Materials Chemistry

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Module - 1
Lecture - 6
Preparative Routes Un Conventional Combustion Technique

In today’s lecture, we are going to cover one of the very important approaches to material synthesis. Because this is both having the capability of making high temperature materials in a conventional way, but yet it involves typical wet chemistry route. As you have seen in the previous lecture, there are portfolios of chemical synthesis available for making wide range of molecules. And in today’s lecture, I am going to single out one of the potential preparative route, which has been exploited by many, many groups. And still this particular approach has not been aborted by the researchers, because there is lot more novel insides coming out of this particular process called combustion synthesis.

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Combustion Synthesis of High Temperature Materials

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And this combustion synthesis is specially year marked for high temperature materials, because the energetic statics involved in this combustion route is actually coming from a chemical synthesis, which achieves very high temperature. And therefore, it is possible for us to realize materials, which are other ways prepared only at very high temperatures. Now, when I talk about high temperature materials, these are not the usual chemical compounds that we prepare in lab scale with simple hydrolysis or reflexing, because this is the typical chemistry routes, which is commonly seen in any wet chemistry lab. But, what we are seeing here is, we are starting with the wet chemical approach, but we are realizing very high temperatures, as you would see from this cartoon. So, I will just take you through this course of combustion synthesis and tell you, what is the principle behind this approach and what are the advantages and what is the chemistry that we learnt from this, and where do we go from here.

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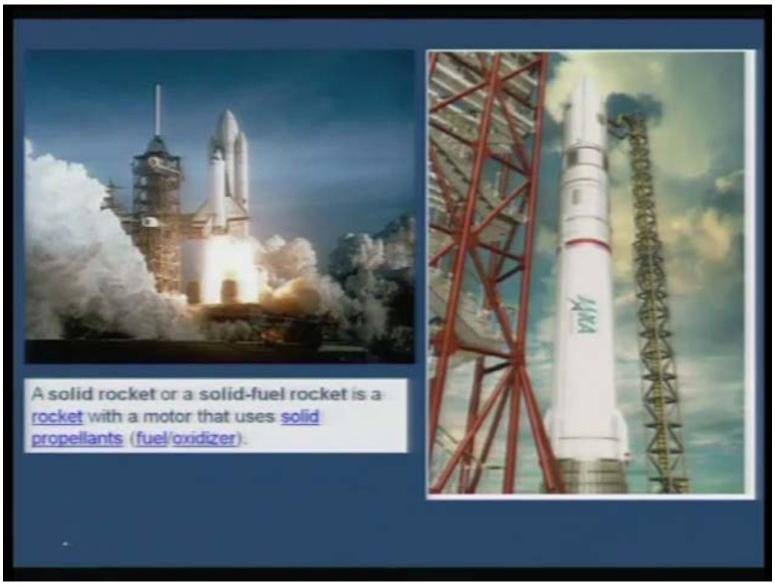

A solid rocket or a solid-fuel rocket is a rocket with a motor that uses solid propellants (fuel/oxidizer).

As you would know, when you talk about a high temperature and when you talk about combustion, the first thing that comes to your mind is about launching of satellites or spacecrafts. Now, what you see here is a launching pad and lot of energy is released to take this space shuttle out of the earth orbit and this is again, what you see in this right side, it is a Japanese space shuttle which is ready for launching. But, the essence of this whole pay load, to go out of Earth’s orbit is dependent on, whether it is a solid rocket or liquid rocket.

Meaning, a solid rocket consists of a solid fuel as the combustion material and as a result, it can carry the whole space shuttle and put it in the outer orbit. Now, what is important in this solid rocket is the solid propellants and solid propellants are nothing but a mixture of fuel and oxidizer and this is the essence of any space applications. Now, the beauty of it is, if we understand the principle that governs this launching then we will know how we can translate that for material synthesis. So, in the first few slides, I will take you through the essence of what a propellant is, what it means to have a fuel and a oxidizer; and how we can convert this enormous energy into a controlled energy for making materials.

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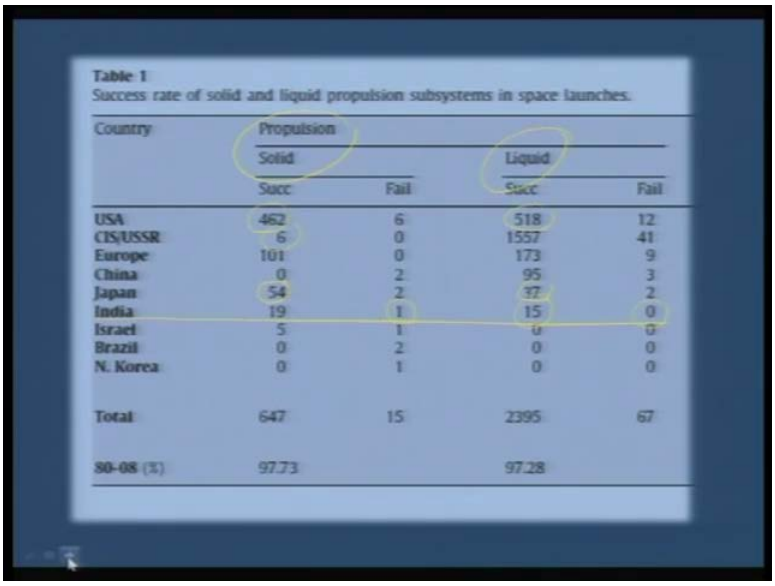

Table 1
Success rate of solid and liquid propulsion subsystems in space launches.

Country	Propulsion			
	Solid		Liquid	
	Succ	Fail	Succ	Fail
USA	462	6	518	12
CIS/USSR	6	0	1557	41
Europe	101	0	173	9
China	0	2	95	3
Japan	54	2	37	2
India	19	1	15	0
Israel	5	1	0	0
Brazil	0	2	0	0
N. Korea	0	1	0	0
Total	647	15	2395	67
80-08 (%)	97.73		97.28	

Now, before I go into the details, I just single out some of the issues related to propulsion, propulsion can be either solid propulsion or liquid propulsion. And many countries have been using this systematically to put there space shuttle in the outer orbit. Now, if you look at India for example, India has attempted many launching space launches, of which they have used 19 with solid propellant and 15 with liquid propellant. And out of 19, they have only failed 1s and with liquid propellant, it is 0 failure and they have done it quite systematically with both solid and liquid propellants.

If you look at USA and USSR, you can see USSR has heavily relayed on liquid propulsion for the space launches, whereas US has been using both the techniques very judiciously. Japan for example, if you see, they have relayed more on solid propellants than the liquid propellants. Now, what is advantage and why these propellants can become divisive in the space launches.

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You can see that, the energy that this propellants carry is very vital and as a course of time, the time evolution of energy is plotted here as early as minus 500 a d or 3500 b c, people were using bow and arrow. Now, if you keep on looking how the energy situation has surfaced, you can see that, from there we have graduated to black powder then to ammonium nitride then potassium perchlorate and silver fulminate, nitro starch so on.

And somewhere here in this arena, where we are now living, we have now transposed mostly to RDX which is nothing but of composite, RDX, Cl20, iron, all these are basically a polymer composite based propellants, which are being used. Now, these are hybrid varieties of propellants that are used in space launches, now these are also used for several difference purposes.

Now, to take a simple definition of a propellant, it is a chemical mixture that is burnt in the presence of air to produce thrust in rockets and it consists of a fuel and a oxidizer. A fuel is a substance, which burns when combined with oxygen, producing gas for propulsion. So, when you need oxygen to burn a fuel then you resort for a material, which can give oxygen within, rather than take oxygen from outside, that is why the concept of a oxidizer comes.

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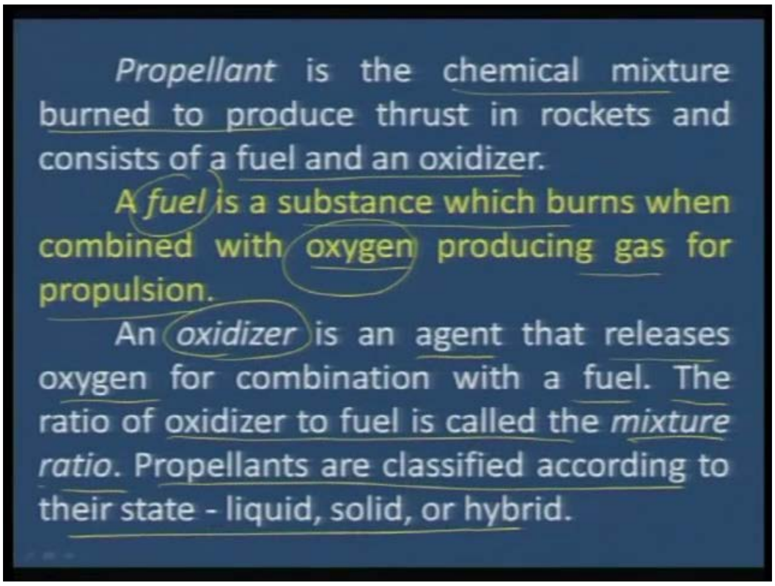

Propellant is the chemical mixture burned to produce thrust in rockets and consists of a fuel and an oxidizer.

A *fuel* is a substance which burns when combined with oxygen producing gas for propulsion.

An *oxidizer* is an agent that releases oxygen for combination with a fuel. The ratio of oxidizer to fuel is called the *mixture ratio*. Propellants are classified according to their state - liquid, solid, or hybrid.

A oxidizer is one which releases enough oxygen for the fuel to burn, so if you have proper chemistry between this fuel and oxidizer, then you can release enormous energy, therefore, you can get the thrust that you want. So, an oxidizer is an agent that releases oxygen for combination with a fuel, the ratio of oxidizer to fuel is called the mixture ratio and in propellant chemistry, it is usually called as mixture ratio. Now, we use this word more frequently and call this oxidizer to fuel ratio or O by F ratio, propellants actually are classified in different ways they need not be solid always.

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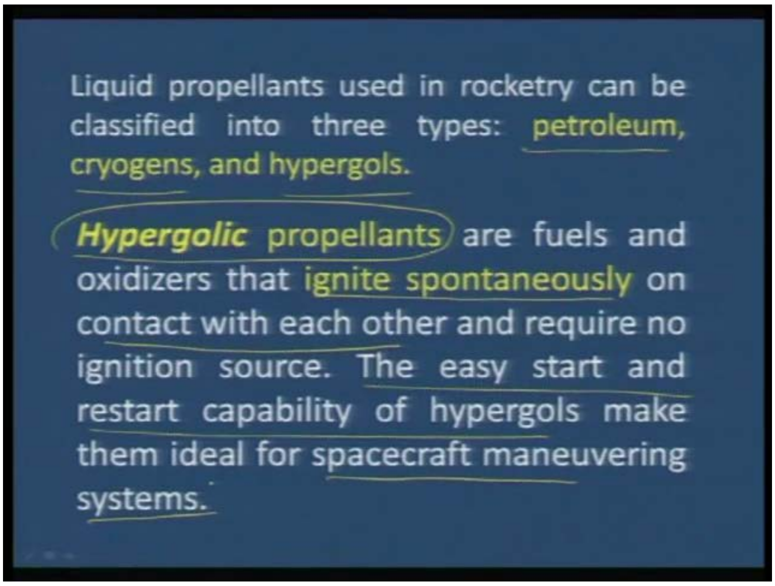

Liquid propellants used in rocketry can be classified into three types: petroleum, cryogens, and hypergols.

Hypergolic propellants are fuels and oxidizers that ignite spontaneously on contact with each other and require no ignition source. The easy start and restart capability of hypergols make them ideal for spacecraft maneuvering systems.

We can look at some of the definitions for example; liquid propellants used in rocket applications can be classified into three, petroleum, cryogens and hypergols. What is important is, this hypergolic propellants, because they are fuels and oxidizers that ignite spontaneously on contact with each other. When you just bring both the oxidizer and fuel in physical contact, immediately they become hypergolic and they get ignited, that is the definition of hypergolic propellant. They are easy and they starting capability of the hypergols make them really ideal for spacecraft maneuvering. Therefore, hypergolic propellants are much more used now a day than even petroleum and other cryogens.

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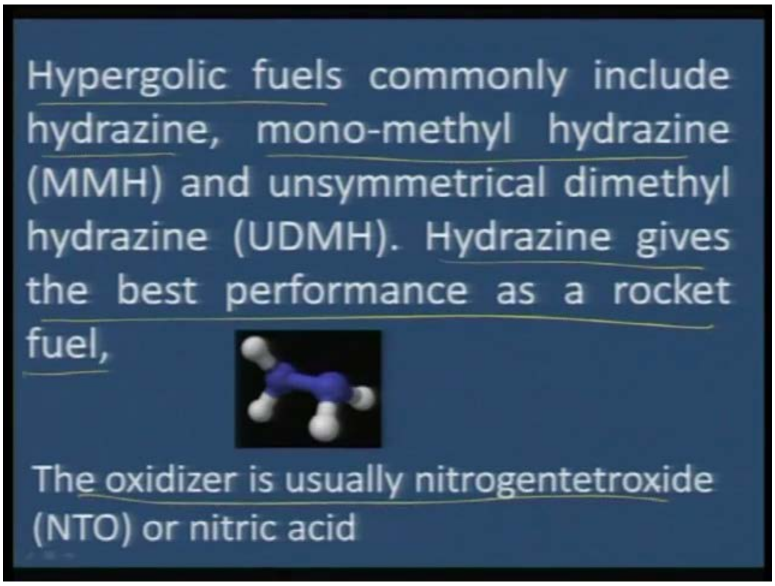

Hypergolic fuels commonly include hydrazine, mono-methyl hydrazine (MMH) and unsymmetrical dimethyl hydrazine (UDMH). Hydrazine gives the best performance as a rocket fuel,

The oxidizer is usually nitrogentetroxide (NTO) or nitric acid

Now, what are these hypergolic fuels, hypergolic fuels are commonly known as a hydrazine or mono methyl hydrazine, popularly abbreviated as MMH in rocket technology and hydrazine gives the best performance as a rocket fuel. As you know, hydrazine is nothing but N 2 H 4 and this N 2 H 4 can be substituted with methyl group. So, they are called as unsymmertrical dimethyl hydrazine, where two methyls can be substituted here and two hydrazines can be here or it can be a mono methyl, both are found to be extremely useful hypergolic fuels. The oxidizer in this case is actually nitrogen tetroxide or nitric acid, so just bring hydrazine and in contact with nitrogen tetroxide, immediately they burn to give the necessary heat.

