Upstream LNG Technology
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Lecture – 51
Sulfur recovery in natural gas systems- I

Welcome. Today, we are going to look into the Sulfur recovery in the natural gas system. As we learned earlier that sulfur in comes in terms of H 2 S in natural gas. And under acid gas removal, we learned the techniques to remove the H 2 S from the natural gas. But, also we understand that sulfur is a very important component in manufacture of many chemicals. So, the recovered H 2 S is not thrown out and also H 2 S is not good for the environment. It has many of the deleterious effects which we learned about in our earlier lecture. And natural gas is the source of large amount of the sulfur. So, instead of throwing out the H 2 S what we do is that we try to recover the sulfur from the H 2 S. So, we have separate recovery systems from the H 2 S to recover the sulfur.

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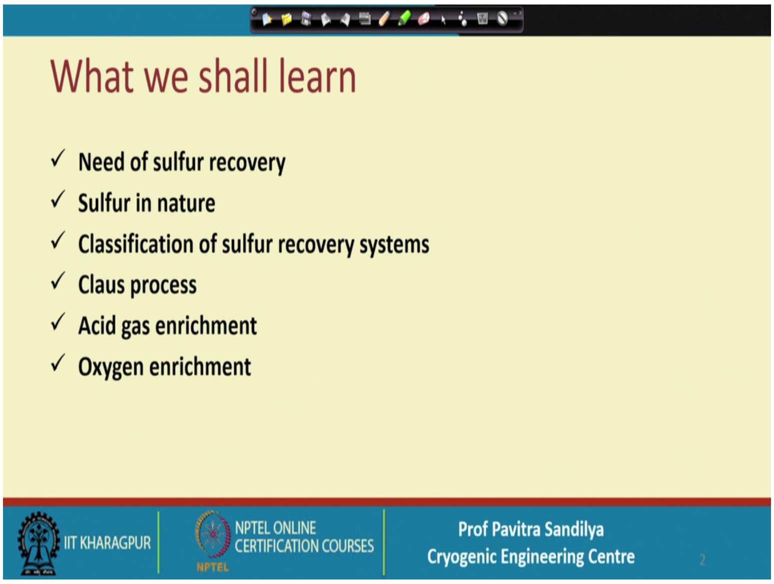

What we shall learn

- ✓ Need of sulfur recovery
- ✓ Sulfur in nature
- ✓ Classification of sulfur recovery systems
- ✓ Claus process
- ✓ Acid gas enrichment
- ✓ Oxygen enrichment

So, in this particular lecture, what is we shall learning about is the need of the sulfur recovery. The nature of sulfur, because this dictates the type of the reacted we shall be using. And sulfur is very peculiar compound about which we shall be seeing later. Then, classification of the sulfur recovery systems, Claus process and acid gas enrichment and oxygen enrichment. So, in this particular the lecture, we shall be looking into some of the recovery processes for sulfur.

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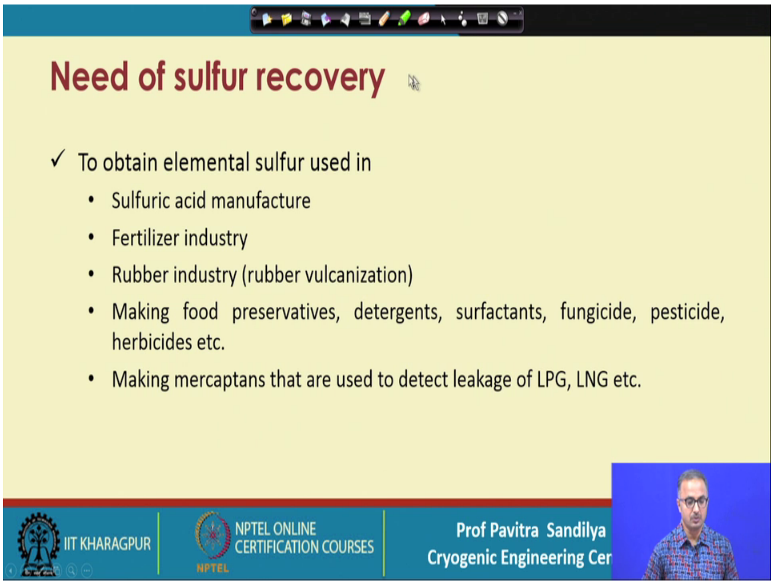

Need of sulfur recovery

✓ To obtain elemental sulfur used in

- Sulfuric acid manufacture
- Fertilizer industry
- Rubber industry (rubber vulcanization)
- Making food preservatives, detergents, surfactants, fungicide, pesticide, herbicides etc.
- Making mercaptans that are used to detect leakage of LPG, LNG etc.

So, first let us see why do we need to recover sulfur because to obtain elemental sulfur which is used in the sulfuric acid manufacture and fertilizer industry.

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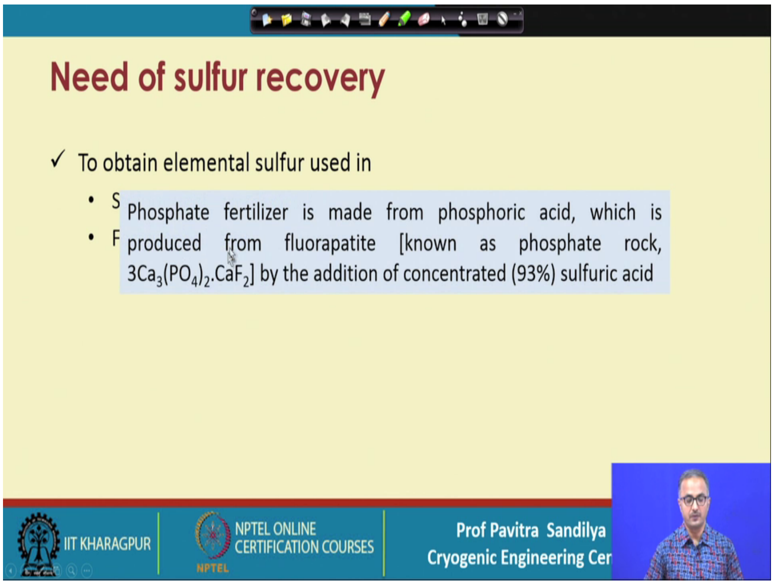

Need of sulfur recovery

✓ To obtain elemental sulfur used in

- S
- F

Phosphate fertilizer is made from phosphoric acid, which is produced from fluorapatite [known as phosphate rock, $3Ca_3(PO_4)_2.CaF_2$] by the addition of concentrated (93%) sulfuric acid

In the fertilizer industry, the phosphate fertilizer is made from phosphoric acid which is produced from fluorapatite which is the phosphate rock with this particular formula. And in this we add very highly concentrated sulfuric acid. So, we find that indirectly we depend on the sulfur to make the phosphate fertilizer. So, we will also need to get this sulfur.

