Non - Metallic Materials

Prof. Subhasish Basu Majumder
Department of Materials Science Centre
Indian Institute of Technology, Kharagpur

Module - 07

Processing of non - metallic materials, sintering and microstructure development

Lecture - 34

Preparation of ceramic powders: auto-combustion, sol - gel synthesis, microwave assisted hydrothermal synthesis

Welcome to my course Non-Metallic Materials and today we are in module number 7 Processing of non-metallic materials, Sintering and microstructure development. This is lecture number 34, where I will be talking about Preparation of ceramic powders by mostly three different techniques.

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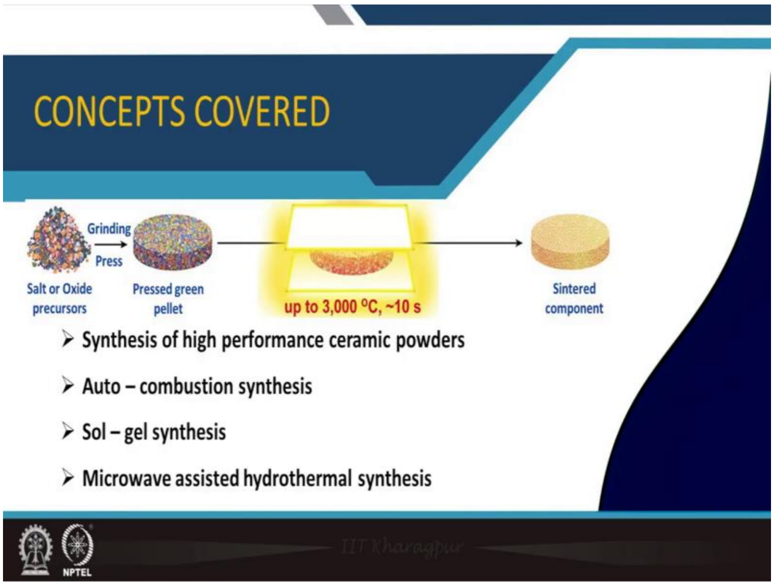

CONCEPTS COVERED

- Synthesis of high performance ceramic powders
- Auto – combustion synthesis
- Sol – gel synthesis
- Microwave assisted hydrothermal synthesis

Now, you know that in the oxide ceramics or the other types of ceramic materials high performance ceramic powder preparation is one of the major issues, and therefore, these three techniques actually is used auto combustion synthesis, sol gel synthesis, and microwave assisted hydrothermal synthesis. These are three very popular powder preparation techniques, and these are economic and very easy to use in the laboratory as well so, they are extremely popular.

So, although there are various other types of ceramic powder processing they are available in the literature, but I have decided to go to take you through these three particular techniques, which are so useful to grow the so called nano materials.

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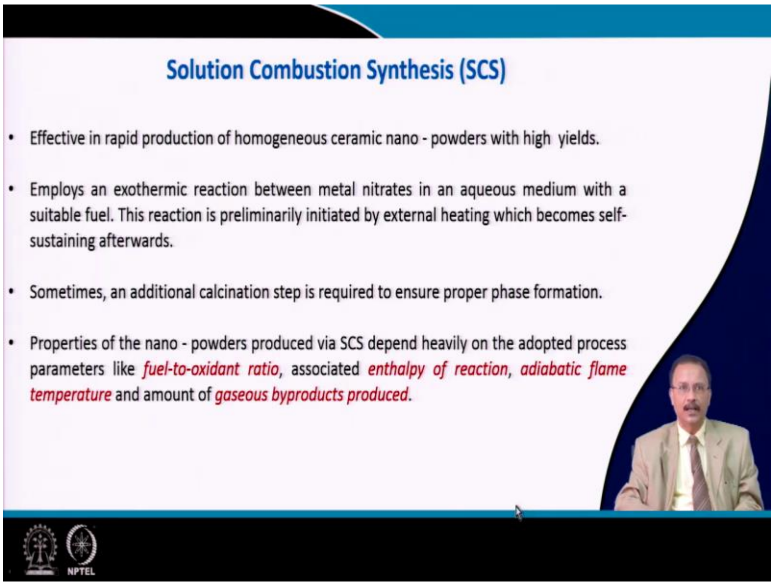

Solution Combustion Synthesis (SCS)

- Effective in rapid production of homogeneous ceramic nano - powders with high yields.
- Employs an exothermic reaction between metal nitrates in an aqueous medium with a suitable fuel. This reaction is preliminarily initiated by external heating which becomes self-sustaining afterwards.
- Sometimes, an additional calcination step is required to ensure proper phase formation.
- Properties of the nano - powders produced via SCS depend heavily on the adopted process parameters like *fuel-to-oxidant ratio*, associated *enthalpy of reaction*, *adiabatic flame temperature* and amount of *gaseous byproducts produced*.

So, first I will start the solution combustion synthesis which is abbreviated at SCS and this is effective in rapid production of homogeneous ceramic nano-powders with high yield. So, if you want to upscale a particular chemistry recipe then this is the technique you should use.

And it employs an exothermic reaction usually between metal nitrates in aqueous media with a suitable fuel so, that this oxidation takes place and this reaction is preliminary initiated by a small external heating which eventually becomes self sustaining. So, at relatively low temperature you can get very high purity powder in nano crystal in form. So, that is why this process is so important.

Sometimes, it also happens although most of the time the prepared powder is crystalline and phase pure, but in some instances they require little bit calcination step to complete the phase formation in a specific phase it forms. So, calcination is required, but otherwise at room temperature you can basically synthesise this powder materials nano crystalline powder materials.

So, properties of the nano powders produced via this solution combustion synthesis this is a this process is actually adopted by many researchers and they depends mainly on the fuel that I have talked about soil and oxidation ratio, and also the enthalpy of the reaction, the adiabatic flame temperature, which I will be defining in a moment and amount of the gaseous by-product that is produced out of this solution combustion synthesis.