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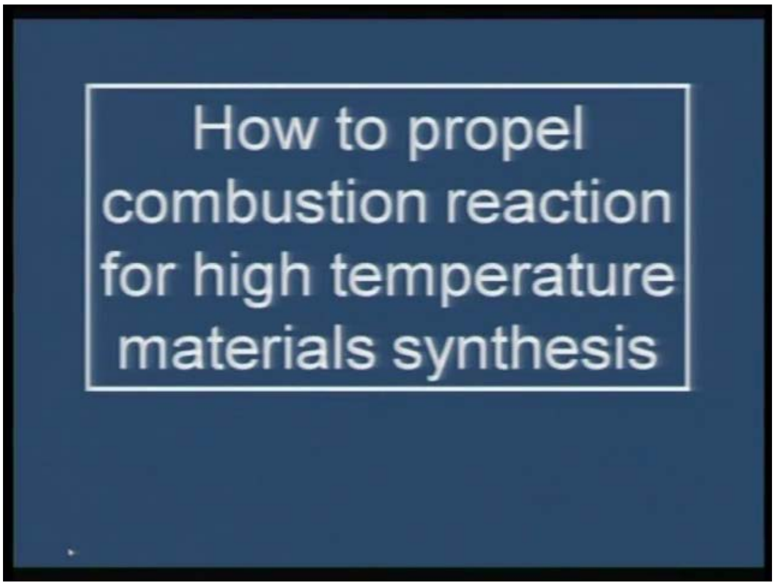

How to propel combustion reaction for high temperature materials synthesis

Now, how do propulsion occurs or how can be propel this combustion principle to make material synthesis is our question, because the energy that is releases uncontrollable, you just cannot fashion it, you can just put a big mass into the space. So, this has to be retained or confined into a lab situation is a big challenge, therefore how do we propel this combustion for material synthesis is a million dollar question, and we will look at it.

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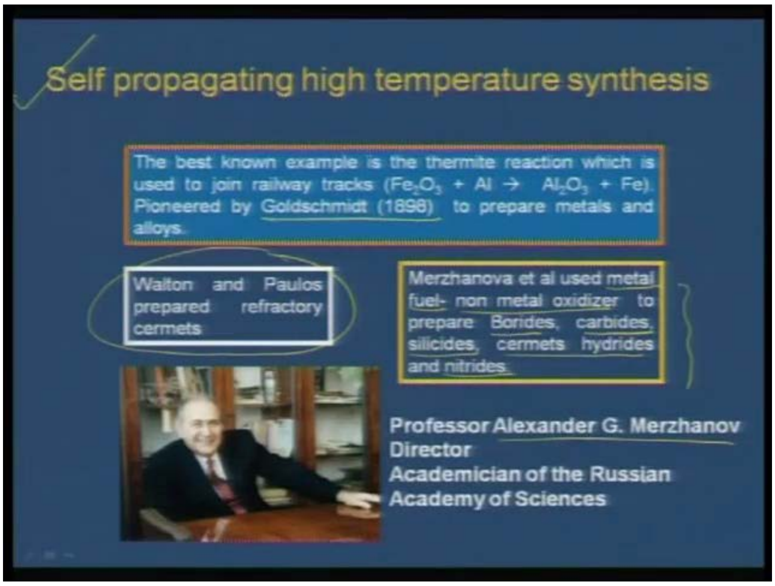

Self propagating high temperature synthesis

The best known example is the thermite reaction which is used to join railway tracks (Fe_2O_3 + Al → Al_2O_3 + Fe). Pioneered by Goldschmidt (1898) to prepare metals and alloys.

Walton and Paulos prepared refractory cermets

Merzhanova et al used metal fuel- non metal oxidizer to prepare Borides, carbides, silicides, cermets hydrides and nitrides.

Professor Alexander G. Merzhanov
Director
Academician of the Russian
Academy of Sciences

It is not new parse, because the principle of combustion for high temperature synthesis has been there for many years. And the most important one is the thermite reaction, which is nothing but burning aluminum with iron oxide. And if you burn, it converts into aluminum oxide and iron, this is called a thermite process, which is usually used in joining or welding railway tracks and this was pioneered by Goldschmidt in 1898 to prepare metals and alloys.

Therefore, this is not a new phenomena to make materials, but this is often categorized as self propagating high temperature synthesis, because this has been used to achieve very high energy to prepare new materials. Walton and Paulos have prepared refractory cermets, which are usually alumina based zirconia based ones, which are doped with some 3 D transition metals. But, the concept of self propagating high temperature synthesis was actually exploited more by Merzhanov.

So, I would like to show his portrait, because he was one of the pioneers in this field, who brought in lot of fundamental applications of this SHS technology for applications. Merzhanov have used a metal fuel and a non metal oxidizer to prepare borides, carbides, silicides, cermets hydrides and nitrides. If you want to prepare nitrides then you need to mix it with some nitrogen producing starting material. If you want to prepared borides, you have to mix it with the boron, elemental boron or diborane, or if you want to prepare carbides then you have to mix it with the reactive carbon powder.

So, when you try to use metal and a non oxidizer as a fuel, you make a mixture of it and then you spark it then you can actually initiate a very high temperature reaction, and thereby you can get all this high temperature ceramics. Otherwise, it is impossible for you to prepare in a lab scale or we need to use a conventional furnace, which is operated at 20 to 100 degree C, which can be used for making borides or carbides.

So, this is a very versatile process and in the next slide, I will show you small clip of a movie, which shows how this high temperature material works. And professor Merzhanov is actually the academician of the Russian academy of science, and he has authored many journals based on SHS technology. In fact, this process is so very popular, that there is a separate journal called international journal for self propagating high temperature synthesis, and it has been there in the shelf for more than 20 years and still many novel approaches are being made using this technology.

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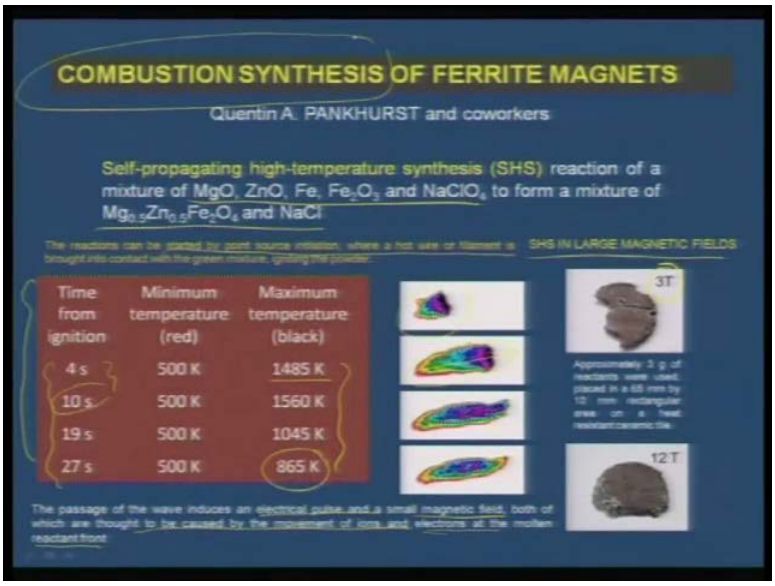

COMBUSTION SYNTHESIS OF FERRITE MAGNETS

Quentin A. PANKHURST and coworkers

Self-propagating high-temperature synthesis (SHS) reaction of a mixture of MgO, ZnO, Fe, Fe_2O_3 and $NaClO_4$ to form a mixture of $Mg_{0.5}Zn_{0.5}Fe_2O_4$ and NaCl

The reactions can be started by point source initiation, where a hot wire or filament is brought into contact with the green mixture, igniting the powder.

Time from ignition	Minimum temperature (red)	Maximum temperature (black)
4 s	500 K	1485 K
10 s	500 K	1560 K
19 s	500 K	1045 K
27 s	500 K	865 K

SHS IN LARGE MAGNETIC FIELDS

Approximately 3 g of reactants were used, placed in a 65 mm by 12 mm rectangular area on a heat resistant ceramic tile.

The passage of the wave induces an electrical pulse and a small magnetic field, both of which are thought to be caused by the movement of ions and electrons at the molten reactant front

This technology also has been used to make ferrites, ferrite magnates, useful for industrial application. Pankhurst and co-workers, they have used this process to prepare this sort of ferrite material. What you do here is, take all the starting materials together like magnesium oxide, zinc oxide, iron, Fe2O3 and potassium perchlorate. Potassium perchlorate in this case acts as a oxidizer and they form a mixture together, and the mixture is actually burnt.

The reactions can be started by point source initiation, where a hot wire or filament is actually touching a palate and that will initiate a very high temperature reaction, and it can give you the final product, which is nothing but a ferrite magnet. Now, the advantage here is, if you want you can make it as a disk or a rod or a spiral ring, anything you can make provided, you make the shape of it in the precursor and then you try to ignite and start this SHS reaction.

As you can see, these are very fast reactions, therefore in 4 seconds you can actually reach temperatures up to 1400 Kelvin. And you can see, the span of reaction is not lasting for more than a minute, it is always less than a minute, therefore it is a rapid self propagating one. So, just initiate that is enough, you do not need to run through or sustain the reaction by heating it, that is why it is called self propagating, because once you initiate, it can propagate on it is own and it can complete the reaction.

And you can see from this profile by 27 seconds, almost the combustion is over and it is cooling back, this is the IR image, infrared image of this zone which clearly shows, how the temperature propagation is occurring from a low temperature to high temperature. And against each time scale, 4 second, this is how the propagation is 10 second, this is how propagation is… So, between 4 to 10 second, it almost hits the maximum temperature and then it starts cooling down.

Now, this is being conducted also in the absence of field and in the presence of the field, for example the same combustion has been prepared in using a external magnetic field of say, 3 Tesla. So, I have included this point SHS in large magnetic fields, so if you take the specimen at 3 Tesla and if you rake the specimen at 12 Tesla, you see a much harder and much sintered magnet is formed at a higher temperature. Why, because the passage of the wave induces an electrical pulse and a small magnetic field, both of which are thought to be caused by the moments of ions and electrons at the molten reactant front.

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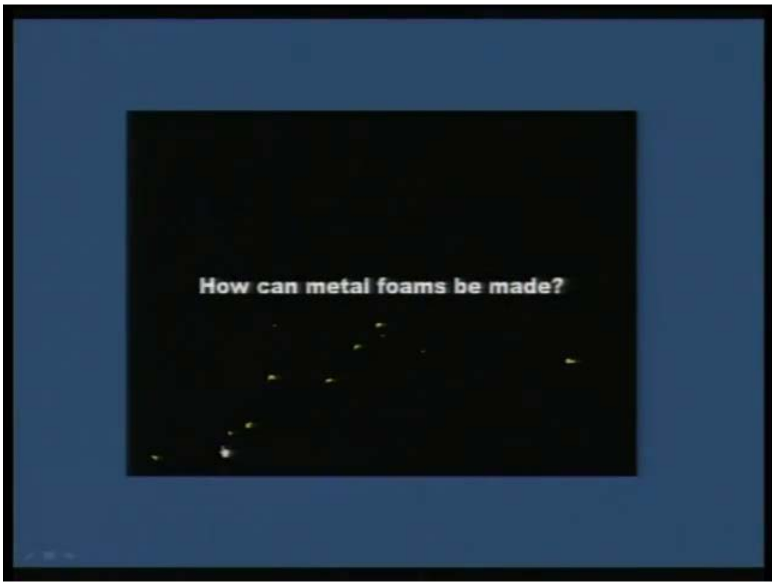

How can metal foams be made?

And because it generates a electrical pulse and a magnetic field then the external magnetic field can also propagate a wave, which can initiate in the soldering or in the sintering process of this final product. So, if you are actually looking for making fine finishing of the preferred form or shape of your ferrite then application of a external magnetic field also influences making such materials. So, this has been widely exploited as SHS, but the word combustion has not been used till then rather it has always been referred to as self-propagating high temperature synthesis.

This is a movie that I want to show just to give you an idea, what this self-propagation is and how much of energy is involved and how we can initiate such a reaction, if you can carefully look at the animation. So, this clip will help us realize, how the combustion can be initiated and what sort of temperatures can be achieved in short time scale and let us look at this movie.

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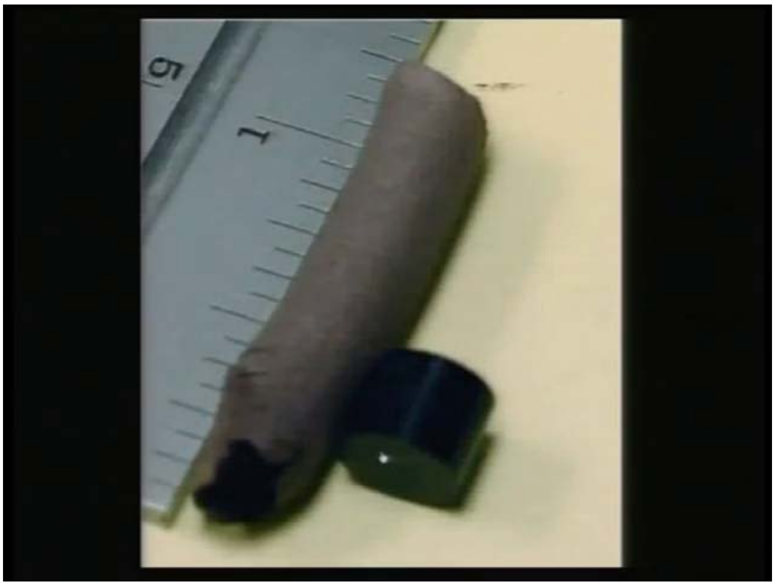

So, what we need is a heat oxidizer and a fuel to initiate such a reaction, so the metal complexes we can form are of this nature and this is made into a pallet like this. In such a short span, in less than 2 minutes or so it is possible for us to realize a product. As you had seen in the last phase, a small pallet can actually give you almost 10 centimeter long metal foam and this is one of the way, this the high temperature reaction can be achieved.

But also, it might look scary, because it is actually kept in the confined medium, where it organize surface through this reaction, because you do not want a oxide. In case of metal foams, you have to supply argon atmosphere, but can we make this bit more easier is the question is a open challenge. And can we extend this sort of high temperature reaction for many of the other materials, other than merely metals and alloys that is the question that we need to understand.

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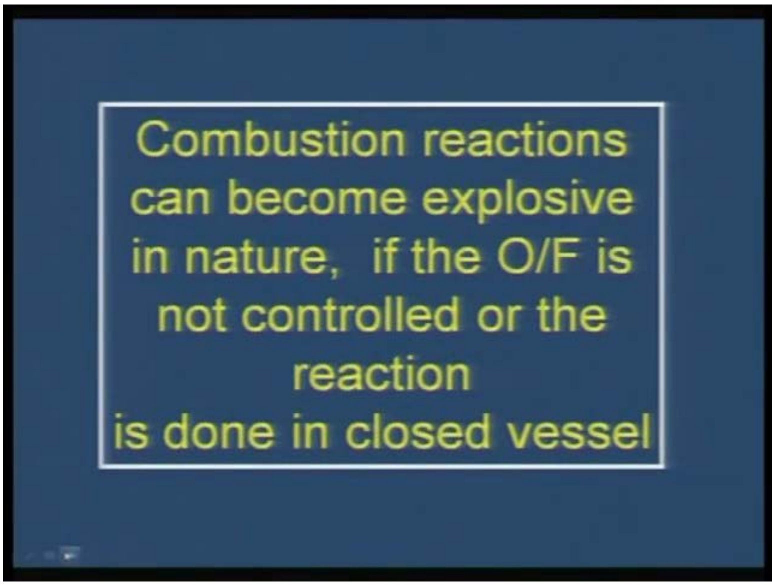

Combustion reactions
can become explosive
in nature, if the O/F is
not controlled or the
reaction
is done in closed vessel

So, the point that we need to notice, combustion reactions can become explosive in nature, if the O by F ratio that is, the oxidizer to fuel combination is not controlled. And if the reaction is not done in a open vessel, this can become a potential explosive situation. Therefore, we need to be careful, to control this reaction by doing such experiments in a open vessel. Now, how to tame combustion synthesis to make material is what we are going to see.