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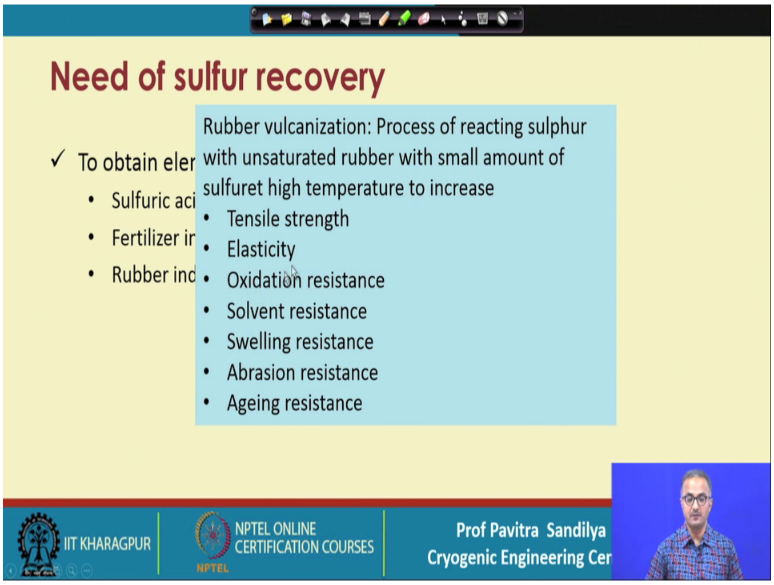

Need of sulfur recovery

✓ To obtain eler

- Sulfuric aci
- Fertilizer ir
- Rubber inc

Rubber vulcanization: Process of reacting sulphur with unsaturated rubber with small amount of sulfuret high temperature to increase

- Tensile strength
- Elasticity
- Oxidation resistance
- Solvent resistance
- Swelling resistance
- Abrasion resistance
- Ageing resistance

Next is the in the rubber industry for the vulcanization of the rubber. And vulcanization is a process of reacting sulfur with unsaturated rubber which is generally sticky in the natural form with small amount of sulfur at high temperature to increase of tensile strength, elasticity, oxidation resistance, solvent resistance, swelling resistance, abrasion resistance and ageing. So, we are trying to enhance the quality of the rubber. So, we are trying to react with small amount of sulfur.

Now, what we do that after this we have some other also this needs of the sulfur. It sulfur is used to make preservative, detergent, surfactants, fungicide, pesticides, herbicide etcetera. And we also use sulfur to make mercaptans which are added to detect the leakage of the LPG, LNG etcetera. So, form various point of view of the for the synthesis of various types of chemicals, this sulfur is very important; and natural gas is the major source of sulfur.

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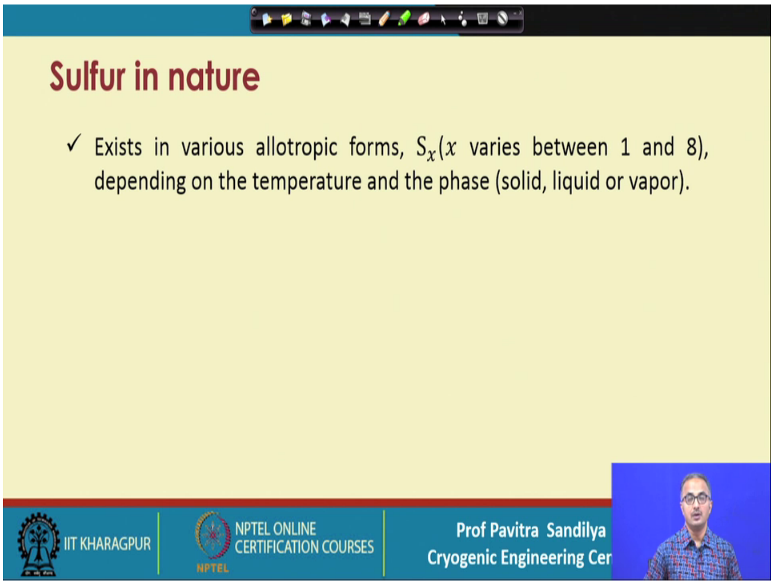

Sulfur in nature

- ✓ Exists in various allotropic forms, S_x(x varies between 1 and 8), depending on the temperature and the phase (solid, liquid or vapor).

Now, before we go on to find out the various methods of recovery, you have to understand the nature of the sulfur, because, this sulfur is a peculiar component and it exists in various allotropic forms. And this has this allotropic means that it has the number of sulfur atoms in an agglomeration may vary with temperature and the phase. So, we find that we are writing representing it in terms of S x and this x may vary between 1 and 8; that means, S 1 means only 1 sulfur atom; S 2 means 2 sulfur atoms; S 3 means 3 sulfur atoms and so on and so forth. So, maximum it can stay in agglomerate with 8 sulfur atoms and which of these forms will exist will be depending on the temperature and the kind of phases that is solid, liquid or vapor, so that means sulfur can exist in all the three phases.

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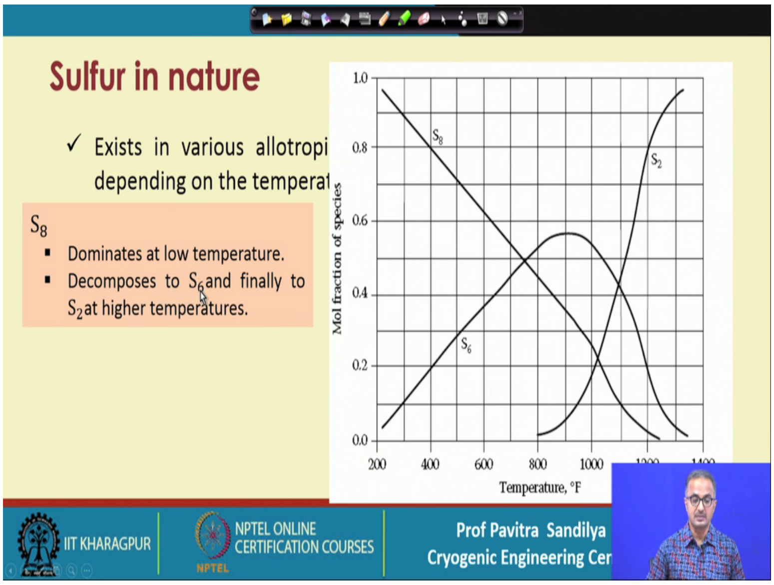

Sulfur in nature

- ✓ Exists in various allotropi depending on the temperat

S_8

- Dominates at low temperature.
- Decomposes to S_6 and finally to S_2 at higher temperatures.

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So, here is one typical representation of the mole fraction of the sulfur with different temperatures. And we find that at the lower temperatures, the acid component is predominating the S 6 and S 2, these are the main allotropic forms of the sulfur. So, acid form is most prevalent at the higher temperature. And as we increase the temperature, we find that the amount of S 6 increases initially; and then at same temperature the S 6 predominates S 8. And then we find also that S 2 starts forming at some still higher temperature; and ultimately we find at very high temperature S 2 is the most prevalent allotropic form of the sulfur.

So, whenever we are carrying out any kind of reaction to recover the sulfur depending on the reaction temperature, we shall be getting either of these forms allotropes. And, because we the direct change in the allotropic forms of this sulfur, what we find the conversion rate of the sulfur also changes. So, S 8 dominates at low temperature and decomposes to S 6; and finally, to S 2 at higher temperatures.