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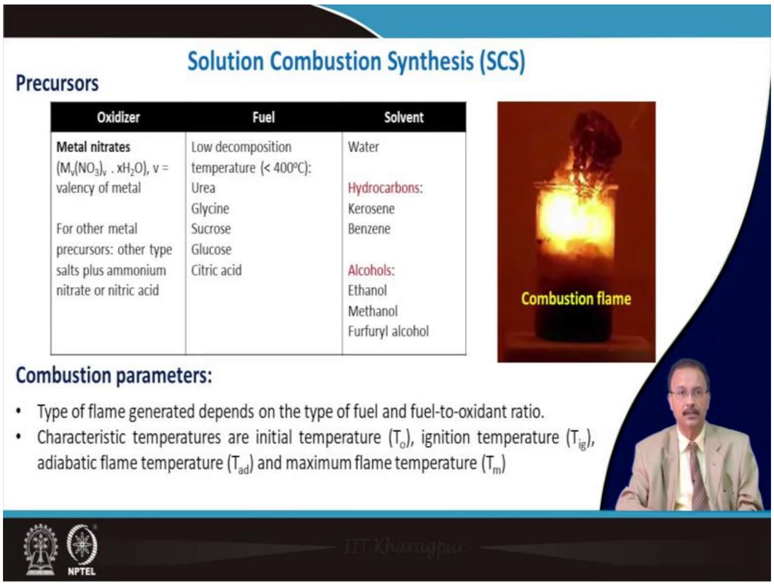

Solution Combustion Synthesis (SCS)

Precursors

Oxidizer	Fuel	Solvent
Metal nitrates $(M_v(NO_3)_v \cdot xH_2O)$, v = valency of metal For other metal precursors: other type salts plus ammonium nitrate or nitric acid	Low decomposition temperature (< 400°C): Urea Glycine Sucrose Glucose Citric acid	Water Hydrocarbons: Kerosene Benzene Alcohols: Ethanol Methanol Furfuryl alcohol

Combustion flame

Combustion parameters:

- Type of flame generated depends on the type of fuel and fuel-to-oxidant ratio.
- Characteristic temperatures are initial temperature (T_o), ignition temperature (T_{ig}), adiabatic flame temperature (T_{ad}) and maximum flame temperature (T_m)

So, as I said that the precursor material they can be subdivided into the oxidizer which are usually metal nitrate. So, most of the cases you will get the metal nitrate precursor material, wherever you are not getting you get some other kind of salt then in association with that those salts you use ammonium nitrate or in some instances nitric acid. So, that eventually they behave like a oxidizer nitrate.

Then you use the fuel typically low decomposition temperature fuels are used which especially, decompose at low lower than 400 degree Celsius. So, use urea, glycine, sucrose, glucose citric acid is also used was citric acid is having additional usefulness in this auto combustion synthesis they acts as a chelating agent, which I am not going to discuss in details, but segregates this is also used.

The solvent is usually water and different types of hydrocarbon can be used like kerosene, benzene, and various types of alcohols. So, eventually you mix all this precursor material and you heat it at relatively low temperature about depends on what kind of solvent and material that you are using, but you can use it in a normal this magnetic stator with heater.

So, that also can be used or a simple mental can be used to heat it. And then eventually it catches fire and a fraught kind of thing will be generated this is a typical picture taken during this kind of auto combustion synthesis. So, the type of flame that generates that depends on the type of fuel and also fuel-to-oxidant ratio.

Characteristic temperatures in this process are mainly four initial temperature where from your starting your experiment then, ignition temperature we call it is a flashpoint also where it is just about to be ignited. Adiabatic flame temperature which can be estimated thermodynamically and the maximum flame temperature which is denoted by T m.

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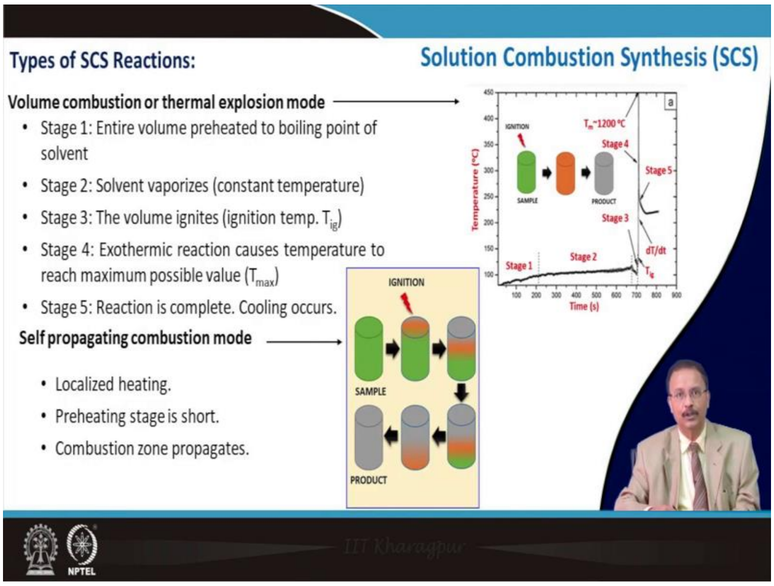

Types of SCS Reactions:

Solution Combustion Synthesis (SCS)

Volume combustion or thermal explosion mode

- Stage 1: Entire volume preheated to boiling point of solvent
- Stage 2: Solvent vaporizes (constant temperature)
- Stage 3: The volume ignites (ignition temp. T_{ig})
- Stage 4: Exothermic reaction causes temperature to reach maximum possible value (T_{max})
- Stage 5: Reaction is complete. Cooling occurs.

Self propagating combustion mode

- Localized heating.
- Preheating stage is short.
- Combustion zone propagates.

So, these are the four type of temperature that is involved. So, this reaction could be of two different types one is the volume combustion or we call it thermal explosion mode also this volume combustion. So, here in stage 1: entire volume created to the boiling point of the solvent then, in stage 2 so, in stage one that temperature is t 0 then there comes stage 2: where the solvent vaporises.

So, when it vaporises then the temperature remains constant. So, it is a linear constant zone then the volume starts to ignite. So, that is the ignite ignition temperature or we call it is a flashpoint. Then exothermic reaction it takes place and it reaches to a maximum temperature which is T m and then finally, it pulls down. So, if you see this view graph. So, this is your starting point and then through the ignition you are getting the final stage final stage of this powder preparation.

If you see the temperature profile so, in stage 1 it is at very low temperature much below than 100 degrees Celsius then, most of the solvents they vaporise around this temperature and it remains more or less constant and it forms a gel kind of mass, and then the flashpoint reaches. So, here the flashpoint reaches within maybe 700 second or something like that.

So, it takes time and then it reaches the flashpoint and then immediately it goes to very high temperatures and the material is formed and then again it pulls down. So, that is the volume combustion technique. There is another one which is a self propagating combustion type. So, it is a localised heating not a bulk heating. So, the preheating stage is relatively short not like 700 second or something like that.

So, and this basically this ignition part the combustion zone that that propagates. So, you see that this has been propagated and finally, your product is formed. So, this is the second type of self propagation mode.