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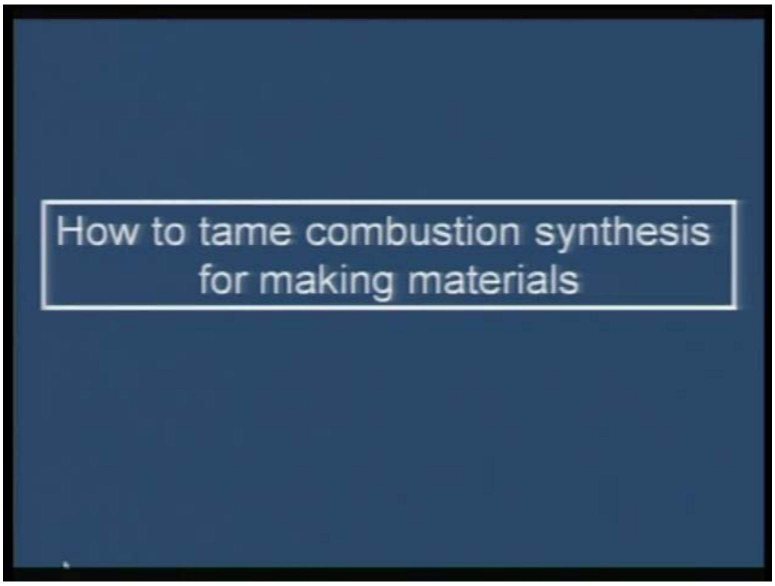

How to tame combustion synthesis
for making materials

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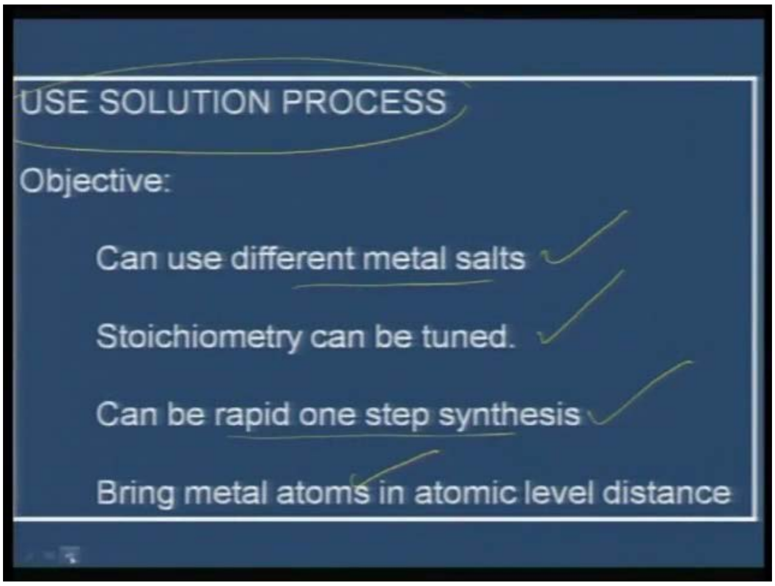

USE SOLUTION PROCESS

Objective:

- Can use different metal salts
- Stoichiometry can be tuned.
- Can be rapid one step synthesis
- Bring metal atoms in atomic level distance

The one of the option is to use a solution process, because as you have seen in the earlier cases, it has always been a dry mixture of all the ingredients mixed with the powerful oxidizer. And when you spark it, you get the corresponding oxide or metal, but the other possibility is to use a solution process, and what is the objective of using a solution combustion reaction. Number 1, we can use different metal salts, because we do not need to necessarily always start with pure metallic powders, which is often very very costly.

But, if you go for metal salts, those are cheaper available easier and you can use it in larger amounts for scale up activities, therefore this is one of the prime motive, why we can resort to a solution process. Number 2, when we try to make a quaternary mixture of metals in the final product then you can actually govern or you can hold the stoichiometry together, because you can carefully start with stoichiometric amounts of corresponding metal salts. Also, this can be rapid one step synthesis, because you do not need to necessarily mix all these solids to bring it into intimate contact.

Because, in solution, when you bring everything together then they are stoichiometrically and intimately mixed and as a result, the metal ions can be brought to atomic level distances. So, once metal ions in atomic distance and then you initiate combustion then the final product actually will be purely stoichiometric compound, which is decide for us. So, for this reason, one can resort to a solution process which is a viable alternative to the known SHS technology.

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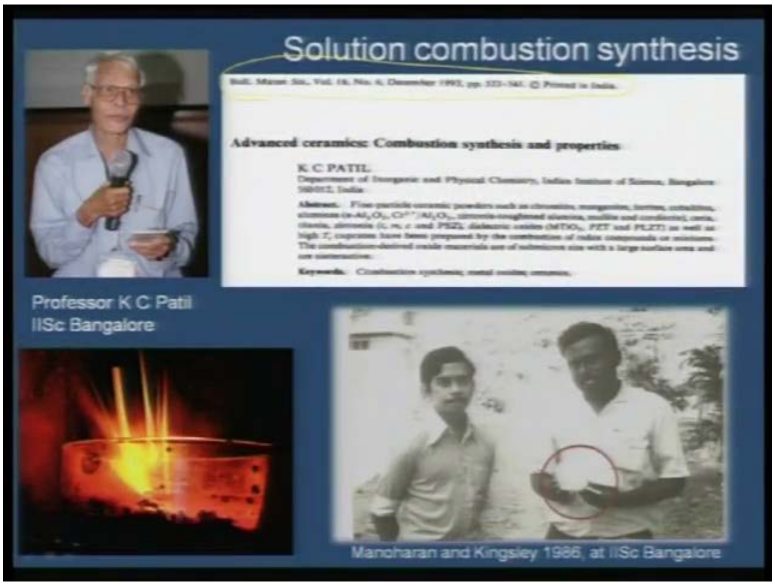

Solution combustion synthesis

Advanced ceramics: Combustion synthesis and properties

K C PATIL

Professor K C Patil
IISc Bangalore

Manoharan and Kingsley 1986, at IISc Bangalore

So, the person that I would like to highlight and bring to notice is professor Patil’s group at IISC, who finite this field called solution combustion synthesis. And it was such a fascinating discovery of making oxide materials that, this was the first ever review that was written on the solution combustion synthesis by professor Patil in bulletin of materials of science in December 1993. For those of you, who are interested in knowing the history of how this solution process was developed, one should read this article, therefore I have quoted this particularly.

And I also would like to record the other pioneers in this field including myself; this is Dr. Kingsley, who was the first person to publish a Ph.D thesis on this combustion synthesis. And this is interesting photograph, which we took in late 1986, where you can see Kingsley holding a petri dish, a glass dish containing alumina powder. I will show this photograph in the next slide and this was the first ever result that came out from solution combustion synthesis, which was developed at IISC, Bangalore.

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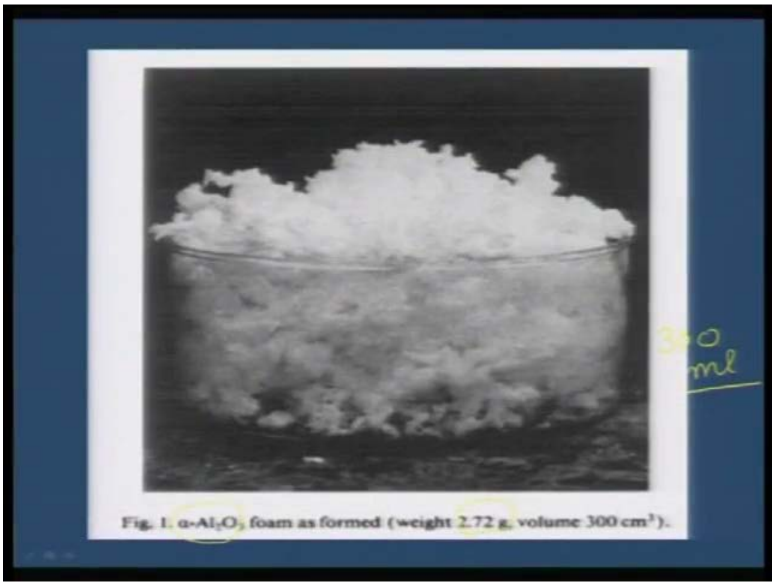

Fig. 1. α-Al_2O_3 foam as formed (weight 2.72 g, volume 300 cm^3).

Now, the solution combustion synthesis when developed, it can actually result in foamy residue of metal oxide particles. For example, you can see this is petri dish which is of 300 ml capacity, and in this 300 ml capacity dish, you can actually fill the whole dish with powder, which will just weigh only 2.72 grams. And this can be such a fine and porous powder that you can prepare, which has very high surface area with very low particle size.

There is no other method, by which one can prepare a compound like alpha alumina, which is a high temperature form. Because, when we try to make alpha alumina, it is usually a high temperature oxide, therefore you have to heat it to very high temperature in a electro heating furnish. And in such case, you will always end up getting a sintered product, which will be 100th of the volume that it will occupy. So, it is a very very important reaction, which gives high temperature phases with lot of special surface features.

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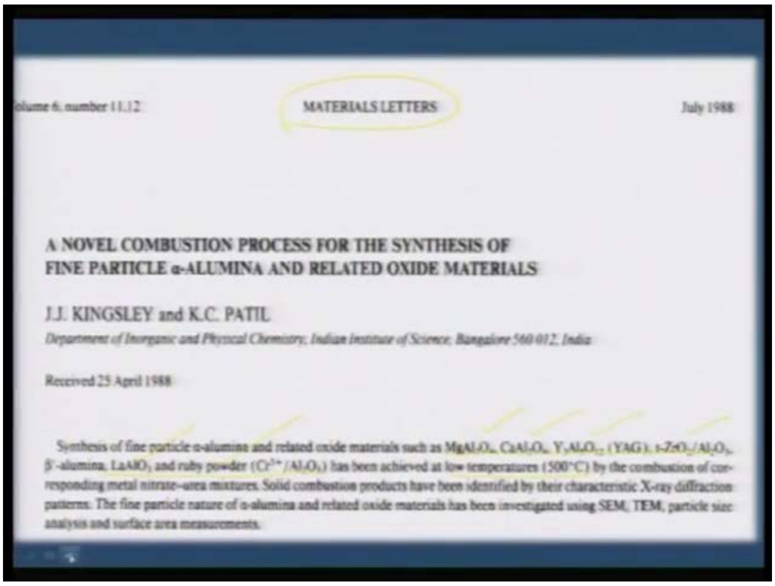

olume 6, number 11,12 MATERIALS LETTERS July 1988

A NOVEL COMBUSTION PROCESS FOR THE SYNTHESIS OF
FINE PARTICLE α-ALUMINA AND RELATED OXIDE MATERIALS

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Received 25 April 1988

Synthesis of fine particle α-alumina and related oxide materials such as $MgAl_2O_4$, $CaAl_2O_4$, $Y_3Al_5O_{12}$ (YAG), t-ZrO_2/Al_2O_3, β''-alumina, $LaAlO_3$ and ruby powder (Cr^{3+}/Al_2O_3) has been achieved at low temperatures (500°C) by the combustion of corresponding metal nitrate–urea mixtures. Solid combustion products have been identified by their characteristic X-ray diffraction patterns. The fine particle nature of α-alumina and related oxide materials has been investigated using SEM, TEM, particle size analysis and surface area measurements.

The first paper that was published on this solution combustion synthesis was published in material's letters, which is the pioneering publication made by Kingsley and Patil. And as you can see here, a list of aluminum based compounds were prepared including magnesium aluminate, calcium aluminate, yttrium aluminum garnet, zirconia lanthanum aluminate and including ruby powder, have been achieved. The main feature of that is, you can prepare all these compounds by just maintaining a solution at 500 degree C, which will get combusted to give this high temperature ceramics, we will look at some of the issues in greater detail in the next few slides.

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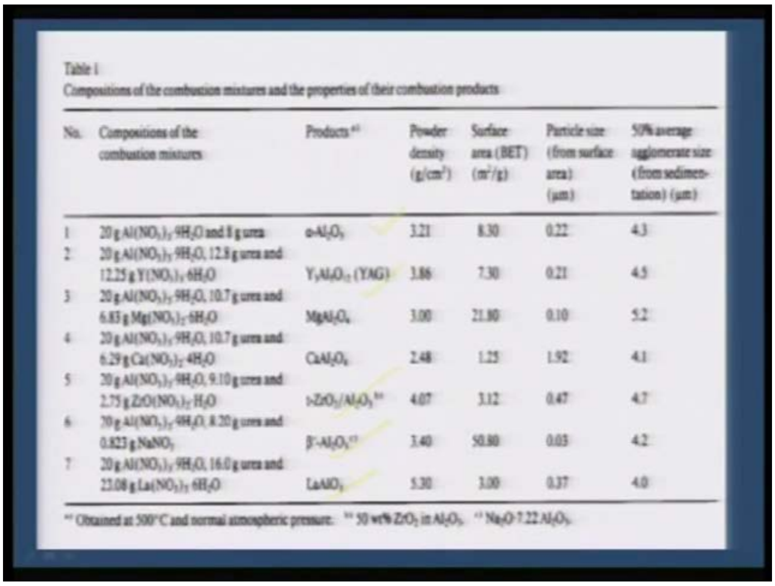

Table 1
Compositions of the combustion mixtures and the properties of their combustion products

No.	Compositions of the combustion mixtures	Products [a]	Powder density (g/cm^3)	Surface area (BET) (m^2/g)	Particle size (from surface area) (μm)	50% average agglomerate size (from sedimentation) (μm)
1	20 g $Al(NO_3)_3 \cdot 9H_2O$ and 8 g urea	α-Al_2O_3	3.21	8.30	0.22	4.3
2	20 g $Al(NO_3)_3 \cdot 9H_2O$, 12.8 g urea and 12.25 g $Y(NO_3)_3 \cdot 6H_2O$	$Y_3Al_5O_{12}$ (YAG)	3.86	7.30	0.21	4.5
3	20 g $Al(NO_3)_3 \cdot 9H_2O$, 10.7 g urea and 6.83 g $Mg(NO_3)_2 \cdot 6H_2O$	$MgAl_2O_4$	3.00	21.80	0.10	5.2
4	20 g $Al(NO_3)_3 \cdot 9H_2O$, 10.7 g urea and 6.29 g $Ca(NO_3)_2 \cdot 4H_2O$	$CaAl_2O_4$	2.48	1.25	1.92	4.1
5	20 g $Al(NO_3)_3 \cdot 9H_2O$, 9.10 g urea and 2.75 g $ZrO(NO_3)_2 \cdot H_2O$	t-ZrO_2/Al_2O_3 [b]	4.07	3.12	0.47	4.7
6	20 g $Al(NO_3)_3 \cdot 9H_2O$, 8.20 g urea and 0.823 g $NaNO_3$	β''-Al_2O_3 [c]	3.40	50.80	0.03	4.2
7	20 g $Al(NO_3)_3 \cdot 9H_2O$, 16.0 g urea and 23.08 g $La(NO_3)_3 \cdot 6H_2O$	$LaAlO_3$	5.30	3.00	0.37	4.0

[a] Obtained at 500°C and normal atmospheric pressure. [b] 50 wt% ZrO_2 in Al_2O_3. [c] $Na_2O \cdot 7.22\ Al_2O_3$.