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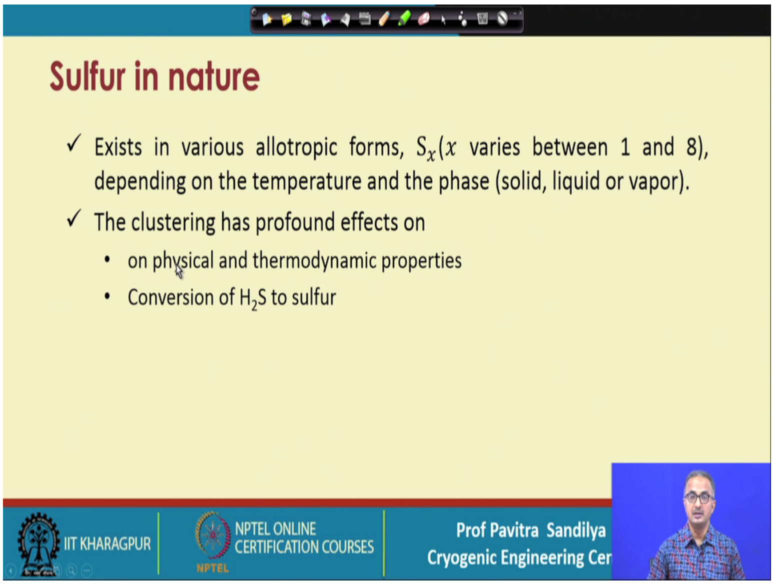

Sulfur in nature

- ✓ Exists in various allotropic forms, S_x(x varies between 1 and 8), depending on the temperature and the phase (solid, liquid or vapor).
- ✓ The clustering has profound effects on
  - on physical and thermodynamic properties
  - Conversion of H_2S to sulfur

And depending on the clustering of the sulfur atoms, we will be having different physical and thermodynamic properties. And the conversion rate of the H 2 S to sulfur will also change.

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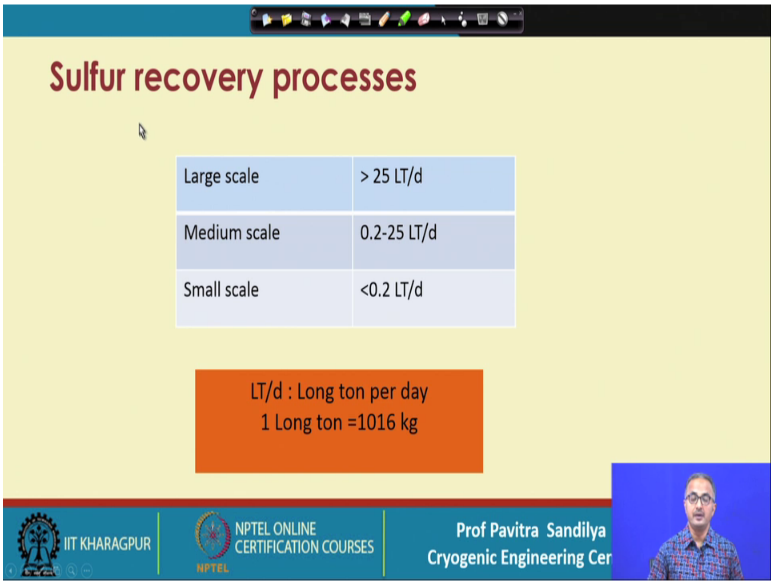

Sulfur recovery processes

Large scale	> 25 LT/d
Medium scale	0.2-25 LT/d
Small scale	<0.2 LT/d

LT/d : Long ton per day
1 Long ton =1016 kg

Under the sulfur recovery process, depending on the capacity, we have divided them into 3 categories large scale, medium scale and small scale. And these has been divided based on this long ton per day. And 1 long ton is defined as 1016 kg. This is in contrast with the ton which is 1000 kg. So, we say that this is 25 long ton per day is more used more than that then we have large scale. And if it is between 0.2 to 25 long ton per day, it is medium scale. And anything less than 0.2 long ton per day is taken to be small scale process.

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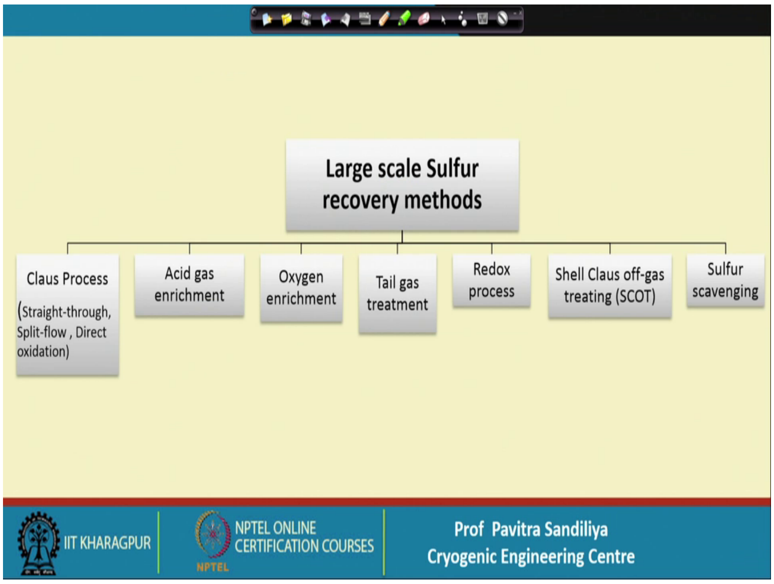

Large scale Sulfur recovery methods

- Claus Process (Straight-through, Split-flow , Direct oxidation)
- Acid gas enrichment
- Oxygen enrichment
- Tail gas treatment
- Redox process
- Shell Claus off-gas treating (SCOT)
- Sulfur scavenging

So, for each of these processes, we have different types of sulfur recovery system. We shall see the various types of sulfur recovery methods on large scale. So, here we have Claus process, acid guest enrichment, oxygen enrichment, tail gas treatment, redox process, shell Claus off-gas off gas treating, that is SCOT process in short and then sulfur scavenging. About the sulfur scavenging, we learned something earlier under the acid gas removal two.

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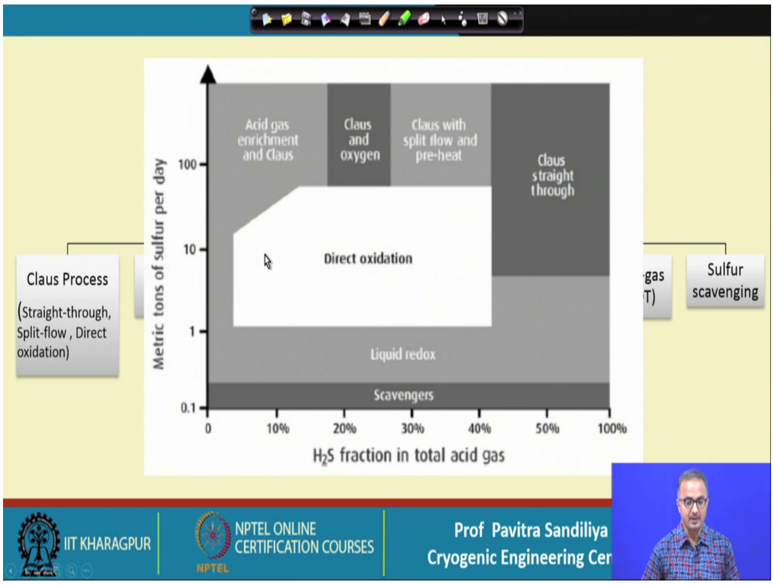

Claus Process

(Straight-through, Split-flow , Direct oxidation)

gas

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Sulfur scavenging

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So, what we find that in this particular graph that it gives us a some kind of guideline to select one or the other of these processes. So, here we find that these graph has been made by taking the H 2 S concentration in the an acid gas from 0 to 100 percent, and then the recovery needed, so on this metric tons of sulfur per day.