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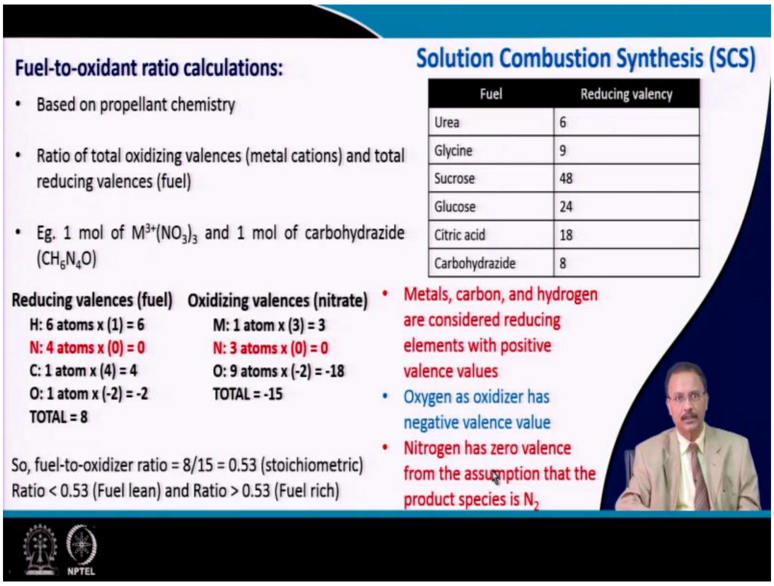

Solution Combustion Synthesis (SCS)

Fuel-to-oxidant ratio calculations:

- Based on propellant chemistry
- Ratio of total oxidizing valences (metal cations) and total reducing valences (fuel)
- Eg. 1 mol of $M^{3+}(NO_3)_3$ and 1 mol of carbohydrazide (CH_6N_4O)

Fuel	Reducing valency
Urea	6
Glycine	9
Sucrose	48
Glucose	24
Citric acid	18
Carbohydrazide	8

Reducing valences (fuel)

H: 6 atoms x (1) = 6
N: 4 atoms x (0) = 0
C: 1 atom x (4) = 4
O: 1 atom x (-2) = -2
TOTAL = 8

Oxidizing valences (nitrate)

M: 1 atom x (3) = 3
N: 3 atoms x (0) = 0
O: 9 atoms x (-2) = -18
TOTAL = -15

So, fuel-to-oxidizer ratio = 8/15 = 0.53 (stoichiometric)
Ratio < 0.53 (Fuel lean) and Ratio > 0.53 (Fuel rich)

- Metals, carbon, and hydrogen are considered reducing elements with positive valence values
- Oxygen as oxidizer has negative valence value
- Nitrogen has zero valence from the assumption that the product species is N_2

So, fuel to accident ratio it is an important aspect and let us see how you can define, that what should be the fuel and oxygen sorry fuel and oxidant ratio right. So, oxidant is basically the nitrate salts and the fuel is whatever I have described urea glycine and other chemicals. So, it is based on propellant chemistry, and ratio of total oxidising agent balances that is the metal cation and total reducing balances that is the fuel that ratio is important.

So, if you take one mole of this metal nitrate and one mole of this carbohydrazide, this is a typical fuel that is used so, this is having this formula. So, what you will have to do? You will have to calculate the balancing. So, this reducing balances whichever is the fuel for hydrogen it is six nitrogen you will have to take it 0. So, this is 4 into 0 and then you have carbon, you have oxygen, so total value is 8.

And in case of the oxidizing agent you have metal, and I have taken a metal atom which is having a valency 3 and then metal nitrate so, these I have calculated as 15. So, the ratio is 8 by 15, the ratio of fuel is to the oxidizing agent that is 0.53. So, if you take it in a stoichiometric ratio of 0.53 then, if it is less than that we will call it is a fuel lean and 0.53 is exactly stoichiometric and if it is more than 0.53 we will call its fuel rich kind of thing.

So, usually the stoichiometric ratio is maintained for auto combustion synthesis. So, metals, carbon, and hydrogen are considered reducing element with positive valence value, oxygen is an oxidizer has a negative balance value. So, these are used here and nitrogen has 0 valence from the assumption that the product species is N 2. So, that is considered as 0 for this kind of propellant chemistry calculations.

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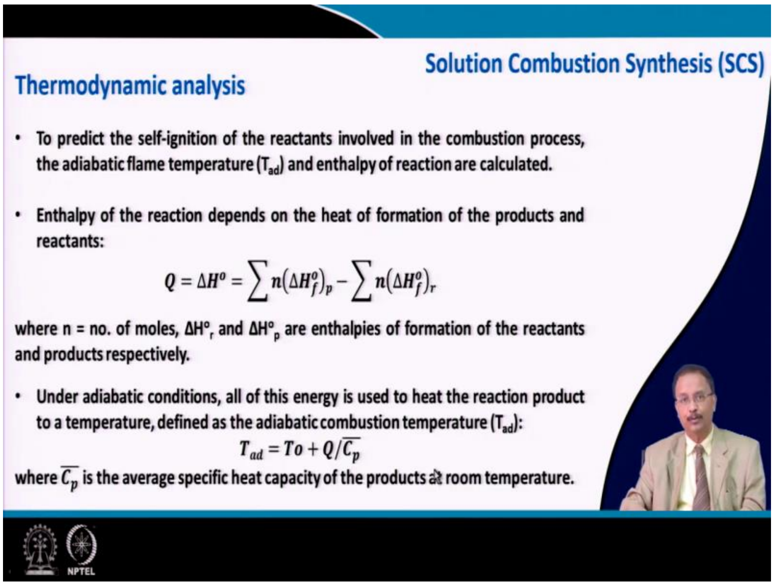

Solution Combustion Synthesis (SCS)

Thermodynamic analysis

- To predict the self-ignition of the reactants involved in the combustion process, the adiabatic flame temperature (T_{ad}) and enthalpy of reaction are calculated.
- Enthalpy of the reaction depends on the heat of formation of the products and reactants:

$$Q = \Delta H^o = \sum n(\Delta H_f^o)_p - \sum n(\Delta H_f^o)_r$$

where n = no. of moles, ΔH°_r and ΔH°_p are enthalpies of formation of the reactants and products respectively.

- Under adiabatic conditions, all of this energy is used to heat the reaction product to a temperature, defined as the adiabatic combustion temperature (T_{ad}):

$$T_{ad} = To + Q/\overline{C_p}$$

where $\overline{C_p}$ is the average specific heat capacity of the products at room temperature.

If you do a thermodynamic analysis to predict this self-ignition the reactants involved in the combustion process, the adiabatic flame temperature which can be theoretically calculated by this relation this heat is enthalpy of formation. So, this is actually the summation of all number of moles of the constituents. So, this one is the enthalpy of formation of the reactants and this one is the entropy of the formation of the product.

So, the formation enthalpy del H 0 you can estimate. Now, adiabatic condition all these energies are used to heat the reaction product eventually it is coming to the reaction product. So, this relation is valid. So, this adiabatic temperature is some initial temperature T 0 the value of Q you can get and you know the value of the specific heat at constant pressure and there are number of components average rule you can use, and this is constant T 0 is constant.

So, you can estimate what is the adiabatic temperature? So, that temperature is actually the flash point of that particular system. So, theoretically you can estimate it if you know that what are the fuels and what are the oxidants that you are using in this process.