The range of compounds what was published in the first paper include alumina and as you know, YAG is a laser material. And then tetragonal zirconia is a toughened ceramics, and then this is a electrolyte beta alumina and then La LO 3 is a very good precursor material for thin films. So, you could see here vividly that, almost all the aluminum based compounds were prepared in the first instance using this combustion procedure.

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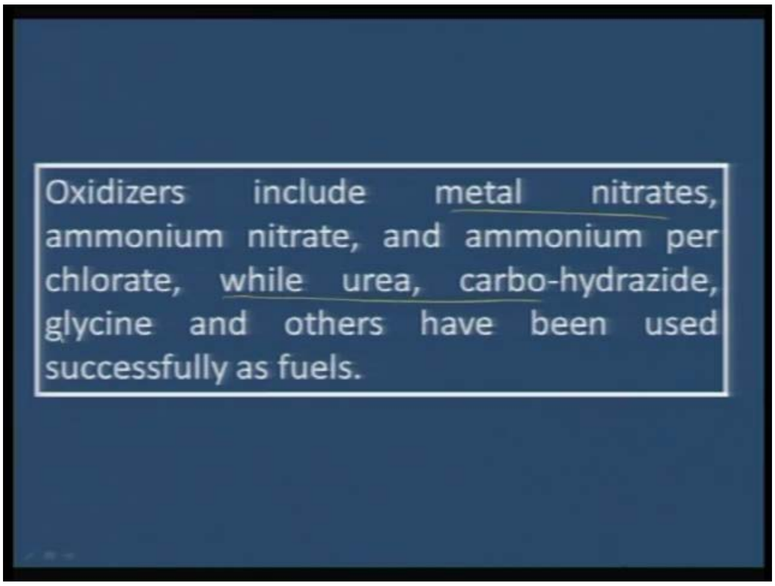

Oxidizers include metal nitrates, ammonium nitrate, and ammonium per chlorate, while urea, carbo-hydrazide, glycine and others have been used successfully as fuels.

So, what is this combustion, combustion of a proper combination of an oxidizer and a fuel, this can produce the exothermicity required for simultaneous synthesis of oxide ceramic powder. So, when combustion is generated, because of proper combination of an oxidizer and fuel, the energy that is released is actually used by a simultaneous formation of the metal powders or metal oxides powder. And for this reason, oxidizers which include metal nitrates and the fuels can be urea, carbohydrazide, glycine and others have been used successfully.

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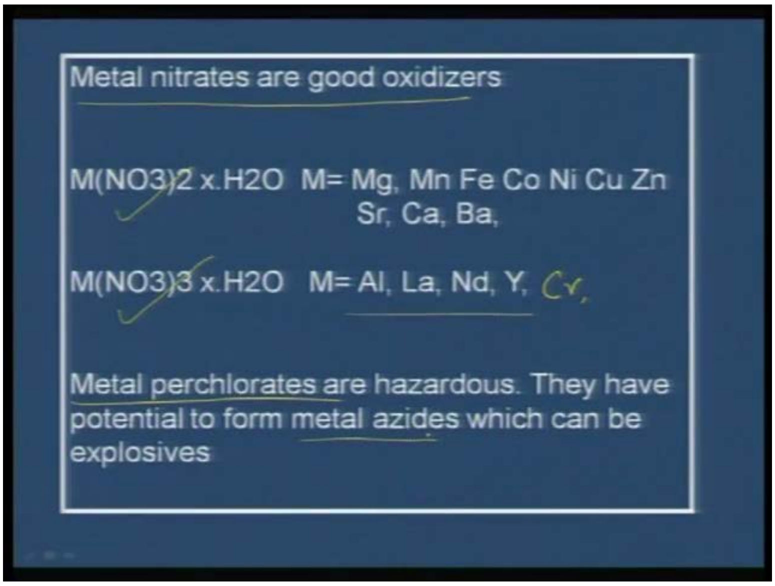

Metal nitrates are good oxidizers

M(NO3)2 x.H2O M= Mg, Mn Fe Co Ni Cu Zn
Sr, Ca, Ba,

M(NO3)3 x.H2O M= Al, La, Nd, Y, Cr,

Metal perchlorates are hazardous. They have potential to form metal azides which can be explosives

As a fuel I will come to this list in greater detail, the metal nitrates are good oxidizers. Therefore, you can either take a divalent metal or a trivalent metal nitrate, those which are divalent or magnesium, manganese, iron, cobalt, nickel, copper, zinc, strontium, calcium, barium. All these have the ability to form nitrates and they are bivalent, and trivalent ones are all the lanthanides lanthanide ions as well as aluminum.

So, if you are looking for in fact, chromium also can be included, because chromium nitrate is also trivalent. So, if you want to prepare chromites then you have to start with chromium nitrate, if you want to prepare aluminates then you have to use aluminum nitrate. Metal perchlorates, as a word of caution I want to register here that, metal perchlorates, although they are oxidizers, they are very very hazardous.

Because, they can produce lot of oxygen or oxidizing atmosphere they can produce, but what happens is, when you try to do a combustion, the perchlorates can actually transform into metal azides in combination with the fuel. And therefore, they can behave as explosives even on a lab scale, therefore caution has to be taken that, there should be no reference to making metal perchlorates as oxidizers, when you do combustion for making material synthesis.

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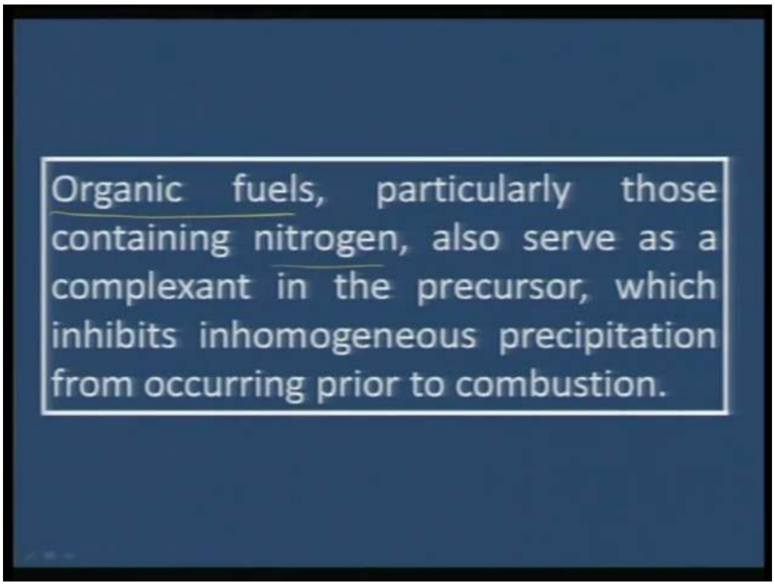

Organic fuels, particularly those containing nitrogen, also serve as a complexant in the precursor, which inhibits inhomogeneous precipitation from occurring prior to combustion.

Organic fuels, particularly those containing nitrogen also serve as a complexant in the precursor, which inhibits inhomogeneous precipitation. Therefore, any fuel that you are taking, if they are rich in nitrogen, they will invariably prevent complexation. As a result, when you add a fuel to a metal nitrate solution, there would not be precipitation, rather it will form a clear liquid. Therefore, the organic fuels that are preferred are those which have more number of nitrogen, and they also participate in a effective combustion reaction.

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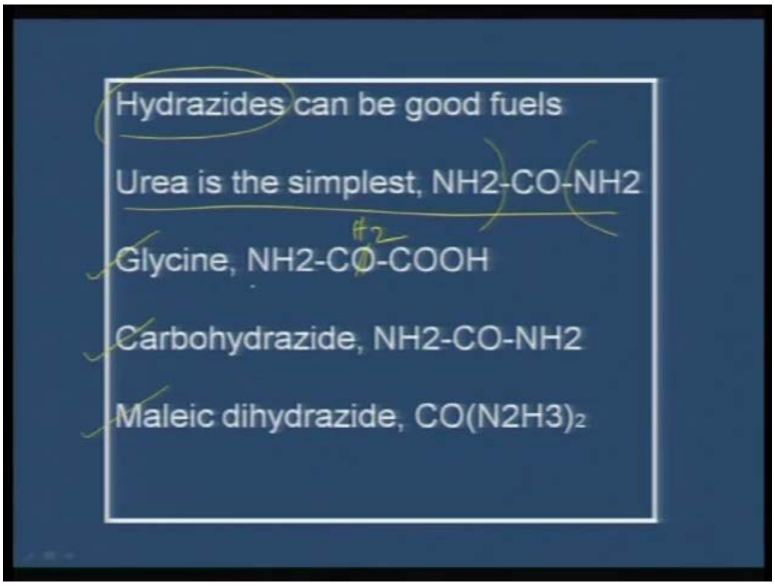

Hydrazides can be good fuels

Urea is the simplest, NH2-CO-NH2

Glycine, NH2-CO-COOH

Carbohydrazide, NH2-CO-NH2

Maleic dihydrazide, CO(N2H3)$_2$

So, such organic fuels are needed, the predominant ones are hydrazides, but do we really need a hydrazides. As I told you in the initial part of my lecture that hydrazides, mono methyl, dimethyl hydrazene, these are used as propellants. But, we do not really need to resort to hydrazine, what we can do is, we can even start with a simplest of such molecules organic fuels as simple as urea. Urea as we know, is not considered to be a fuel, it is considered to be a good fertilizer, otherwise.

But, from the propellant chemistry principle, we can observe that, even urea can play a role why, because on oxidative decomposition, urea will actually go out either as ammonia and carbon dioxide or they will go as nitrogen and hydrogen and carbon dioxide upon complete combustion. So, urea is a very good fuel plus it does not leave any residue as impurity, the simplest other one, other than urea is glycine, I will show some examples of, how they should be actually CH 2.

This is glycine and then you have carbohydrazide and then you have maleic dihydrazide, all this sort of hydrazides are simple ammonia based organic molecules can be used for the combustion as fuels.

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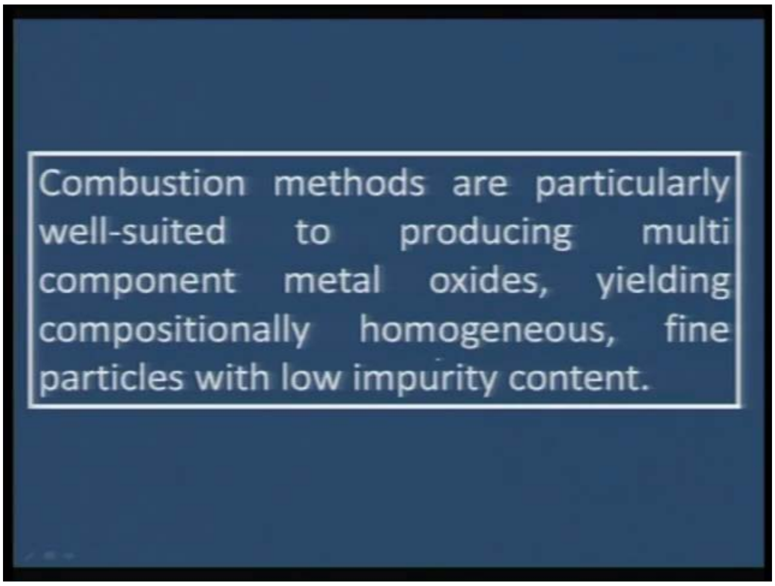

Combustion methods are particularly well-suited to producing multi component metal oxides, yielding compositionally homogeneous, fine particles with low impurity content.

Combustion methods are particularly well suited to producing multi component metal oxides, yielding compositionally homogeneous fine particles with low impurity content, this is the specialty of this wet chemical route.

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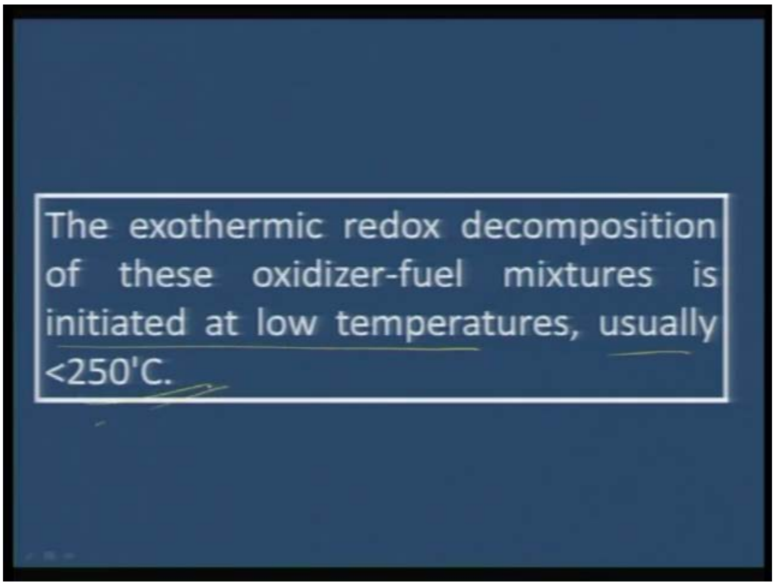

The exothermic redox decomposition of these oxidizer-fuel mixtures is initiated at low temperatures, usually <250'C.

The exothermic redox decomposition of these oxidizer fuel mixtures is actually initiated at low temperatures. Usually less than 250 degree C, as you would see from another animation that it is very easy for us to prepare such high temperature materials using a wet chemical combustion procedure, where you just initiate the reaction with as low as 250 to 500 degree C.