Now, we find that how much recovery we want and how much H 2 S is present, we choose one or the other type of processes. In this we find at the lowest recovery, we have the scavengers and we learned at the scavengers that involved some kind of irreversible reactions, but they cannot give us high amount of sulfur recovery, because it is irreversible reaction. So, we find that for but they can spend from very small amount to very large amount of H 2 S in the natural gas, but they give very small recovery. However, if you want to increase the recovery, we have to go for other processes like starting from liquid redox.

And ultimately, find that is Claus process or its variants are important. If you want to go for very high recovery, but we find that why we have different types of Claus process, because this different types are obtained for different H 2 S content. For high H 2 S content that is about more than 40 percent, we have Claus straight through and between this 40 percent to about say 25 percent or so, so we find that we have Claus with split flow and preheat. And then between about 25 to about 15 percent, we have about Claus with oxygen enrichment. And then below this we find that we have some variation of

Claus with acid gas enrichment. And then, if we have some medium recovery, we have direct oxidation process, and still for lower we have this liquid redox process, so that is why we find we have so many variants of the sulfur recovery process is depending on the amount of H 2 S and the amount of recovery needed.

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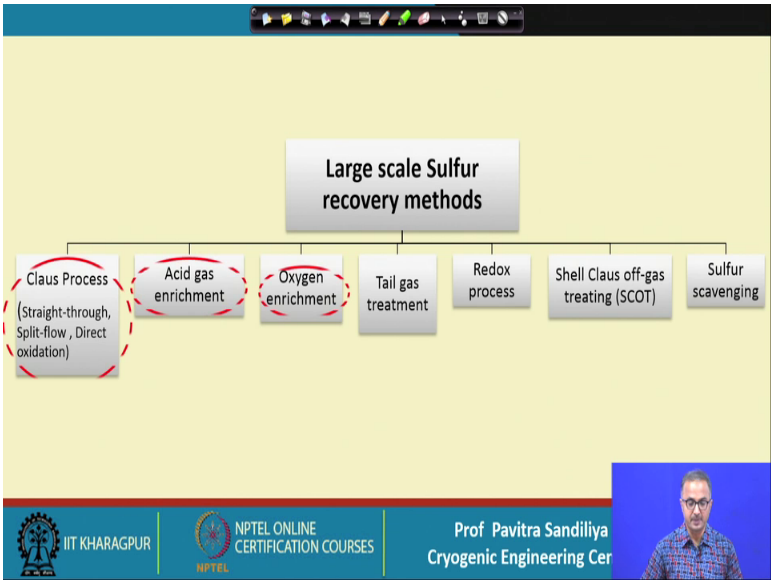

Large scale Sulfur recovery methods

- Claus Process (Straight-through, Split-flow , Direct oxidation)
- Acid gas enrichment
- Oxygen enrichment
- Tail gas treatment
- Redox process
- Shell Claus off-gas treating (SCOT)
- Sulfur scavenging

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So, in this particular lecture, we shall be looking into this three processes rest of them will be taken up separately in a separate lecture.

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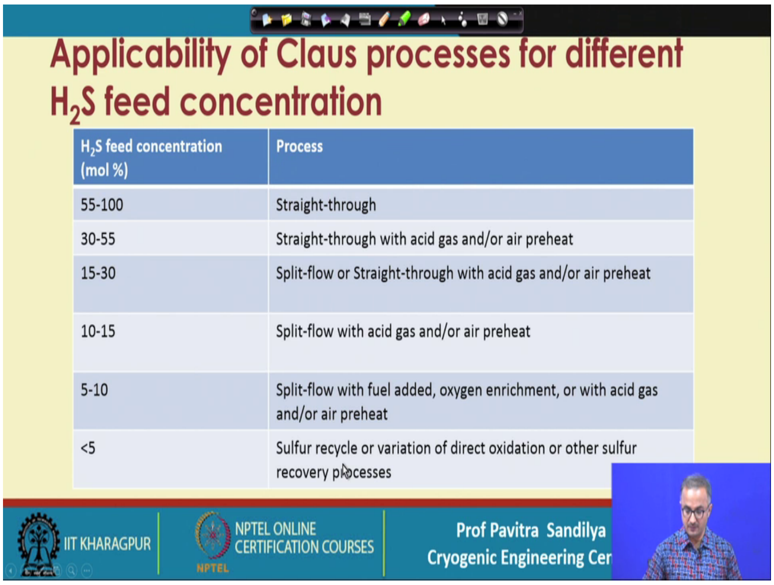

Applicability of Claus processes for different H_2S feed concentration

H_2S feed concentration (mol %)	Process
55-100	Straight-through
30-55	Straight-through with acid gas and/or air preheat
15-30	Split-flow or Straight-through with acid gas and/or air preheat
10-15	Split-flow with acid gas and/or air preheat
5-10	Split-flow with fuel added, oxygen enrichment, or with acid gas and/or air preheat
<5	Sulfur recycle or variation of direct oxidation or other sulfur recovery processes

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So, first let us see that the applicability of this Claus processes for different H 2 S feed concentration which we have seen in the graph. And here we again put them in this table that depending on the various types of composition of the H 2 S in the sour gas we have different methods for the Claus process. So, I am not going to explain these things here we shall be looking into these in our future slides.

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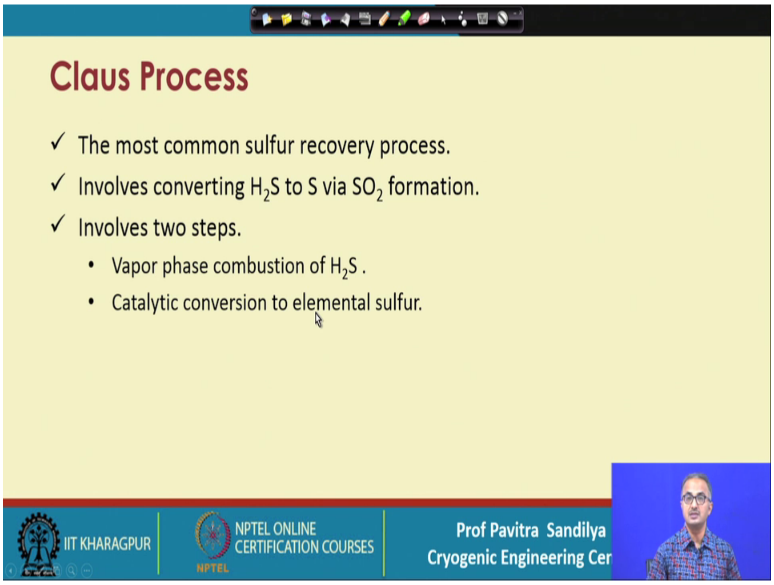

Claus Process

- ✓ The most common sulfur recovery process.
- ✓ Involves converting H_2S to S via SO_2 formation.
- ✓ Involves two steps.
  - Vapor phase combustion of H_2S .
  - Catalytic conversion to elemental sulfur.