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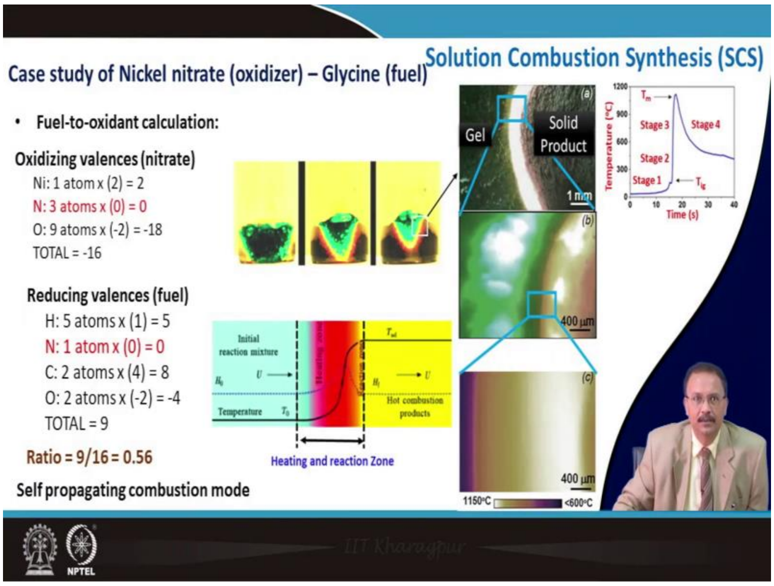

Solution Combustion Synthesis (SCS)

Case study of Nickel nitrate (oxidizer) – Glycine (fuel)

- Fuel-to-oxidant calculation:

Oxidizing valences (nitrate)
Ni: 1 atom x (2) = 2
N: 3 atoms x (0) = 0
O: 9 atoms x (-2) = -18
TOTAL = -16

Reducing valences (fuel)
H: 5 atoms x (1) = 5
N: 1 atom x (0) = 0
C: 2 atoms x (4) = 8
O: 2 atoms x (-2) = -4
TOTAL = 9

Ratio = 9/16 = 0.56

Self propagating combustion mode

So, nickel nitrate oxidizer if you use a glycine a fuel if you use this is one typical calculation which already I have described and you estimate this ratio value, and it actually operates in a self propagating mode. So, you see that this is the stage of the reaction that is going in so, you can see that this wave front this thermal front is going from your gel state to the solid product.

So, progressively it is going I already showed in one of the view graph; and this is different stage and the corresponding temperature which I told about the ignition temperature the maximum temperature and what is stage 1, stage 2, and stage 3 that has been shown pictographically, and this is basically the temperature scale.

So, from the colour you can identify that what is the typical temperature of this wave front. There is another parameter called the velocity of the combustion wave process, because actually the heat is the temperature is also changing right with the front which is proceeding. So, there is a transient of temperature also with this kind of front that is going.

So, depending on that you can have some kind of idea that what type of temperature is there during the auto combustion process, and it has its own usefulness because you know that if you have very high temperatures then the nucleation density the nuclei that is forming from your solution process the nucleation density is very high and if the nucleation density is very high the growth rate is relatively small. So, you end up with a very face pure and nano crystalline material using this kind of things.

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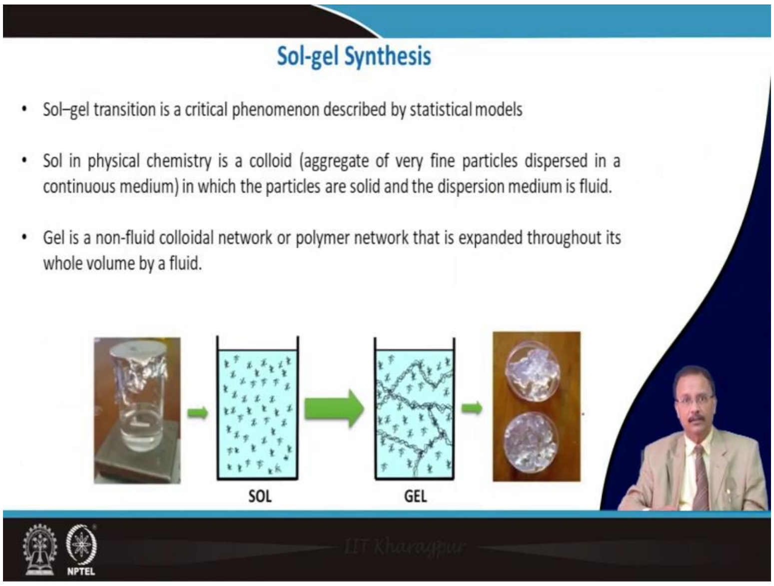

Sol-gel Synthesis

- Sol–gel transition is a critical phenomenon described by statistical models
- Sol in physical chemistry is a colloid (aggregate of very fine particles dispersed in a continuous medium) in which the particles are solid and the dispersion medium is fluid.
- Gel is a non-fluid colloidal network or polymer network that is expanded throughout its whole volume by a fluid.

So, second procedure is a sol-gel synthesis sol gel transition is critical phenomena that has defined in various statistical model. So, in physical chemistry term sol is a colloid aggregate of a very fine particle dispersed in a continuous medium in which the particles are solid and the dispersion medium is fluid. And gel is a colloidal network or a polymer network where the all the solvents are trapped.

So, initially this is a sol state it looks like this and this colloidal particles they are evenly dispersed inside this media and then sol to gel transition takes place. So, all the solvents are entrapped here and you get a gel something like this.

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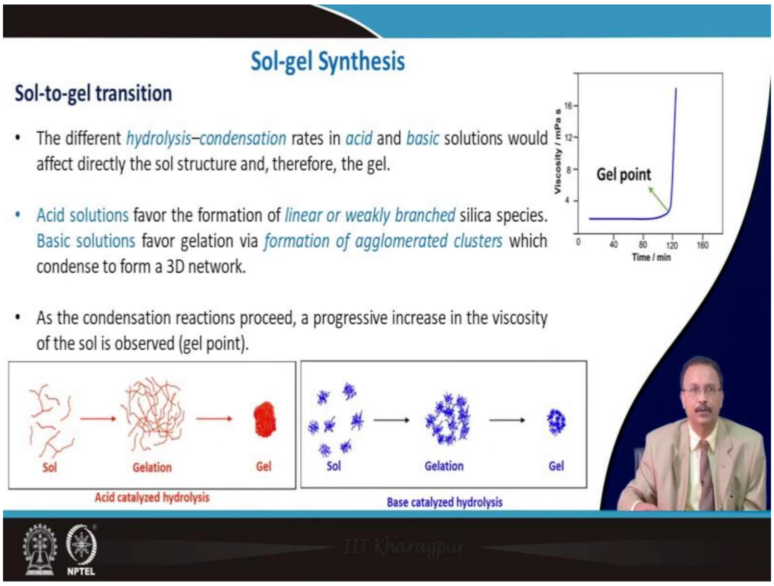

Sol-gel Synthesis

Sol-to-gel transition

- The different *hydrolysis–condensation* rates in *acid* and *basic* solutions would affect directly the sol structure and, therefore, the gel.
- Acid solutions favor the formation of *linear or weakly branched* silica species. Basic solutions favor gelation via *formation of agglomerated clusters* which condense to form a 3D network.
- As the condensation reactions proceed, a progressive increase in the viscosity of the sol is observed (gel point).