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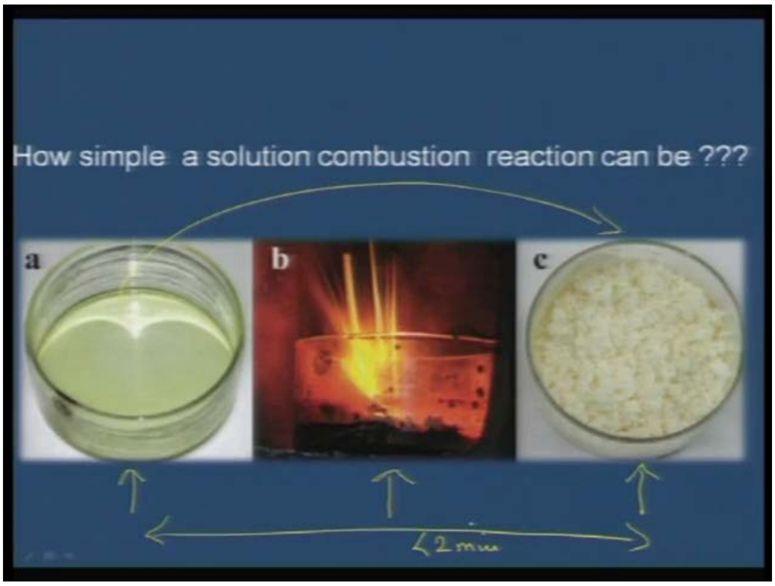

How simple a solution combustion reaction can be ???

Now, in a regular protocol for solution combustion synthesis is very easy, because in step one all that you need to do is, take a proper combination of a fuel and oxidizer and make a homogeneous solution in a beaker such as, this. This is a glass beaker and you have to take very minimum amount of water and not excess amount. Because, when the reaction is occurring, sometimes the fuel can be destroyed when it goes through a long heating time.

Therefore, we need to have minimum amount of water just to dissolve both the oxidizer and fuel. And once you insert it into a furnace then you see a typical combustion reaction such as, this happening and after the combustion reaction, the solution is actually converted to a product. And this is a example of preparing ceria doped with platinum, example I will quote in the slides to follow. So, what is a time scale, from the time you make this solution and you get this product after inserting through a furnace. The whole thing takes less than 2 minutes, so in 2 minutes time; you can achieve such a high temperature reaction.

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This is a movie, which I want to show before I touch upon the issues related to combustion synthesis. So, I just want you to watch this movie, what you see here is a muffle furnace, a open muffle furnace and this furnace is actually maintained at 500 degree C. So, what you can do, instead of taking a petri dish, you can even use a 250 or 500 ml beaker and take this solution and put it inside a preheated furnace and then you would see the sought of reaction that is happening.

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Al(2-x)Cr(x)O3

Ruby

So, as you see, you have the oxidizer and the fuel, which is going through complete dehydration and the excess water is actually coming out, as you are heating it. The first few seconds, you can see some sort of frothing, that is happening and after that, this combustion reaction occurs. During the combustion, you can actually see how the wave propagation is, the exothermicity is actually propagating from the top downwards. And as and when the reaction is, the temperature is used for converting that solution mixture into oxide then the oxide is coming out and then the propagation is actually going down.

And once this is done, you can see that the ruby powder that is formed is coming out and you can see the pinkish tinch in this solid product. And (Refer Slide Time: 36:15) if you take the p l for this ruby powder, you can see the excitation spectra and the emission spectra exactly matches with that of a ruby crystal. And as you know, ruby crystal is nothing but a laser material, which is used for making ruby lasers and they show a characteristic emission around 695 nanometer. So, if you get such a sharp emission peak then you can be sure that, it is actually a lasing action that is coming from ruby powder.

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solution
Combustion
Synthesis
of
metal oxides

And this ruby powder incidentally can be prepared from mixing less than 1 percent of chromium nitrite in aluminum nitrite. So, whatever that is coming out, is not alumina powder, but it is actually ruby powder that is, chromium atomically doped in the aluminum sites of aluminum oxide. So, this is a fabulous reaction, where such low level impurities can be very nicely incorporated into alumina matrix, this is not the only compound, we can make n number of compounds out of such approach. All you need to do is, depending on the end stoichiometry, we need to take the corresponding metal nitrides to prepare such oxides.

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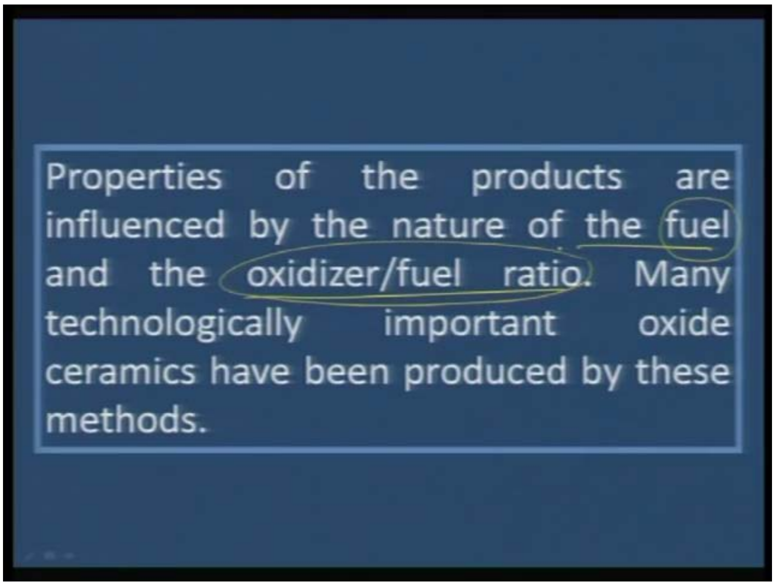

Properties of the products are influenced by the nature of the fuel and the oxidizer/fuel ratio. Many technologically important oxide ceramics have been produced by these methods.

Properties of these products are therefore, influenced they are influenced by the nature of the fuel and the oxidizer fuel ratio, many technologically important oxide ceramics can be produced by this method. So, I will try to take you through few examples to show you how the fuel content is important, and what is the relevance of this oxidizer fuel ratio, and how do you calculate this oxidizer fuel ratio to make such combinations.

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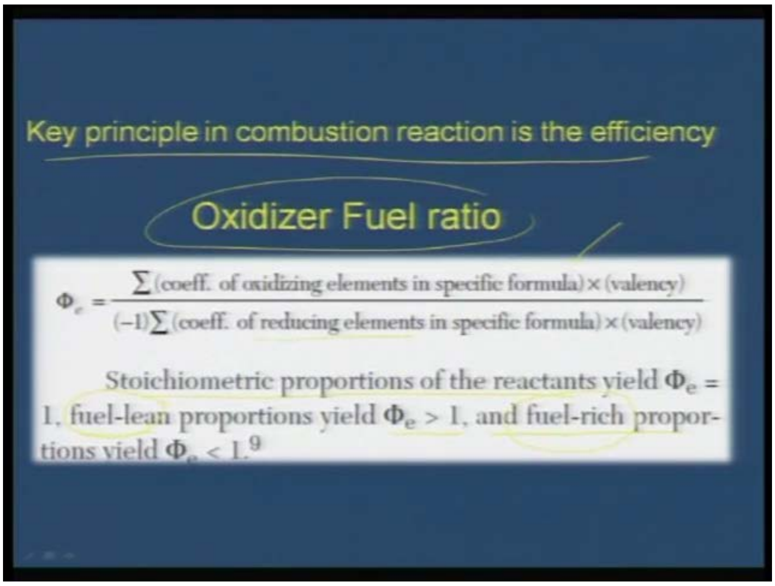

Key principle in combustion reaction is the efficiency

Oxidizer Fuel ratio

$$\Phi_e = \frac{\sum(\text{coeff. of oxidizing elements in specific formula}) \times (\text{valency})}{(-1)\sum(\text{coeff. of reducing elements in specific formula}) \times (\text{valency})}$$

Stoichiometric proportions of the reactants yield $\Phi_e = 1$, fuel-lean proportions yield $\Phi_e > 1$, and fuel-rich proportions yield $\Phi_e < 1$.[9]

The key principle in combustion reaction is the efficiency, and this efficiency of combustion is actually calculated by the oxidizer to fuel ratio which is given as Q e or phi e. We can calculate is nothing but a summation of the coefficient of oxidizing elements in the specific formula into valency over minus 1 into a sum of coefficient of reducing elements in the specific formula into valency. Therefore, if you have your metal nitrates, you can actually calculate the oxidizing valency in the numerator.

And if you have a fuel then you can calculate reducing valency of your organic fuels, based on their valency. So, accordingly a stoichiometric proportion of these reactants should yield Q by e should be equal to 1, if Q by e is equal to 1 then you can say then the combustion efficiency will be attached maximum. But, there is a case when if the fuel efficiency is less than 1 that means, it is a fuel lean composition, meaning there is more oxidizer though, so you need to lower down on the oxidizer proportion.

And there is fuel rich case when the phi e is less than 1, so both this has to be avoided and preferably it is always better to take a fully stoichiometric situation. But, then when you are actually taking metals, metals in the initial during the combustion can get reduced to metallic metal, which can also catalyze the combustion. Therefore, the exothermicity can be more, in such cases always it is better to play down with a fuel lean mixture than fuel rich mixtures. So, depending on the type of reaction that you are aiming you need to fine tune on the oxidizer to fuel ratio.

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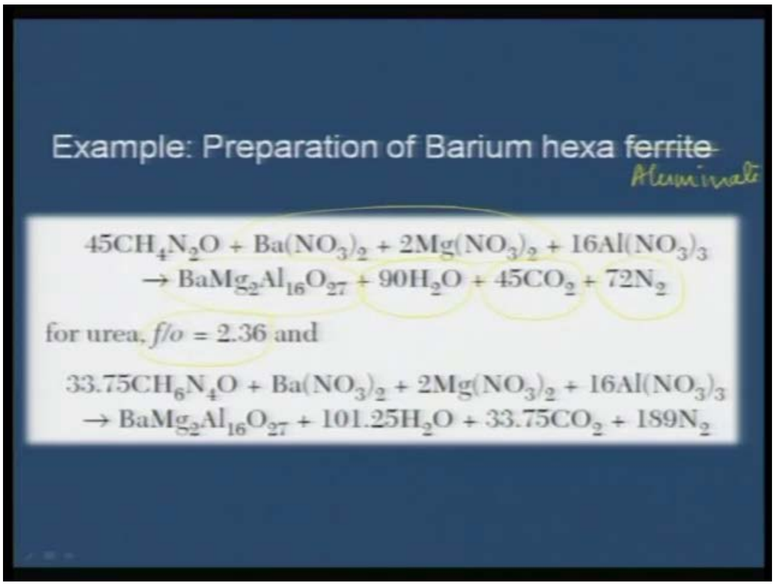

Example: Preparation of Barium hexa ~~ferrite~~ Aluminate

$$45CH_4N_2O + Ba(NO_3)_2 + 2Mg(NO_3)_2 + 16Al(NO_3)_3 \rightarrow BaMg_2Al_{16}O_{27} + 90H_2O + 45CO_2 + 72N_2$$

for urea, $f/o = 2.36$ and

$$33.75CH_6N_4O + Ba(NO_3)_2 + 2Mg(NO_3)_2 + 16Al(NO_3)_3 \rightarrow BaMg_2Al_{16}O_{27} + 101.25H_2O + 33.75CO_2 + 189N_2$$

For example, let us take the case of barium hexaferrite which is one of the compound and barium hexaferrite has a composition BaMg2 Al16O27, and this barium hexaferrite can be prepared using urea. And if you want to balance this equation for a complete combustion this is how it looks like, where you take 45 moles of this barium nitrate, magnesium nitrate, aluminum nitrate to give, this should be barium hexaaluminate, this is barium hexaaluminate.

But, what should you understand is during complete combustion, you will have 90 moles of water which is released, and this water will actually go as the initial step in the decomposition process before the combustion occurs. But, when the combustion occurs you actually have 45 moles theoretically possible, 45 moles of carbon dioxide and 72 moles of nitrogen that is released upon complete combustion. And this is one of the important features of this solution combustion process, because it is a gas releasing exercise, enormous amount of gasses are released as a result the final residue will always be a porous and a finally, divided metal oxide.

If this much amount of gas is not released, then will be a highly sintered complex, so if you want to prepare a nano dimensional stuff, then you should actually favor a solution combustion process. Because, of the gas evolution it gives a highly reactive solid, and as you saw from the movie the whole process is actually occurring in less than 1 minute time. And the gases which are trapped is the ones which are hypergolic which is giving this high temperature, and because of these gases which are released during the combustion, they are not sintering the particles.

Another thing the whole reaction is also run in a beaker, in a glass beaker it is a phenomenal reaction. Because, even though such high temperatures above 1000 degrees are achieved, yet we are not seeing the glass melting and this glass is not melting even at very high temperatures. Because, the energy that is released during the combustion is actually dissipated or flushed out by the escaping gases number 1 and the reaction mixture is also consuming this energy to transform into the corresponding oxide product.

As a result, even though you are using a glass vessel with very high temperature, the glass does not melt at all, because it is a fast quenching technique. So, one of the things that we need to understand is that, when we try to take a mixture of metal nitrate and fuel in this case, the calculated ratio as far as the oxidizer fuel ratio is concerned has to be 2.36. So, when you control this fuel to oxidizer ratio, then it will be possible for us to control the exothermicity of the reaction.

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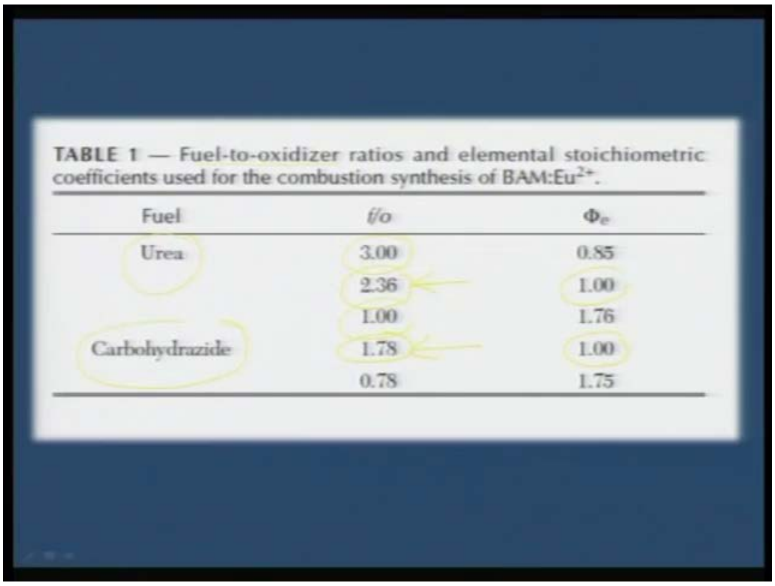

TABLE 1 — Fuel-to-oxidizer ratios and elemental stoichiometric coefficients used for the combustion synthesis of BAM:Eu^{2+}.