So, first let us start with the Claus process. This is the most common sulfur recovery process. And it involves conversion of the H 2 S to sulfur via sulfur dioxide formation. And this goes in two step process. In the first step, what happens that vapor phase combustion of H 2 S happens. And then catalytically whatever sulfur dioxide is formed by the combustion of the H 2 S is converted to elemental sulfur.

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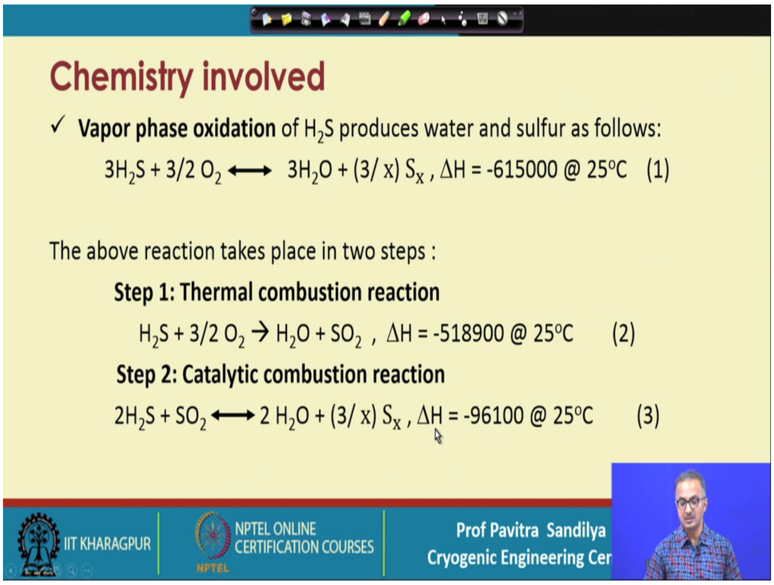

Chemistry involved

✓ **Vapor phase oxidation** of H_2S produces water and sulfur as follows:

$3H_2S + 3/2\ O_2 \longleftrightarrow 3H_2O + (3/x)\ S_x$, $\Delta H = -615000$ @ 25°C (1)

The above reaction takes place in two steps :

Step 1: Thermal combustion reaction

$H_2S + 3/2\ O_2 \rightarrow H_2O + SO_2$, $\Delta H = -518900$ @ 25°C (2)

Step 2: Catalytic combustion reaction

$2H_2S + SO_2 \longleftrightarrow 2\ H_2O + (3/x)\ S_x$, $\Delta H = -96100$ @ 25°C (3)

So, let us see the chemistry involved behind this recovery process. So, in the vapor phase oxidation of the H 2 S, we find this is the overall process that is H 2 S goes to oxygen, and it gives out H 2 O and sulfur. And this has this is a heat of the reaction at 25 degrees centigrade. But this reaction takes place in two steps. First is a thermal combustion in which S 2 S reacts with oxygen to give H 2 O and S O 2 with this heat of reaction here at 25 degrees centigrade. And then in the second step catalytically this H 2 S and S O 2 react to give us again this sulfur and H 2 O. Now, in this case, the rate of reaction is this heat of reaction is this. So, we find that this first one is the one which required oxygen, and this oxygen is generally supplied through air.

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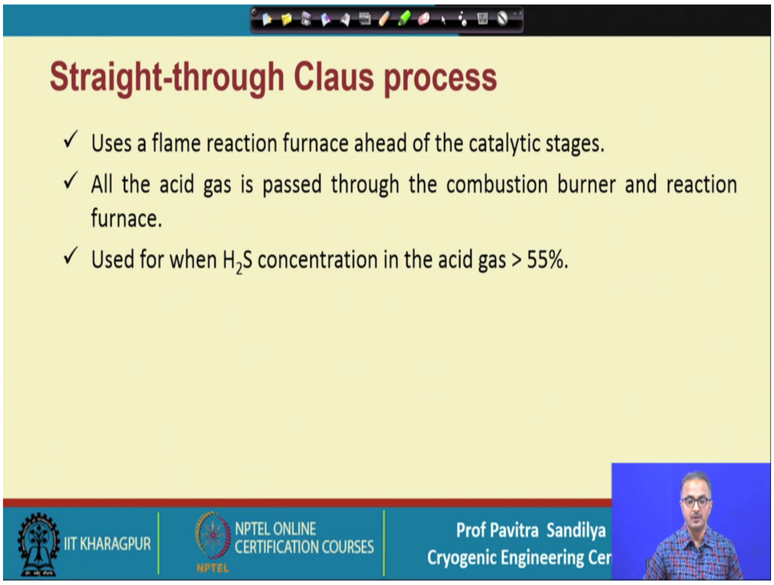

Straight-through Claus process

- ✓ Uses a flame reaction furnace ahead of the catalytic stages.
- ✓ All the acid gas is passed through the combustion burner and reaction furnace.
- ✓ Used for when H_2S concentration in the acid gas > 55%.

In the straight Claus through process it there is some flame reaction is there in a furnace ahead of the catalytic stages. And all the acid gas is passed through the combustion burner, and reaction furnace. This is quite important. In this case, we find the whole amount of the acid gas which is coming is taken to the furnace and then to the catalytic reactor. And this is used when the H 2 S concentration is more than about 50 to 55 percent.

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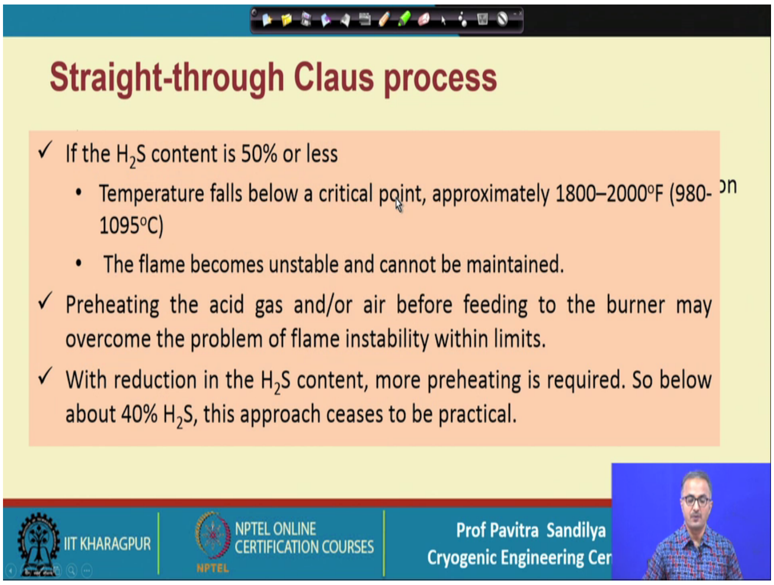

Straight-through Claus process

- ✓ If the H_2S content is 50% or less
  - Temperature falls below a critical point, approximately 1800–2000°F (980-1095°C)
  - The flame becomes unstable and cannot be maintained.
- ✓ Preheating the acid gas and/or air before feeding to the burner may overcome the problem of flame instability within limits.
- ✓ With reduction in the H_2S content, more preheating is required. So below about 40% H_2S, this approach ceases to be practical.