So, this is a very interesting process. So, the reaction that is operative here is the hydrolysis and condensation reaction I will be just describing in a moment, and you can see the viscosity at the gel point is suddenly shoots up. So, structurally if you can see the solids something like this and then after gelation it goes through hydrolysis and condensation reaction to form a gel something like this.

If it is acid catalyzed so, in the solution you have acid catalyzed during the salty gel transition then, it gets a linear or weakly branch kind of polymeric spaces it is shown for silica. And if it is a basic solution then it forms a highly agglomerated cluster and a 3D network. So, as the condensation reaction proceeds, then progressive increase of the viscosity takes place and the sol is transformed into a gel structure.

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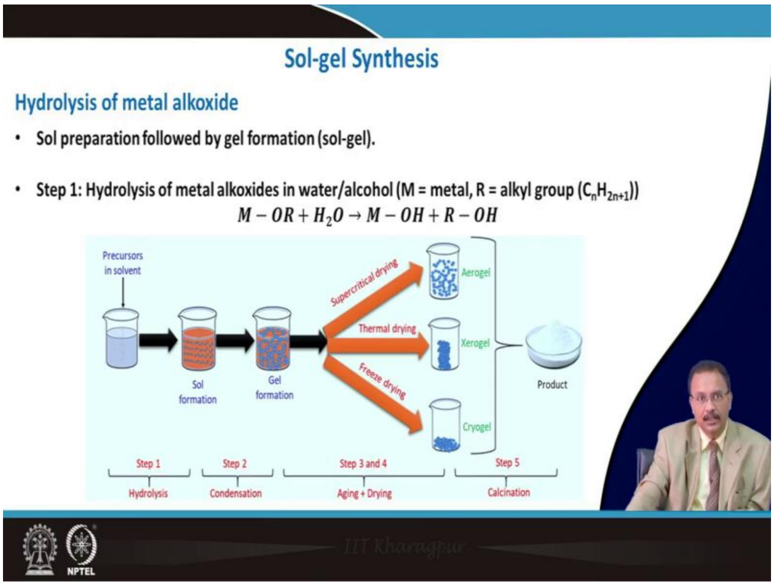

Sol-gel Synthesis

Hydrolysis of metal alkoxide

- Sol preparation followed by gel formation (sol-gel).
- Step 1: Hydrolysis of metal alkoxides in water/alcohol (M = metal, R = alkyl group (C_nH_{2n+1}))

$$M - OR + H_2O \rightarrow M - OH + R - OH$$

So, sol preparation followed by gel formation is a sol gel process, the first step is hydrolyzes and the raw material is a metal alkoxide it reacts with water to form metal hydroxide and the by product is alcohol the corresponding alcohol which formed the alkoxide.

So, here you see that this is a colloidal structure and then, it is connected porous structure which call we call gel you can do many things with a gel either you can do a supercritical drying to form a aerogel where lot of porosity is maintained; you can do a simple thermal drying to form xerogel where the porosity is lost and you get powder out of it you can do a freeze drying to form a cryogel.

So, there are 4 steps in fact, 5 steps starting from hydrolysis then condensation then aging and drying then finally, calcination is done to this final powder to make it crystalline.

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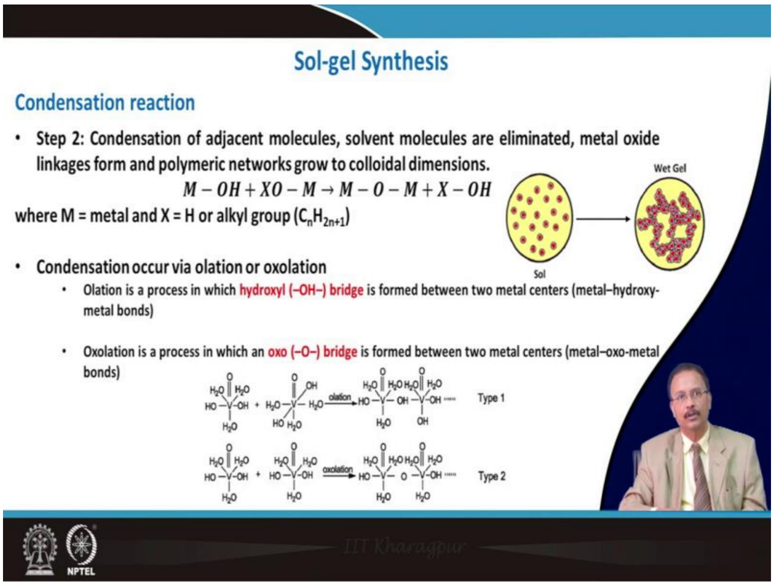

Sol-gel Synthesis

Condensation reaction

- Step 2: Condensation of adjacent molecules, solvent molecules are eliminated, metal oxide linkages form and polymeric networks grow to colloidal dimensions.

$$M - OH + XO - M \rightarrow M - O - M + X - OH$$

where M = metal and X = H or alkyl group (C_nH_{2n+1})

- Condensation occur via olation or oxolation
  - Olation is a process in which hydroxyl (–OH–) bridge is formed between two metal centers (metal–hydroxy-metal bonds)
  - Oxolation is a process in which an oxo (–O–) bridge is formed between two metal centers (metal–oxo-metal bonds)

So, after hydrolysis where is the by-product is an alcohol then you go for condensation reaction where this hydrolyzed product reacts with each other. So, here you can see one hydroxide it reacts with another metal hydroxide group or it could be other type of group as well, either it could be h or it could be any other alkyl group.

So, eventually it will form a metal oxygen type of bond and then X OH will be the product which is coming out. So, condensation can occur either by olation or oxolation process, if it is a olation process then the bonding between metal two metals as you can see here two metal here vanadium is shown. So, vanadium is connected with hydroxyl ion; and if there is oxygen in between these two metal after condensation then we call it is a oxolation process.