Fuel	*f/o*	Φ_e
Urea	3.00	0.85
	2.36	1.00
	1.00	1.76
Carbohydrazide	1.78	1.00
	0.78	1.75

Now, we can also play around with different compositions or different combinations for example, the fuel to oxidizer ratio can vary with the sort of the reducing valences of the fuel. And in this case urea, if you take urea then for the energy to be maximum this is the dictated formula, fuel to oxidizer ratio. But, if you can also play around with this three or with one, which is a fuel lean is situation. In such cases, you will see the efficiency will vary and it will not be equal to 1. Now, if you go for carbohydrazide, you see for the fuel efficiency to be 1, in this case the fuel to oxidizer ratio has to be 1.78. Whereas, in this case for urea, it was 2.36, that is because the number of reducing valencies in carbohydrazide is different the number of reducing valencies in urea.

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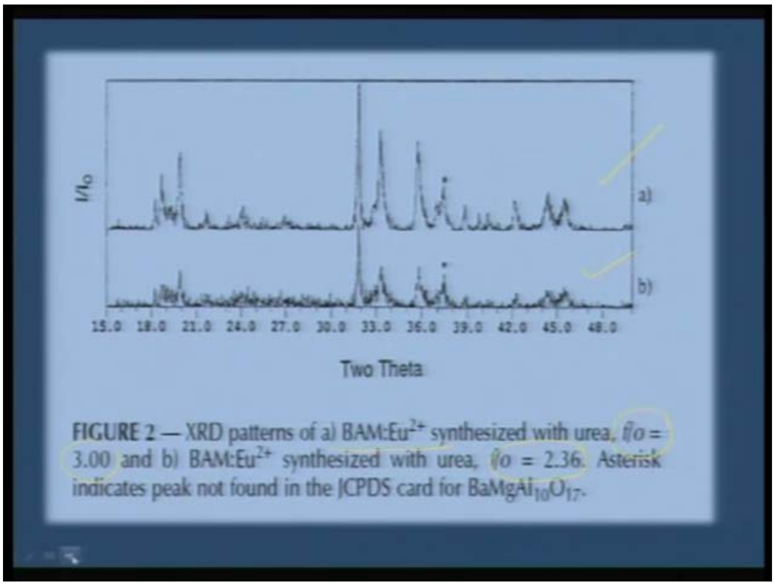

FIGURE 2 — XRD patterns of a) BAM:Eu^{2+} synthesized with urea, f/o = 3.00 and b) BAM:Eu^{2+} synthesized with urea, f/o = 2.36. Asterisk indicates peak not found in the JCPDS card for $BaMgAl_{10}O_{17}$.

I will give you one more example of, how this fuel ratio can influence, for example you take the barium hexaaluminate, which is doped with urea. If you take the fuel to oxidizer ratio at 3 or if you take fuel to oxidizer ratio at 2.36, you can see the change in the crystallinity. This is according to the O by F ratio, but this is not according to the O by F ratio, as a result you can clearly see the crystallinity of the n product is varying.

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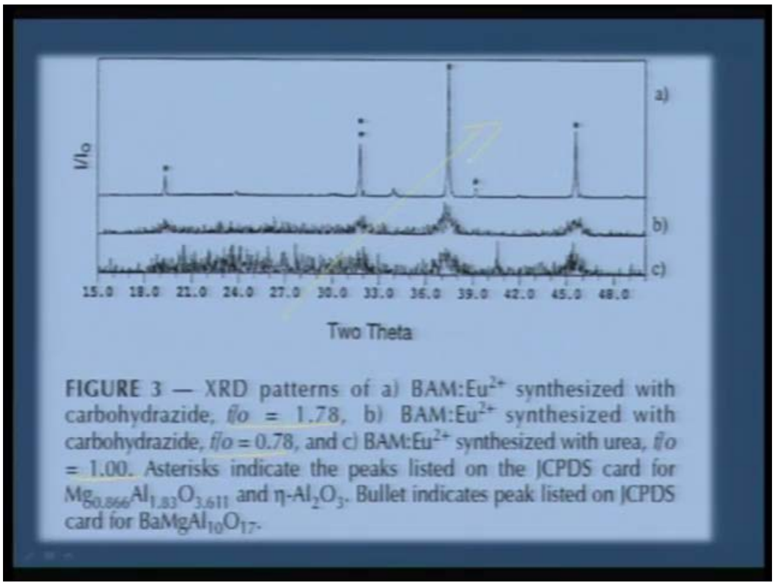

FIGURE 3 — XRD patterns of a) BAM:Eu^{2+} synthesized with carbohydrazide, f/o = 1.78, b) BAM:Eu^{2+} synthesized with carbohydrazide, f/o = 0.78, and c) BAM:Eu^{2+} synthesized with urea, f/o = 1.00. Asterisks indicate the peaks listed on the JCPDS card for $Mg_{0.866}Al_{1.83}O_{3.611}$ and η-Al_2O_3. Bullet indicates peak listed on JCPDS card for $BaMgAl_{10}O_{17}$.

So, this is proving very crucial and again in this example, you can find out that, if you vary the fuel to oxidizer ratio depending on, whether it is a fuel rich or fuel lean or stoichiometric, you can see that there is a enormous change in the crystallinity for the same composition. So, the oxidizer to fuel ratio is very, very critical in this example.

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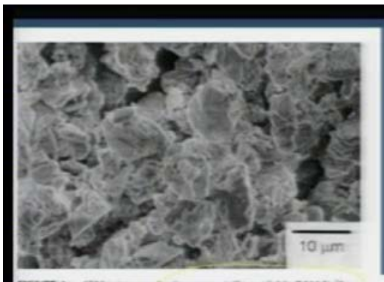

FIGURE 4 — SEM micrograph of commercially available BAM:Eu^{2+}.

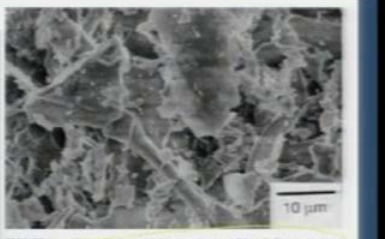

FIGURE 5 — SEM micrograph of as-synthesized BAM:Eu^{2+} synthesized with urea, f/o = 1.00.

And once you make that, you can clearly see that the commercially available barium hexaaluminate is almost similar to the features of combustion derived powders. So, we can make commercially viable synthesis, if you can scale it up to proper proportion.

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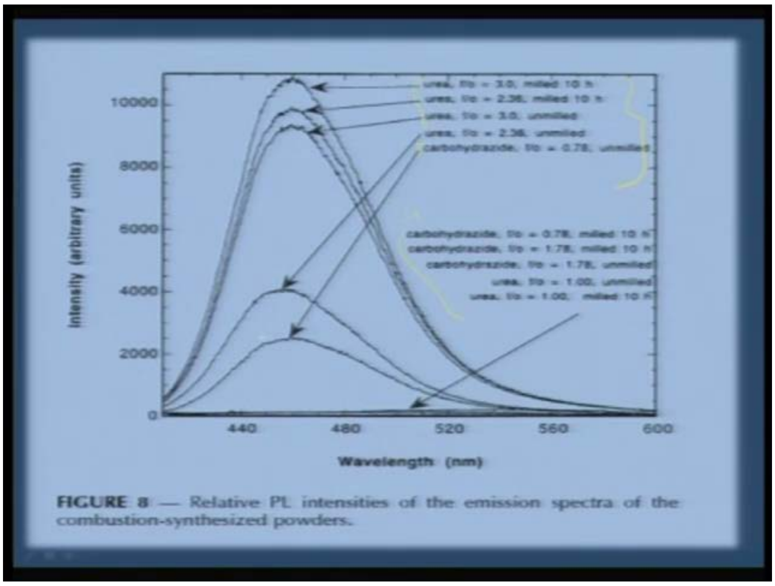

FIGURE 8 — Relative PL intensities of the emission spectra of the combustion-synthesized powders.

We can also see in this profile, the relative intensities of this barium hexaaluminate doped with europium, you can see depending on the oxidizer to fuel ratio, either using urea or carbohydrazide, the p l is drastically changing for this phosphor. So, we have to optimize, which is the proper stoichiometry for getting the right type of property that we desire.

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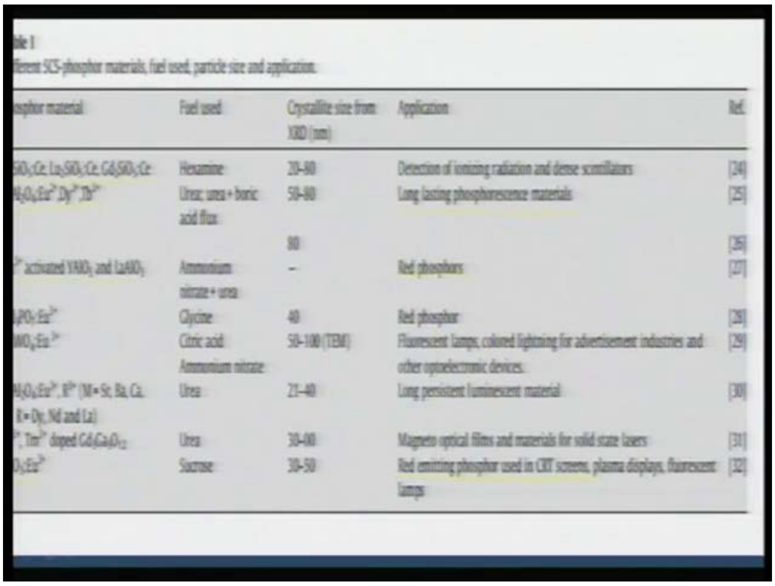

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ferent SCS-phosphor materials, fuel used, particle size and application.

sphor material	Fuel used	Crystallite size from XRD (nm)	Application	Ref.
SiO$_5$:Ce, Lu$_2$SiO$_5$:Ce, Gd$_2$SiO$_5$:Ce	Hexamine	20–80	Detection of ionizing radiation and dense scintillators	[24]
Al$_2$O$_4$:Eu^{2+},Dy^{3+},Tb^{3+}	Urea; urea + boric acid flux	50–80	Long lasting phosphorescence materials	[25]
		80		[26]
$^{3+}$ activated YAlO$_3$ and LaAlO$_3$	Ammonium nitrate + urea	–	Red phosphors	[27]
PO$_4$:Eu^{3+}	Glycine	40	Red phosphor	[28]
WO$_4$:Eu^{3+}	Citric acid Ammonium nitrate	50–100 (TEM)	Fluorescent lamps, colored lightning for advertisement industries and other optoelectronic devices.	[29]
Al$_2$O$_4$:Eu^{2+}, R^{3+} (M = Sr, Ba, Ca, R = Dy, Nd and La)	Urea	21–40	Long persistent luminescent material	[30]
$^{3+}$, Tm^{3+} doped Gd$_3$Ga$_5$O$_{12}$	Urea	30–80	Magneto optical films and materials for solid state lasers	[31]
O$_3$:Eu^{3+}	Sucrose	30–50	Red emitting phosphor used in CRT screens, plasma displays, fluorescent lamps	[32]

This is another example of, what are all the important phosphors that can be prepared using the combustion synthesis. As you know, yttrium silicate doped with the ceria can be used for centiliters application, we have strontium aluminate doped with europium for long lasting phosphorescence material, strontium aluminate with the dysprosium terbium then we have europium activated LaLo 3 as red phosphors, we also have yttria doped with europium as red phosphor in CRTA tubes. So, host of phosphorous can be prepared using this combustion synthesis.

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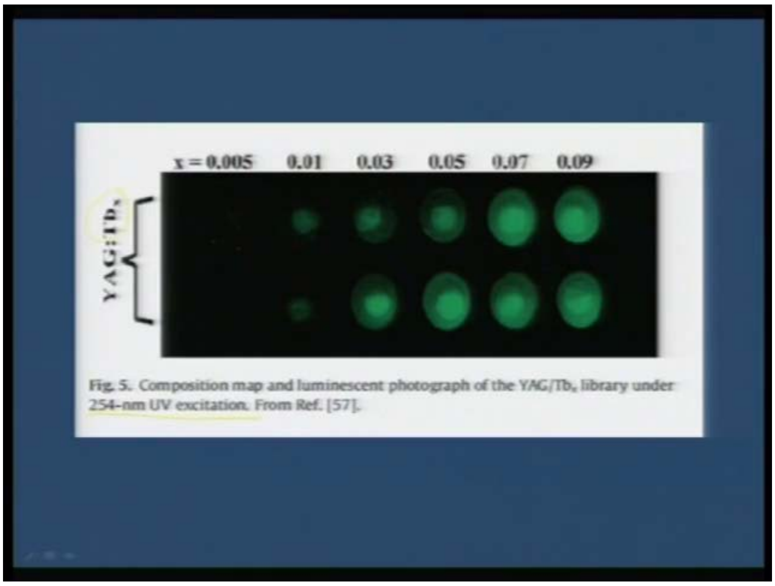

Fig. 5. Composition map and luminescent photograph of the YAG/Tb$_x$ library under 254-nm UV excitation. From Ref. [57].

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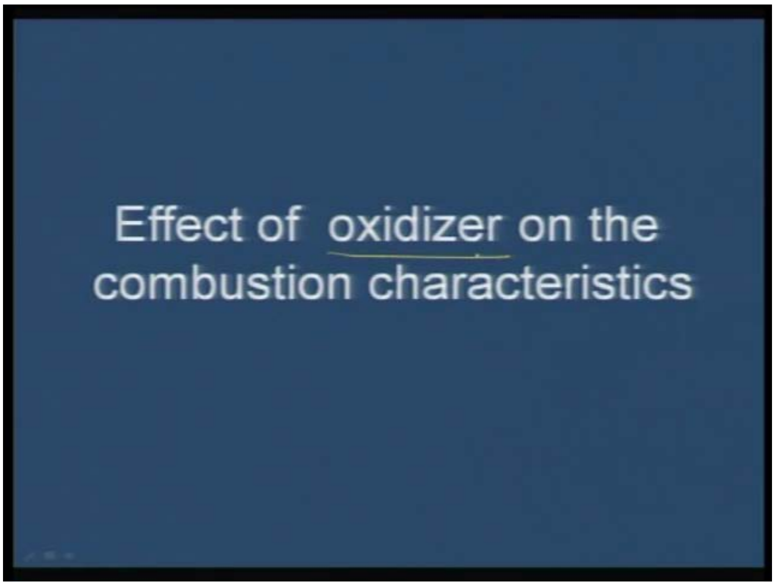

Effect of oxidizer on the combustion characteristics

Here is another example of, how with the doping concentration of terbium in yttrium aluminum garnet, one can change the luminescence of resulting powder using a combustion synthesis. And these are all the YAG powders, which are activated at 254 nanometer excitation. Now, I will give one example of, how crucial the oxidizer can play the role, effect of oxidizer on the combustion characteristics.