And why this so, because for conversion of these H 2 S, 2 S O 2 we need some minimum temperature of 982 1095 degree centigrade, but at lower than this temperature the flame becomes unstable. And this unstable because the amount of H 2 S comes down, so that enough feed is not generated. So, the flame cannot be maintained so, we cannot go for the oxidation of the H 2 S to S O 2. So, in this case, we need to preheat the acid gas, and or the air that is a source of the oxygen before we feed it to the burner; and this may to some extent take care of the flame instability problem. Now as the amount of H 2 S decreases what we find the, at certain amount like about 40 percent and less, we are not able to make the flame stable. So, the practicability of this particular straight-through Claus process see this below 40 percent of H 2 S.

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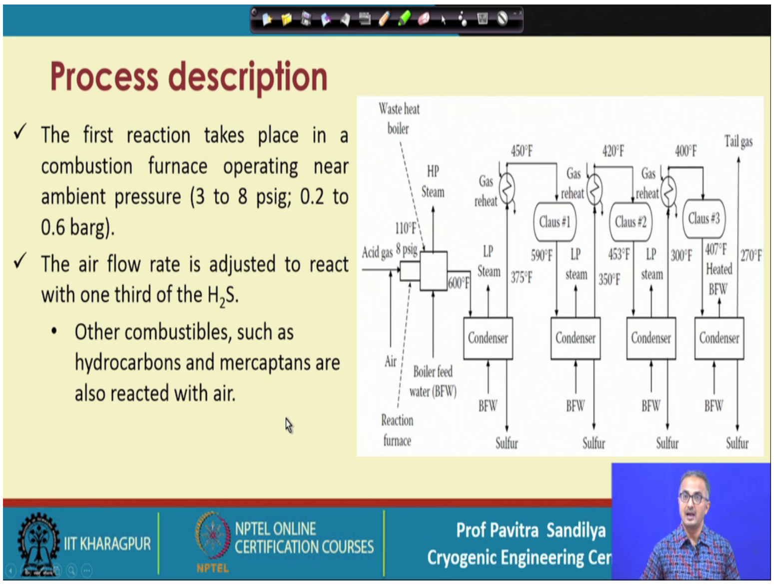

Process description

- ✓ The first reaction takes place in a combustion furnace operating near ambient pressure (3 to 8 psig; 0.2 to 0.6 barg).
- ✓ The air flow rate is adjusted to react with one third of the H_2S.
  - Other combustibles, such as hydrocarbons and mercaptans are also reacted with air.

Now, here is an overall process diagram of the state-through Claus process. Here we find we have the acid gas at about 3 to 8 P s i g about 0.2 to 0.6 bar gauge that means, it is almost nearly the atmospheric pressure. And this is mixed with the air that is a source of the oxygen. And, this whole thing goes to the burner and in which we are putting some boiler feed water also to make it steam with the heat of reaction that is generated being the conversion of H 2 S to S O 2. And there is some other combustibles also like hydrocarbons like BTEX that is benzene, toluene, xylene, etcetera, and some mercaptans they also react with the air and get decomposed.

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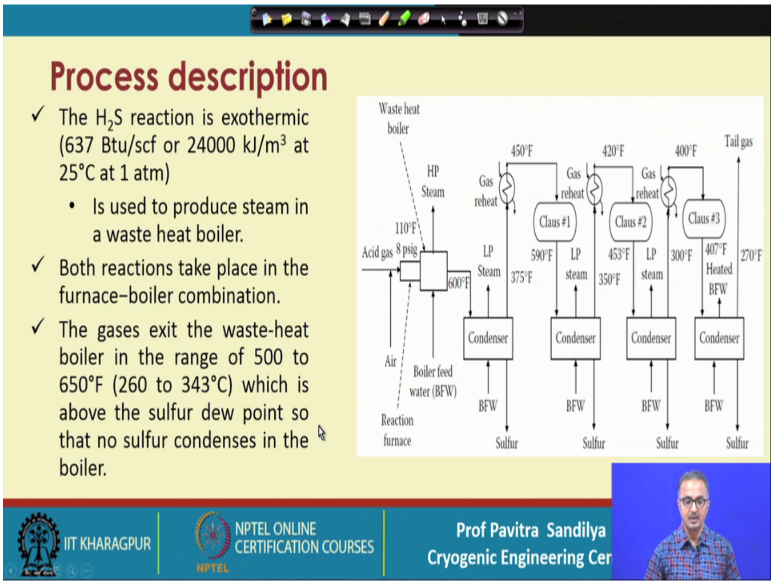

Process description

- ✓ The H_2S reaction is exothermic (637 Btu/scf or 24000 kJ/m^3 at 25°C at 1 atm)
  - Is used to produce steam in a waste heat boiler.
- ✓ Both reactions take place in the furnace–boiler combination.
- ✓ The gases exit the waste-heat boiler in the range of 500 to 650°F (260 to 343°C) which is above the sulfur dew point so that no sulfur condenses in the boiler.

And what happens after that that this H 2 S burns exothermically and the heat is taken for making the steam for the for the boiler feed water. And then the exit gate is about 500 to 65 degree Fahrenheit that is about 260 to 343 centigrade which is above the sulfur dew point and that is why no sulfur condenses in the boiler.

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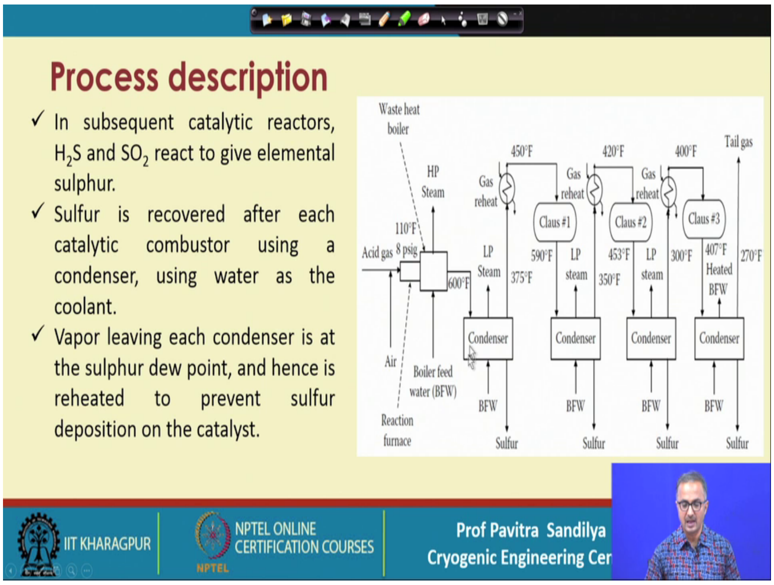

Process description

- ✓ In subsequent catalytic reactors, H_2S and SO_2 react to give elemental sulphur.
- ✓ Sulfur is recovered after each catalytic combustor using a condenser, using water as the coolant.
- ✓ Vapor leaving each condenser is at the sulphur dew point, and hence is reheated to prevent sulfur deposition on the catalyst.

After this what happens that they are taken to condenser. And in the condenser, some of the sulfur is recovered. And then the rest of the gas is taken to the this Claus reactor which are nothing per some catalytic reactor. And we find that after each of these reactors, this there is some or some amount of sulfur is recovered. And from the condenser, we are able to recover the sulfur. And ultimately in the tail gas of the recovery, we find that here the tail gas which has less then less H 2 S then what we had is the initial feed acid gas.

