and also you can get the diffraction pattern which can give you an alternative way to essentially analyze the materials microstructure.

electron beam interactions also has been described here. You have the incident high energy electron beam as I said that 200 kV or 300 kV electron beam and then this goes through direct beam. There is elastically scattered beam and there is inelastically scattered beam. And they have a secondary electrons, characteristic X-rays those you have seen in earlier one, earlier slides in this particular lecture. And these are like different signals which are generated when high energy beam of electrons interacts with a thin specimen. it is indeed important to use SEM and microcomputed tomography which I have also mentioned in one of the last lectures, few lectures and this microcomputed tomography they use X beam.

But this X-ray beam typically is around much higher than that of the X-ray diffraction. It is like 70 kilo volt and your samples like 3-dimensional let us say additive manufacturing porous scaffolds are being there. this particular sample holder will rotate right and if it rotates the X-ray beam continuously falling then you get 2-dimensional slices. and these two dimensional slices they are stacked up and they get a 3D image.

this brings me to the end of this lecture on materials characterization. what I have not shown you but perhaps that would be important in some specific cases is the surface topography like additively manufactured part, how to make the surface topography. This is very sophisticated atomic force microscope but normally you can use some of the other like optical perimetry and so on that you can use for the surface topography. I have already shown you some microstructure and phase assemblies that is good enough for additive manufacture In case of surface chemistry, XPS, FTIR, Raman spectroscopy.

That is not something very widely used, regularly used for additive manufacture But just to give you a complete idea, there are also several other techniques which are used for materials characterization. Since it is not a course on material science, I am just showing you this last slide just to show you that in case of other material science courses, an instructor professor may teach you on how to measure the mechanical magnetic properties, how to measure the electrical properties and surface chemistry. this brings me to the end of the materials characterization. Thank you.