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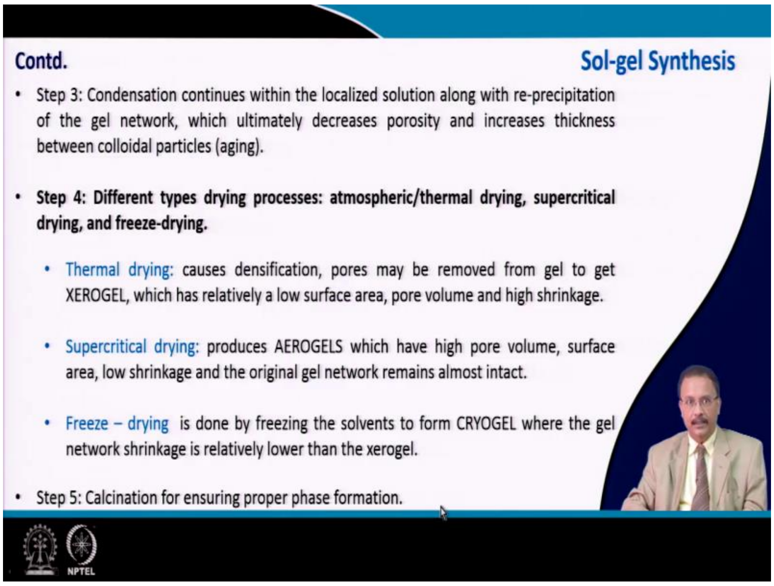

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Sol-gel Synthesis

- Step 3: Condensation continues within the localized solution along with re-precipitation of the gel network, which ultimately decreases porosity and increases thickness between colloidal particles (aging).
- **Step 4: Different types drying processes: atmospheric/thermal drying, supercritical drying, and freeze-drying.**
  - Thermal drying: causes densification, pores may be removed from gel to get XEROGEL, which has relatively a low surface area, pore volume and high shrinkage.
  - Supercritical drying: produces AEROGELS which have high pore volume, surface area, low shrinkage and the original gel network remains almost intact.
  - Freeze – drying is done by freezing the solvents to form CRYOGEL where the gel network shrinkage is relatively lower than the xerogel.
- Step 5: Calcination for ensuring proper phase formation.

So, these two types of condensation is valid usually happens for sol gel processing. Now, condensation continues within the localized solution along with re precipitation of the gel network and ultimately it is a porous solid which is happening. And as I have said then different types of drying process you can adopt to make functional article and in fact, when the condensation reaction is going on when the viscosity is increasing. So, that step taking that sol still it is in a liquid state you can deposit a film.

So, you can make a 2D kind of structure. When the viscosity is going up at that stage you can put a simple glass rod inside the beaker and draw fibres so it is spinnable. So, you can make a 1 dimensional structure then, you can do simple thermal drying so, that the drying shrinkage will take place and due to the capillary action it will all get scrambled and you get powder out of it.

Of course, you will have to calcify in it to get a phase I already I will talk in the next class about the difference in calcination followed by sintering in more details, but thermal drying is one of them. You can do supercritical drying where you maintain the vapour pressure outside.

So, that there is no drying stress involved and you get a nice monolithic structure out of this. So, that is a 3D structure, you can do freeze drying the freezing the solvent to form a cryogel where the gel network shrinkage is relatively low then does the aerogel. So, that is also possible.

So, the shape flexibility is tremendous for sol gel processing, you can prepare nano powder you can prepare 1 dimensional fibre you can prepare two dimensional thin film you can prepare monolithic structure monolith brass is possible to make. So, it is a very very powerful processing route as far as the ceramic material is concerned.

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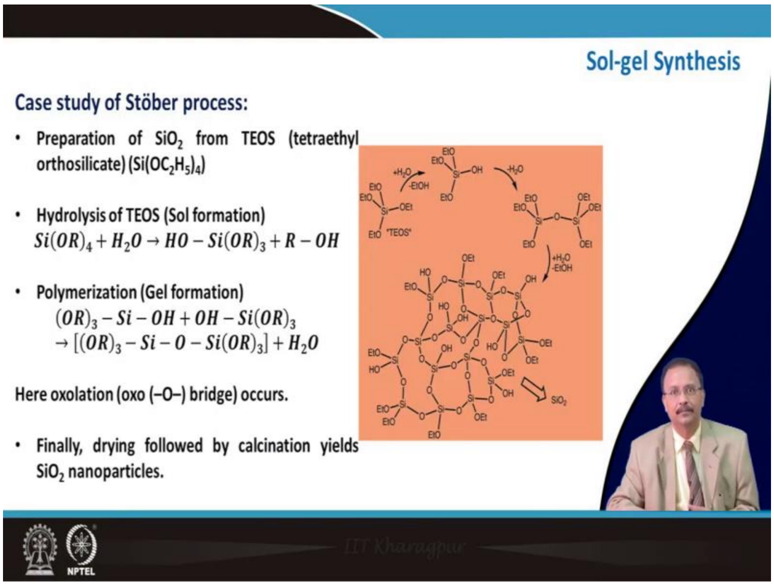

Sol-gel Synthesis

Case study of Stöber process:

- Preparation of SiO_2 from TEOS (tetraethyl orthosilicate) ($Si(OC_2H_5)_4$)
- Hydrolysis of TEOS (Sol formation)
  $Si(OR)_4 + H_2O \rightarrow HO - Si(OR)_3 + R - OH$
- Polymerization (Gel formation)
  $(OR)_3 - Si - OH + OH - Si(OR)_3 \rightarrow [(OR)_3 - Si - O - Si(OR)_3] + H_2O$

Here oxolation (oxo (–O–) bridge) occurs.

- Finally, drying followed by calcination yields SiO_2 nanoparticles.

So, Stober process to make silicon dioxide exactly goes in the same way first the hydrolysis is done in TEOS tetraethyl orthosilicate to form Si OR structure it is partially hydrolyzed then, it is polymerized condensation reaction takes place to form this Sio Si kind of bond and this is by the way one example of oxolation bridge and finally, you can dry and followed by calcination to form Sio 2 nanoparticles by this Stober process.

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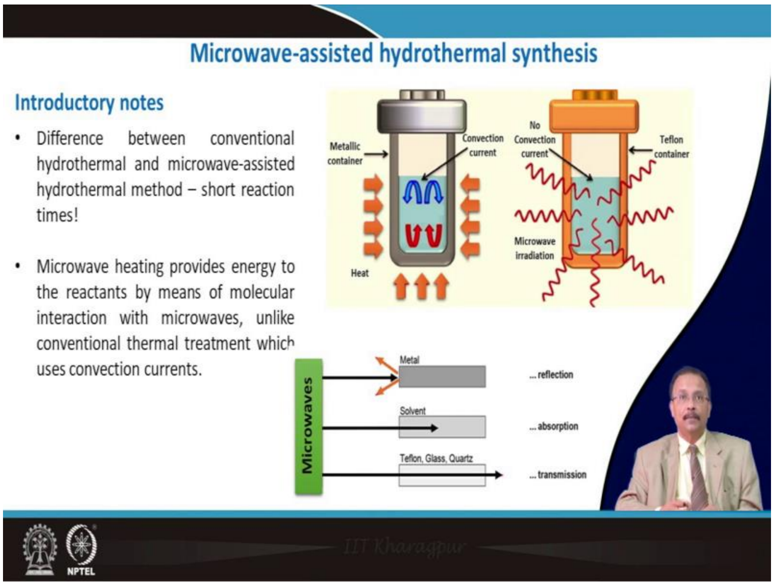

Microwave-assisted hydrothermal synthesis

Introductory notes

- Difference between conventional hydrothermal and microwave-assisted hydrothermal method – short reaction times!
- Microwave heating provides energy to the reactants by means of molecular interaction with microwaves, unlike conventional thermal treatment which uses convection currents.