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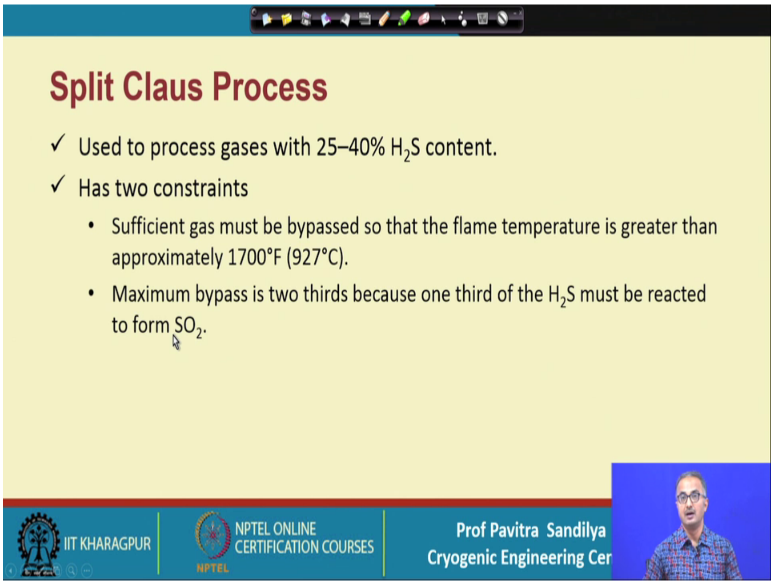

Split Claus Process

- ✓ Used to process gases with 25–40% H_2S content.
- ✓ Has two constraints
  - Sufficient gas must be bypassed so that the flame temperature is greater than approximately 1700°F (927°C).
  - Maximum bypass is two thirds because one third of the H_2S must be reacted to form SO_2.

Now, as we said that because this cannot work below 40 percent; in that case we use the split Claus process and this is process used for 25 to 40 percent of H 2 S content. Now, in this we have two constraints, one constraint is that the sufficient gas must be bypassed that means, we are not passing the whole gas into the furnace only part of is sent to the furnace and rest of feed is send directly to the catalytic reactors.

So, what happens that if we do not send enough H 2 S, so what happens that the flame temperature is greater than approximate this. So, the gas must be bypassed so that the flame temperature is greater than this 927 degree centigrade. And then the maximum bypass is about two-thirds because one-thirds of H 2 S must be reacted to form S O 2 that means we need S O 2 to recover or to get sulfur. So, we need to see that what is the minimum amount that can be put to the furnace.

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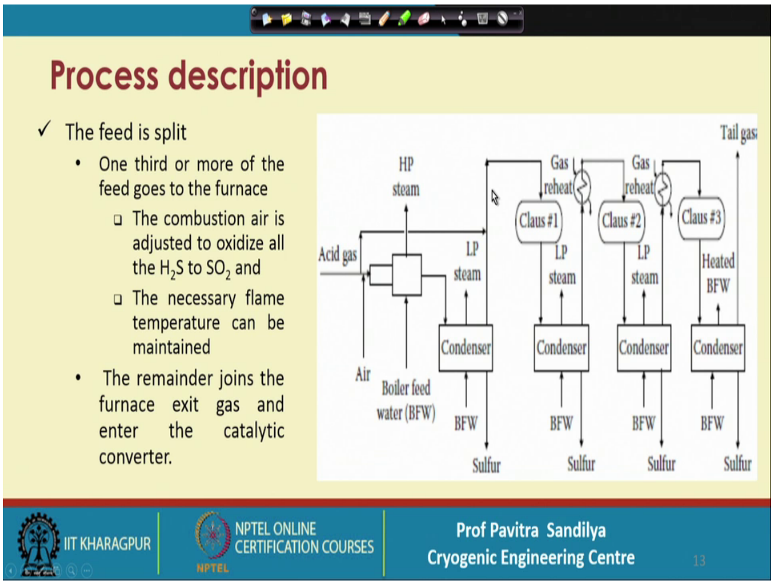

Process description

- ✓ The feed is split
  - One third or more of the feed goes to the furnace
    - ❑ The combustion air is adjusted to oxidize all the H_2S to SO_2 and
    - ❑ The necessary flame temperature can be maintained
  - The remainder joins the furnace exit gas and enter the catalytic converter.

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So, here we find that the acid get comes and with split over here say about two-third that is most of it goes is bypass. And only a small fraction that can be one-third goes into the furnace which is now reacted with the air. And this combustion of the air is adjusted to oxidize all the H 2 S to S O 2. And also the flame temperature that is required is also maintained by this particular reactions. So, these goes again here and again we find the heat of reaction is used to make steam high pressure steam from the boiler feed water. And it goes here and we get some amount of sulfur recovered here. And then what you find this gas which is coming out is mixed with the feed gas. Now, it goes to the catalytic reactor.

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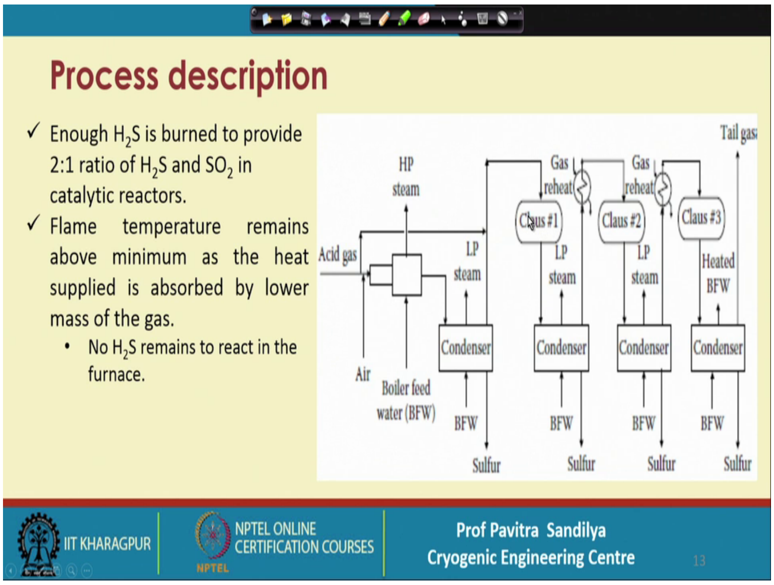

Process description

- ✓ Enough H_2S is burned to provide 2:1 ratio of H_2S and SO_2 in catalytic reactors.
- ✓ Flame temperature remains above minimum as the heat supplied is absorbed by lower mass of the gas.
  - No H_2S remains to react in the furnace.

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And after that this be maintained this required ration that is 2 is to 1 ratio of H 2 S, and S O 2 that is (Refer Time: 18:12) of stoichiometry at 2 in the catalytic reactor. We are typically using three catalytic reactors. And after each catalytic reactor, we are able to recover some amount of sulfur and ultimately we have the tail gas coming out from the last condenser, so that is how we are carrying out the split Claus process.