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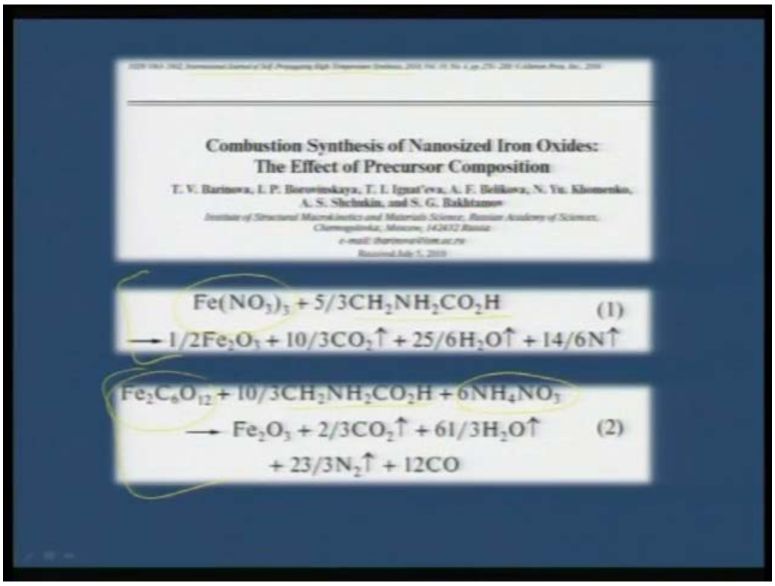

Combustion Synthesis of Nanosized Iron Oxides:
The Effect of Precursor Composition

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$$Fe(NO_3)_3 + 5/3CH_2NH_2CO_2H \quad (1)$$
$$\rightarrow 1/2Fe_2O_3 + 10/3CO_2\uparrow + 25/6H_2O\uparrow + 14/6N\uparrow$$

$$Fe_2C_6O_{12} + 10/3CH_2NH_2CO_2H + 6NH_4NO_3$$
$$\rightarrow Fe_2O_3 + 2/3CO_2\uparrow + 61/3H_2O\uparrow \quad (2)$$
$$+ 23/3N_2\uparrow + 12CO$$

This is a paper which appeared in 2010, I just picked out this paper, because even now people are experimenting on making nano sized iron oxidizes, this has appeared in international journal of self propagating high temperature synthesis. Although it is nearly 24 years since this process was devised, still lot of activities going on, I am just going to point out to you the use of ferric nitrate and ferric oxalate.

And ferric oxalate is used as a oxidizer in one hand and ferric nitrate is also used as a oxidizer, where glycine has been used as the fuel. In both cases glycine is used, but because we do not have nitrate here, extra nitrate used in the form of ammonium nitrate. Suppose, I do not have a metal nitrate then we do not have to worry about it, you can take a salt of that particular metal and you can compensate for the combustion reaction to occur by taking ammonium nitrate separately, that is also possible. So, if I take this combination or if I take this combination, you can see how the property varies.

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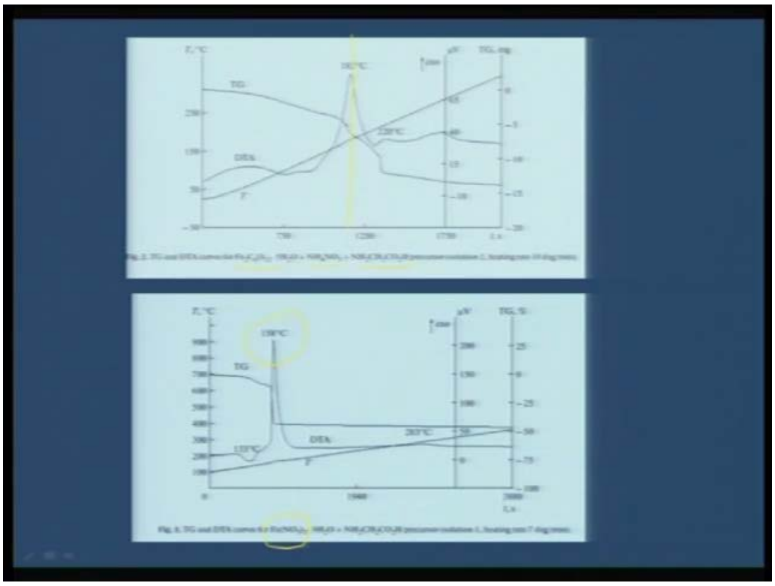

As you see here, this is the TGDTA for both this combinations, if you take iron oxalate, ammonium nitrate and glycine together, the exothermic decomposition is occurring somewhere around 182 degree C. Whereas, if you take metal nitrate, iron nitrate with the glycine, you can see phenomenally the combustion reaction occurs at a very early stages. There is a difference of around 40 degrees, if you take metal nitrate compared to iron oxalates. So, the starting material can actually play a important role in the combustion, not only that, the resulting powders also determine the quality of sample that you can see.

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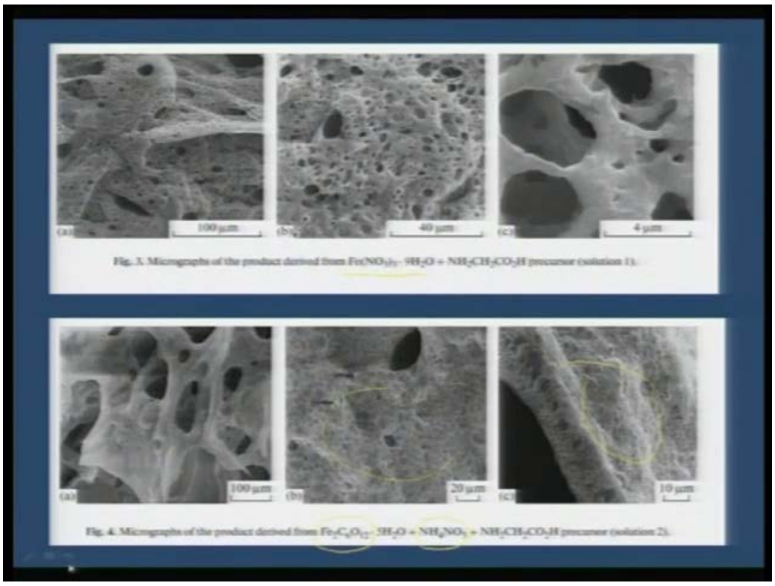

Fig. 3. Micrographs of the product derived from $Fe(NO_3)_3 \cdot 9H_2O + NH_2CH_2CO_2H$ precursor (solution 1).

Fig. 4. Micrographs of the product derived from $Fe_2C_6O_{12} \cdot 5H_2O + NH_4NO_3 + NH_2CH_2CO_2H$ precursor (solution 2).

If you take iron nitrate as a starting material with glycine, look at the porosity here, 100 micron, 40 micron the enlarged version you can see, so much of porosity on the metallic oxide foams that are produced and this porosity will determine the surface area also. Whereas, if you take iron oxalate and ammonium nitrate separately then you can see that, hardly there is any porosity and it is all well centered. Therefore, this iron oxide what you get here, will be more centered and less surface area material compared to the other one. And you can also see the morphology of the iron oxide powders starting with the oxalate and nitrate.

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Fig. 5. Morphology of Fe_2O_3 particles obtained from $Fe_2C_6O_{12} \cdot 5H_2O + NH_4NO_3 + NH_2CH_2CO_2H$ precursor (solution 2) at normal (a) and elevated (b) amount of glycine in starting composition.

Fig. 6. Micrograph of Fe_2O_3 particles derived from $Fe(NO_3)_3 \cdot 9H_2O + NH_2CH_2CO_2H$ precursor (solution 1).

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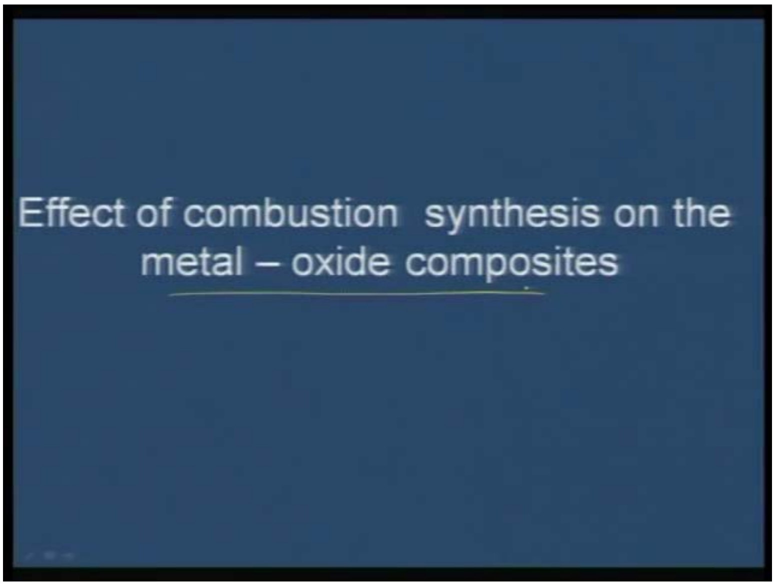

Effect of combustion synthesis on the metal – oxide composites

So, distinctly it alters the surface properties, now the effect of combustion synthesis on the metal oxide composite. I will now show you some example of the systems, so far we have seen the effect of fuel and oxidizer, now I will tell you, what all we can achieve out of such combustion reaction. We do not have to exactly make metals alone or oxides alone, we can also make composite materials.

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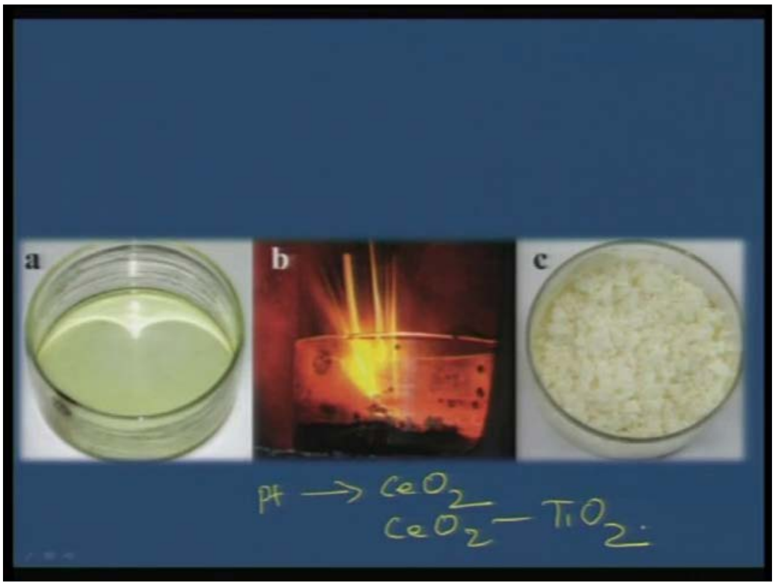

Pt → CeO_2
CeO_2 — TiO_2.

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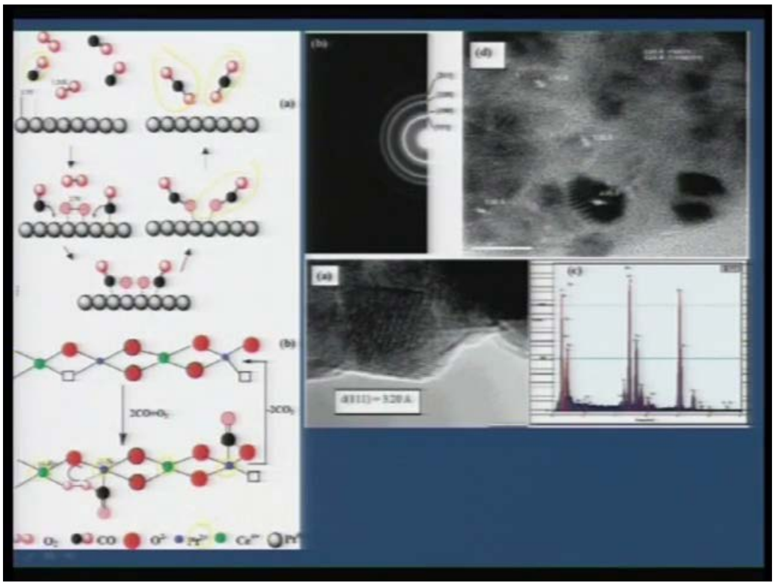

This example I have already shown you, the reaction is nothing but doping platinum in ceria or cerium oxide, titanium oxide composites. Why they are used, because these are very powerful auto exhaust catalyst, ceria impregnated with the platinum or platinum doped with the TiO 2 is used as a auto mobile exhaust catalyst. But, what you see here after some cycle, this catalytic convertors are dead, they are poisoned, because of the out coming exhaust, as a result the efficiency goes down.

But, if you use combustion reaction, you can see the activity is enhanced to a greater order, the mechanistic path way for such reaction is given here, where your carbon dioxide is coming getting adsorbed with the molecular oxygen. And you can see, there is a computing interaction on the surface and then the carbon monoxide goes as carbon dioxide and it cleaves out.

And this is the mechanistic path way, but what makes this combustion process interesting is that, unlike the other cases where you try to prepare alumina with platinum or platinum with ceria or platinum with the titania, they are all surface absorbed. So, the noble metals will actually do the catalytic process with the host material almost as a mute spectator. But, in the combustion process you can see, that the platinum, cerium, palladium, everything is actually doped, they are not as 0 valent metals, but they are here as ions.

Once they are substituted effectively into this host lattice then the conversion process of carbon monoxide to carbon dioxide is 15 to 20 times faster than the conventional reactions.

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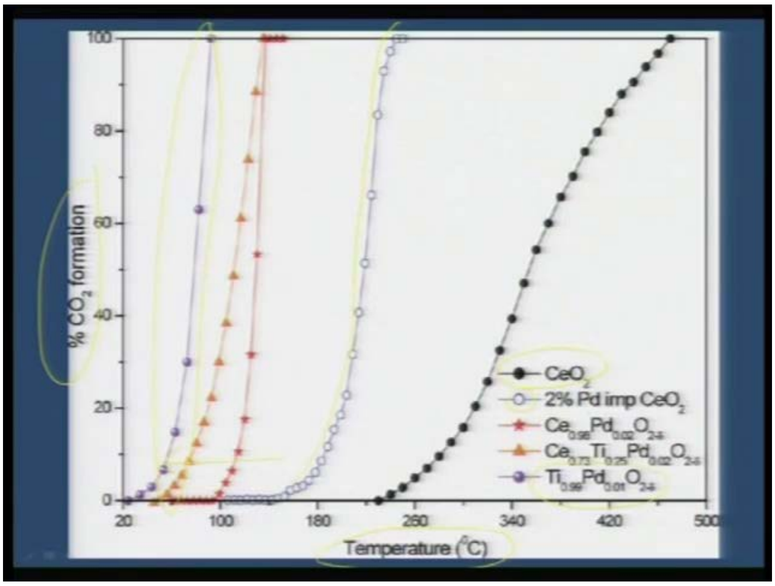

As you can see here, this is a profile of carbon dioxide formation as function of temperature. If you just take ceria without doping then this is the conversion profile, but once you dope just with the 2 percent of the palladium, you can see the shift in the temperature range and the way it is so highly selective. The conversion is very very selective and it is much faster mainly because palladium is not actually sitting on the ceria, but it is actually doped into the lattice and palladium actually goes to palladium 2 plus and ceria, cerium 4 plus goes to cerium 3 plus.