The third important aspect which I would like to introduce is microwave assisted hydrothermal synthesis you know that if you process the material in a hydrothermal condition.

So, additional one thing you are using you are using pressure in order to transform the phase; phase formation is activated here and simultaneously you apply microwaves. So, difference between the conventional hydrothermal and microwave assisted hydrothermal lies, that in case of microwave treatment along with the hydro thermal use is that the reaction time is dramatically dropped down.

So, if it is for conventional hydrothermal if it is in 3 hours it takes place maybe for microwave hydrothermal it will take mostly about 2 minutes. So, that is the advantage and microwave heating provides energy to the reactant as you know by means of the molecular interaction with the microwave unlike conventional thermal treatment which use convection current. So, that is the one thing.

So, there are many materials which are not particularly suitable for microwave for example, metal is not suitable it will reflect microwave some of the solvent they absorb microwave and some of the material they are transparent to microwave. So, that thing is important.

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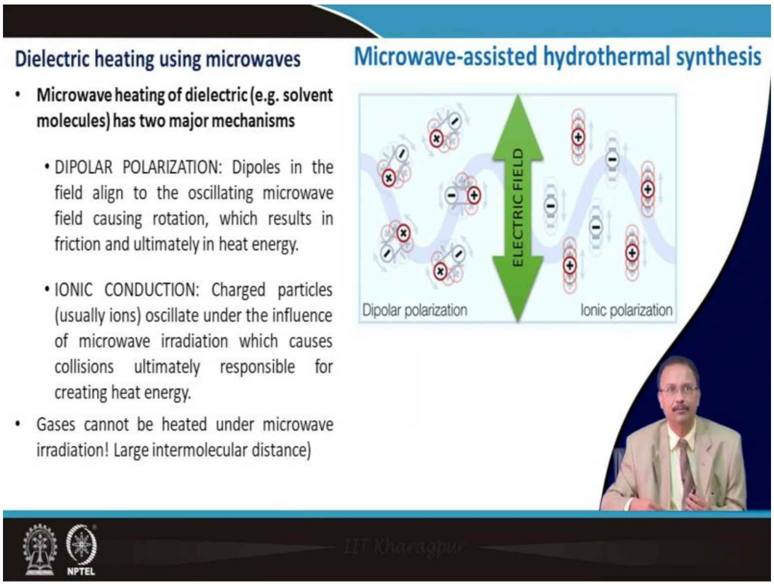

Dielectric heating using microwaves

- **Microwave heating of dielectric (e.g. solvent molecules) has two major mechanisms**
  - DIPOLAR POLARIZATION: Dipoles in the field align to the oscillating microwave field causing rotation, which results in friction and ultimately in heat energy.
  - IONIC CONDUCTION: Charged particles (usually ions) oscillate under the influence of microwave irradiation which causes collisions ultimately responsible for creating heat energy.
- Gases cannot be heated under microwave irradiation! Large intermolecular distance)

Microwave-assisted hydrothermal synthesis

So, what we are interested in is the dielectric heating using microwave. So, microwave heating of dielectric usually the dielectric is in the form of a solvent molecule this is having to mechanism and part of it you know because, already we talked about the dielectric material you know different types of polarization that is undergone in the dielectric material. So, dipolar polarization dipoles in the field aligned this aligned to the oscillating microwave field causing a rotation and they are in close proximity.

So, when it rotates it results in friction and ultimately it exchange in heat. In case of ionic conduction charged particles oscillates under the influence of the microwave irradiation, which causes collision again and also ultimately responsible for the generation of heat energy. And of course, gas cannot be used heated by microwave because of their large intermolecular distances.

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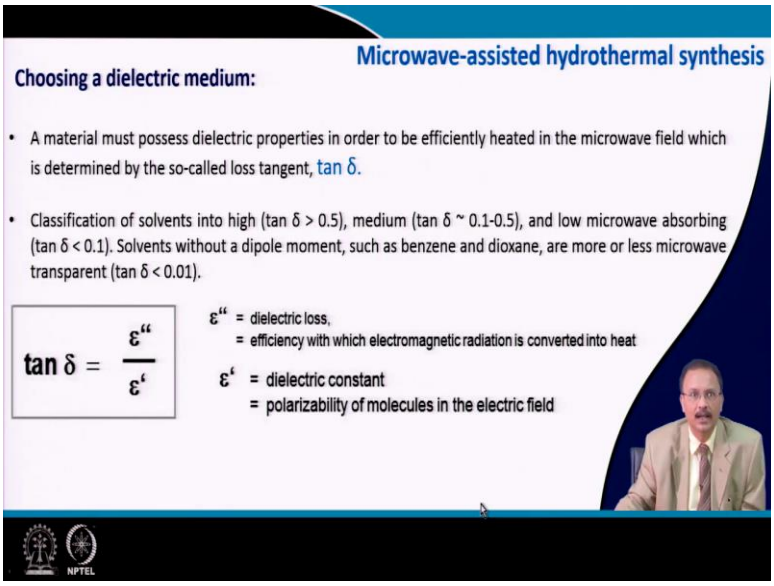

Microwave-assisted hydrothermal synthesis

Choosing a dielectric medium:

- A material must possess dielectric properties in order to be efficiently heated in the microwave field which is determined by the so-called loss tangent, tan δ.
- Classification of solvents into high ($\tan\delta > 0.5$), medium ($\tan\delta \sim 0.1\text{-}0.5$), and low microwave absorbing ($\tan\delta < 0.1$). Solvents without a dipole moment, such as benzene and dioxane, are more or less microwave transparent ($\tan\delta < 0.01$).

$$\boxed{\tan\delta = \frac{\varepsilon''}{\varepsilon'}}$$

ε'' = dielectric loss,
= efficiency with which electromagnetic radiation is converted into heat

ε' = dielectric constant
= polarizability of molecules in the electric field

So, material mass pauses dielectric properties in order to be efficiently heated for microwave field that is determined by the so called loss tangent is responsible. So, the solvents you can classify which are having high loss tangent medium loss tangent and low microwave absorbing material, which is having lower or lowest tan delta value typically less than 0.1.

And solvents which do not have dipole moment they are more or less microwave transparent and typical dielectric loss tangent is less than 0.01 in fact, and tan delta is defined is the from the complex permittivity configuration you can have a loss tangent by imaginary and real part ratio.

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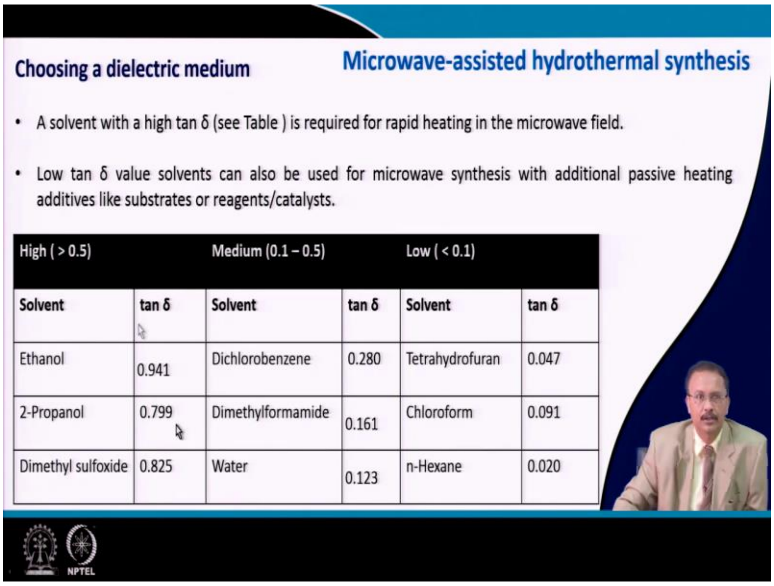

Choosing a dielectric medium

Microwave-assisted hydrothermal synthesis

- A solvent with a high tan δ (see Table) is required for rapid heating in the microwave field.
- Low tan δ value solvents can also be used for microwave synthesis with additional passive heating additives like substrates or reagents/catalysts.

High (> 0.5)		Medium (0.1 – 0.5)		Low (< 0.1)	
Solvent	tan δ	Solvent	tan δ	Solvent	tan δ
Ethanol	0.941	Dichlorobenzene	0.280	Tetrahydrofuran	0.047
2-Propanol	0.799	Dimethylformamide	0.161	Chloroform	0.091
Dimethyl sulfoxide	0.825	Water	0.123	n-Hexane	0.020

So, the solvent which are good for this purpose is ethanol, 2- propanol, and dimethyl sulfoxide and there are some solvent which are having medium range of this dielectric constant and typical tan delta you can see from this table.

So, the low tangent value solvents can also be used for microwave synthesis with additional passive heating additives like, substrate some kind of substrate can be used and or reagents or catalyst etcetera also can be used to make use of that.

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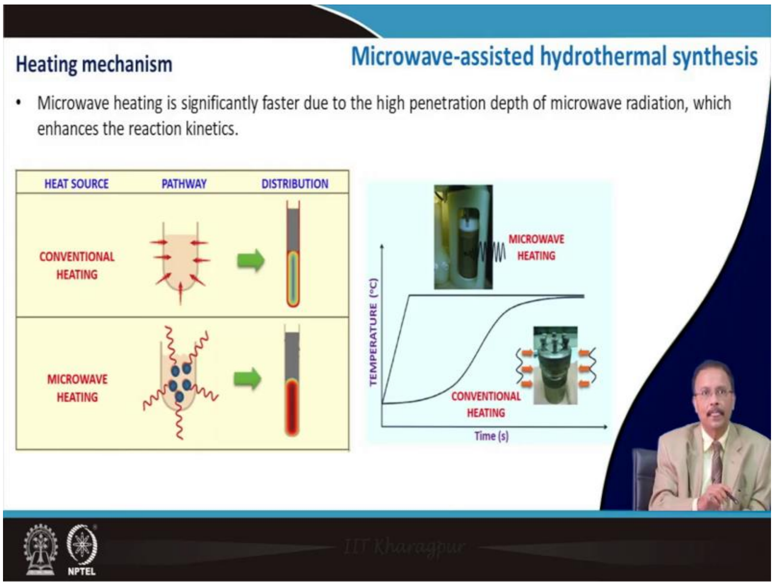

Heating mechanism

Microwave-assisted hydrothermal synthesis

- Microwave heating is significantly faster due to the high penetration depth of microwave radiation, which enhances the reaction kinetics.

So, here is the heating mechanism you can see here the heat is mostly confined in case of conventional heating the container actually takes most of the heat and it is transmitted to a lesser extent because, the crucible or whatever you use it they are thermally non conductive a low thermal conductivity is there.

But in case of microwave heating you see that instantly your material gets the heat. So, it has if it is a Teflon container it is transparent to the microwave heat and all the heat is getting absorbed by the material itself. And that is reflected in the temperature time profile as you can see or within no time it attends this temperature and remain stable, but here it takes long time to achieve for conventional heating this kind of temperature.

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Study Materials

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So, in this particular lecture I introduced three important processing route of ceramic and ceramic with shape flexibility. So, you can have the shape flexible ceramic also made using this type of processing route, but there is no specific study material this first book by K.C. Patil that will give you a lot of information about the combustion synthesis of ceramic and related materials, but for the other things specifically you will have to read this literature which are pretty comprehensive and that will give you a basic idea about the concept that I have covered in the class.

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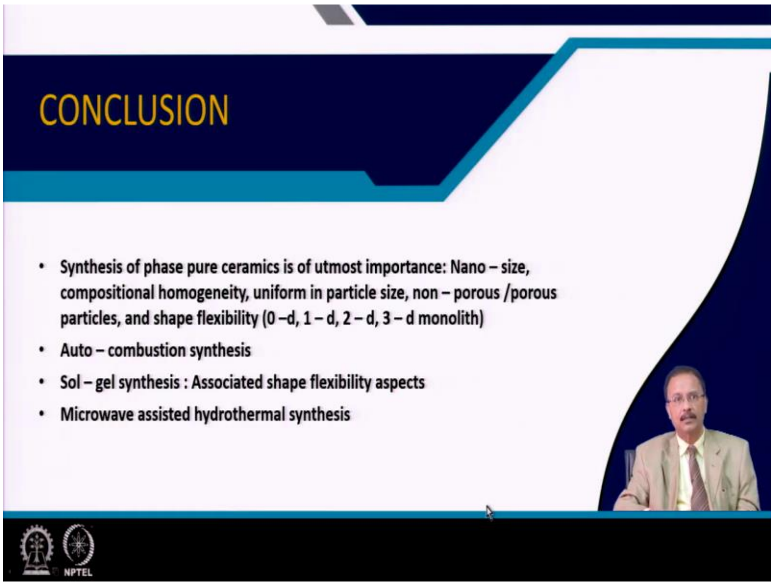

CONCLUSION

- Synthesis of phase pure ceramics is of utmost importance: Nano – size, compositional homogeneity, uniform in particle size, non – porous /porous particles, and shape flexibility (0 –d, 1 – d, 2 – d, 3 – d monolith)
- Auto – combustion synthesis
- Sol – gel synthesis : Associated shape flexibility aspects
- Microwave assisted hydrothermal synthesis

So, synthesis of phase pure ceramics is of utmost importance you will see that how these materials are sintered for a definitive shape in my next lecture, nano-size, compositional homogeneity, uniform in particle size, non porous or porous type of particle, and shape flexible material like 0-d, 1- d, 2- d, 3- d they all can be made by sol gel process or by the other two process I have described in this class.

So, initially we started with auto combustion then, we have introduced sol gel synthesis and associated shape flexibility issues and finally, microwave assisted hydrothermal synthesis.

Thank you for your attention.