And therefore, it is now more as a substituted catalyst, rather than a composite. As a result, you see a remarkable conversion of CeO to CeO2 and this can easily replace all the noble metal catalyst in today’s automotive industry. Similarly, you can also see, just 1 percent of palladium that is doped into TiO2 can actually push the conversion even to near to room temperature. So, if you use this 1 percent palladium doped CeO2, you can see the conversion as low as 50 degree C.

At 50, you can convert all the carbon monoxide to carbon dioxide, why this is useful, because the combustion route has a peculiar way of pushing the noble metal into the lattice. And because they are sitting inside the lattice, the way the conversion of CO to CO 2 goes is very, very fissile.

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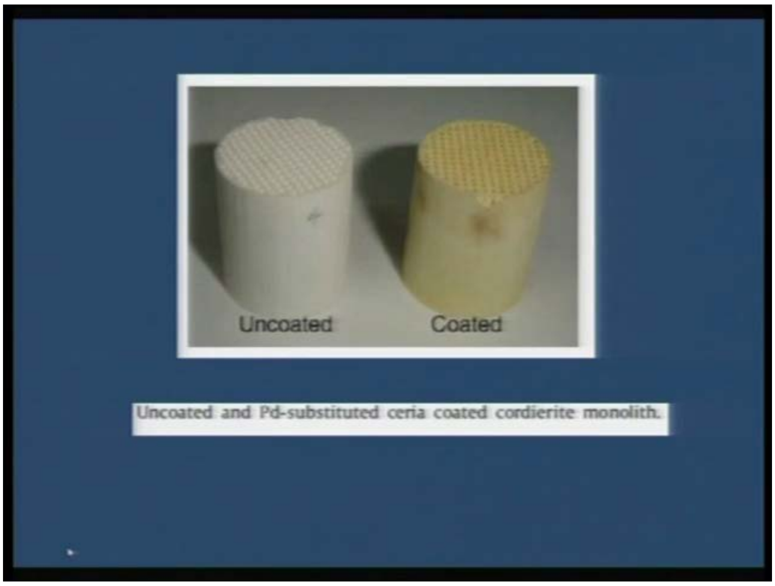

Uncoated and Pd-substituted ceria coated cordierite monolith.

We can also try to impregnate this catalyst onto this sort of cordierite monoliths; this is typically the way the exhaust catalysts are embedded in the convertors. You can uniformly coat it and make it a useful catalyst bed.

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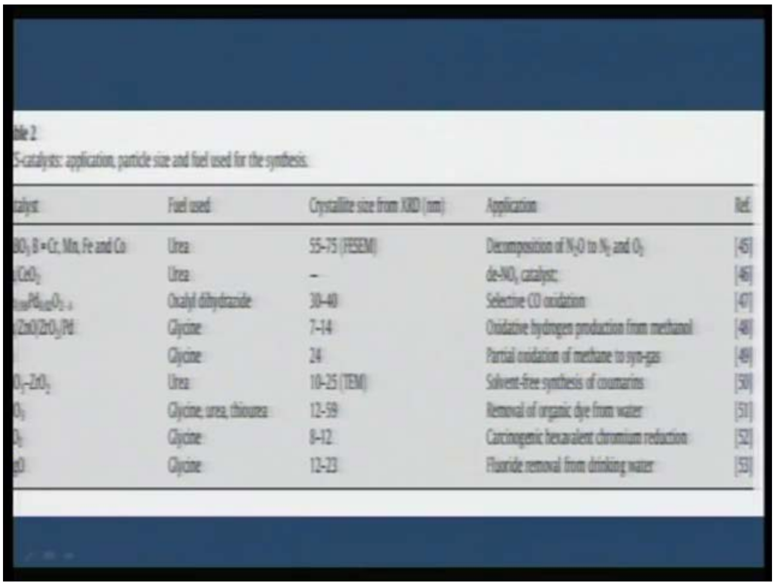

ble 2

S-catalysts: application, particle size and fuel used for the synthesis.

talyst	Fuel used	Crystallite size from XRD (nm)	Application	Ref.
BO$_3$ B = Cr, Mn, Fe and Co	Urea	55–75 (FESEM)	Decomposition of N_2O to N_2 and O_2	[45]
CeO$_2$	Urea	–	de-NO$_x$ catalyst	[46]
$_{0.98}$Pd$_{0.02}$O$_{2-\delta}$	Oxalyl dihydrazide	30–40	Selective CO oxidation	[47]
ZnO)ZrO$_2$/Pd	Glycine	7–14	Oxidative hydrogen production from methanol	[48]
	Glycine	24	Partial oxidation of methane to syn-gas	[49]
O$_2$–ZrO$_2$	Urea	10–25 (TEM)	Solvent-free synthesis of coumarins	[50]
O$_3$	Glycine, urea, thiourea	12–59	Removal of organic dye from water	[51]
O$_3$	Glycine	8–12	Carcinogenic hexavalent chromium reduction	[52]
gO	Glycine	12–23	Fluoride removal from drinking water	[53]

We can go for several such catalysts to get this done.

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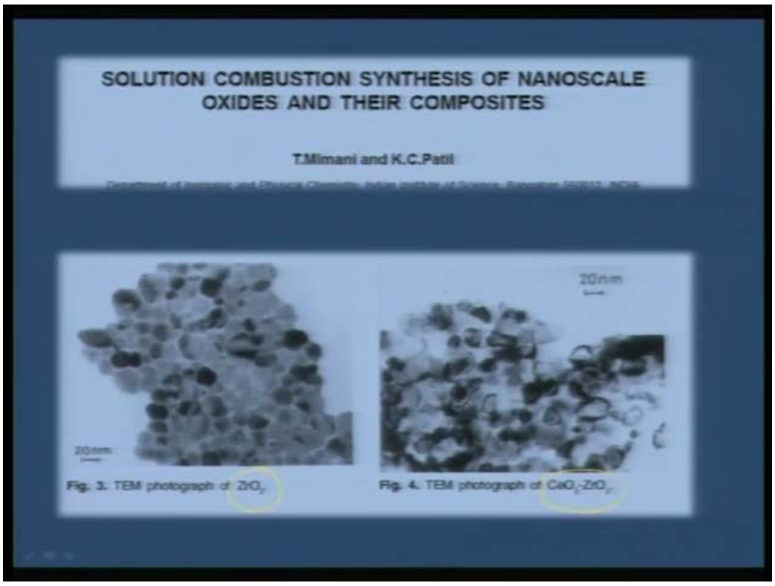

SOLUTION COMBUSTION SYNTHESIS OF NANOSCALE OXIDES AND THEIR COMPOSITES

T.Mimani and K.C.Patil

[illegible]

Fig. 3. TEM photograph of ZrO_2

Fig. 4. TEM photograph of CeO_2-ZrO_2

And similarly, we can make composites of ZrO 2 and CeO2, ZrO2.

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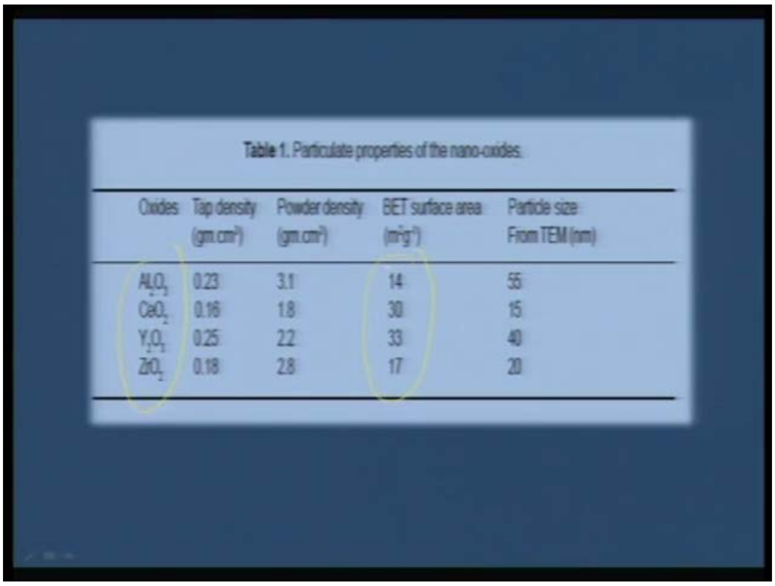

Table 1. Particulate properties of the nano-oxides.

Oxides	Tap density (gm.cm^3)	Powder density (gm.cm^3)	BET surface area (m^{2}g^{-1})	Particle size From TEM (nm)
Al_2O_3	0.23	3.1	14	55
CeO_2	0.16	1.8	30	15
Y_2O_3	0.25	2.2	33	40
ZrO_2	0.18	2.8	17	20

Several such composites can be made of which alumina, ceria, yttria, zirconia, you can see all these powders, which are known to show surface area less than 1 meter per gram, can show such a very high porosity and high surface area.

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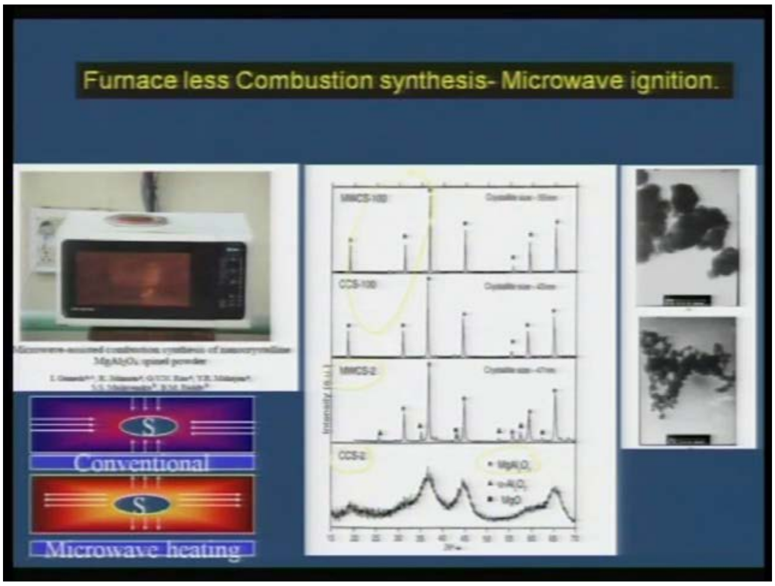
Furnace less Combustion synthesis- Microwave ignition.



Now, just two three slides, I will try to wind up to show that, there are other ways to initiate this combustion, other than merely using a furnace. So, I call this as furnace less combustion synthesis, which is actually microwave ignited. If you do not have a furnace, you can use a simple kitchen microwave to initiate this process. Advantages, you are generating the heat from within the solution mixture, rather than heating from outside and you can clearly see that, in this the mass also plays role. Instead of using a conventional synthesis, you can use a microwave synthesis and in 2 gram quantity, this is the way the crystalline magnesium aluminate formed. But, when you use 100 gram quantity, you can get a crystalline form, this is one reaction.

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Combustion Synthesis -

One project in the field of combustion synthesis is currently being co-ordinated by ESA.

COSMIC

Combustion Synthesis under Microgravity Conditions

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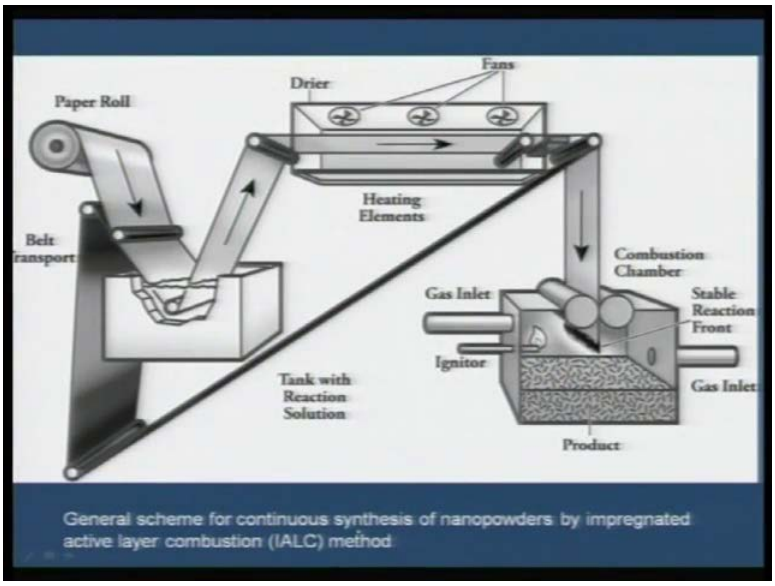

General scheme for continuous synthesis of nanopowders by impregnated active layer combustion (IALC) method

And I can show you another example, this is another thing that is happening, which is of interest, where combustion reactions are experimented in micro gravity. Therefore, for space applications, you need to study at zero gravity, how this combustion proceeds. And NASA is particularly taking this as a active program, to consider how combustion synthesis can occur at zero gravity or microgravity. And this combustion reaction can also be used for making a reel to reel impregnation of this nano powders into several layers coatings. So, this is not only done in lab scale, but this can be done also in a industrial level.

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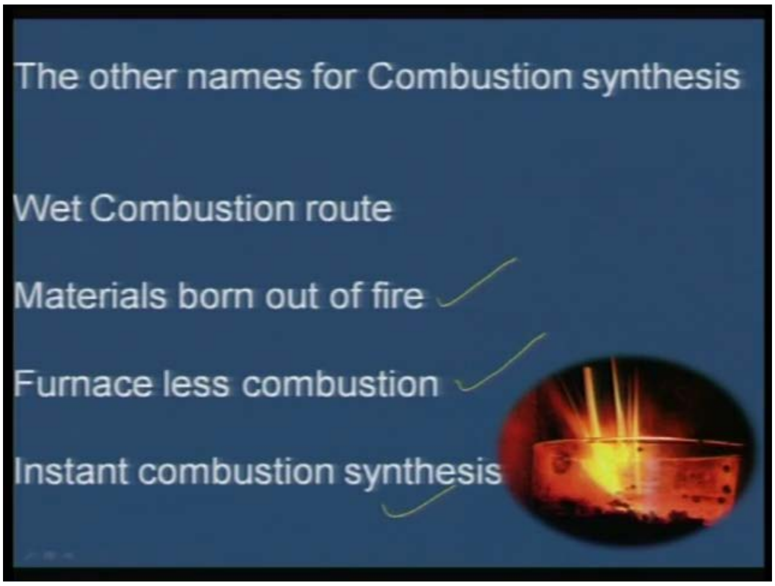

The other names for Combustion synthesis

Wet Combustion route

Materials born out of fire

Furnace less combustion

Instant combustion synthesis

Lastly, I just want to conclude that, this combustion process has gone into many spears, now it is being referred with many names, it is called popularly wet combustion route. Some people call this as materials born out of fire and this is also referred to as furnace less combustion and instant combustion synthesis. So, there is lot of potential for many more interesting physical and chemical properties to be studied using this simple combustion technique.