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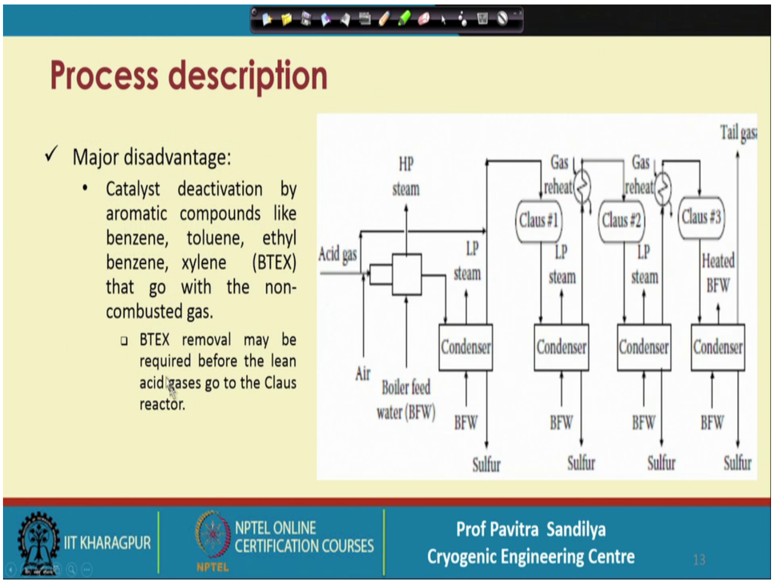

Process description

- ✓ Major disadvantage:
  - Catalyst deactivation by aromatic compounds like benzene, toluene, ethyl benzene, xylene (BTEX) that go with the non-combusted gas.
    - ❑ BTEX removal may be required before the lean acid gases go to the Claus reactor.

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The major disadvantage of this is, because the large amount the majority of the gas is going bypassed. So, we find that all the hard hydrocarbons like benzene, toluene, xylene, ethyl benzene etcetera; we call them BTEX in short. They also go along with this to the catalytic reactor. And what happens they do not get decomposed, and they may be settling on the catalyst here by deactivating the catalyst. And we need to have some kind of removal equipment before we send it to the Claus reactor that will be add to the capital cost of the whole process, but these becomes inevitable if we are going for, less than 40 percent H 2 S.

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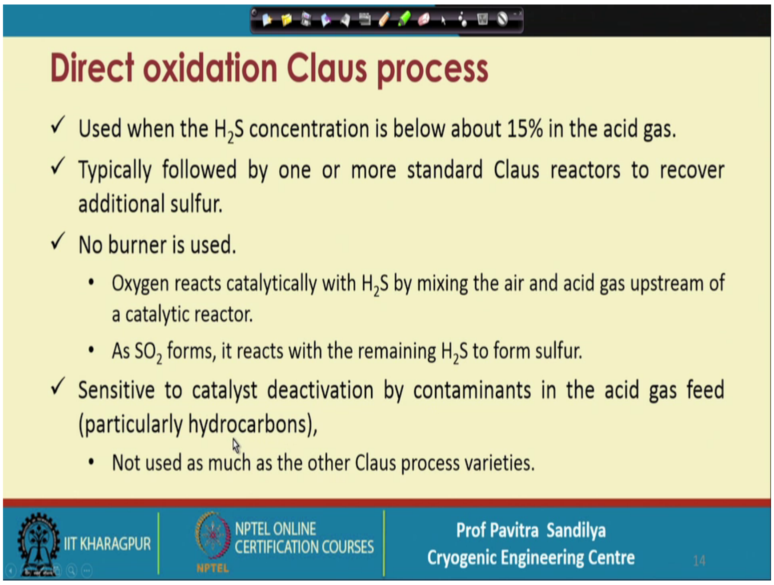

Direct oxidation Claus process

- ✓ Used when the H_2S concentration is below about 15% in the acid gas.
- ✓ Typically followed by one or more standard Claus reactors to recover additional sulfur.
- ✓ No burner is used.
  - Oxygen reacts catalytically with H_2S by mixing the air and acid gas upstream of a catalytic reactor.
  - As SO_2 forms, it reacts with the remaining H_2S to form sulfur.
- ✓ Sensitive to catalyst deactivation by contaminants in the acid gas feed (particularly hydrocarbons),
  - Not used as much as the other Claus process varieties.

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Next, we have the direct oxidation process in this the amount of H 2 S is less than 15 percent in the acid gas. And this is generally followed by one or more standard Claus reactors to recover the additional sulfur. Here we do not use any burner. Oxygen reacts catalytically with H 2 S by mixing with air and acid gas upstream of the catalytic reactor. And then whatever S O 2 is formed as it is formed it is reacting with the H 2 S to form sulfur that is there is no stepwise as it H 2 S, S O 2 is getting formed it is reacting with H 2 S.

And it is sensitive to catalyst deactivation, because the contaminants like the hydrocarbons like in the split Claus process, these hydrocarbons are not getting decomposed separately, so that them a again deactivate the catalyst. And it is not used as much as the other Claus process varieties.

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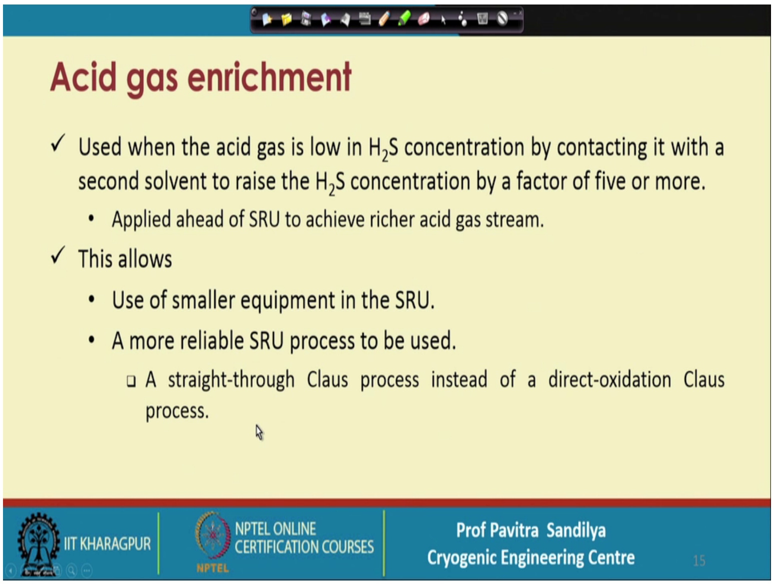

Acid gas enrichment

- ✓ Used when the acid gas is low in H_2S concentration by contacting it with a second solvent to raise the H_2S concentration by a factor of five or more.
  - Applied ahead of SRU to achieve richer acid gas stream.
- ✓ This allows
  - Use of smaller equipment in the SRU.
  - A more reliable SRU process to be used.
    - ❑ A straight-through Claus process instead of a direct-oxidation Claus process.

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Next we have the acid gas enrichment. In this, what we do the acid gas when the amount is very very low. So, we have to enrich the acid gas to increase its H 2 S content and that is done by contacting the acid gas with a second solvent to raise the H 2 S concentration by a factor of five or more. And, this is applied ahead of the sulfur recovery unit to achieve richer acid gas stream. Now, by increasing the amount of the H 2 S, the use of smaller equipment results and a more reliable SRU process is to be used. And then what happens once we increase the H 2 S content in the natural gas, now we can use some straight through Claus process instead of direct oxidation Claus process.

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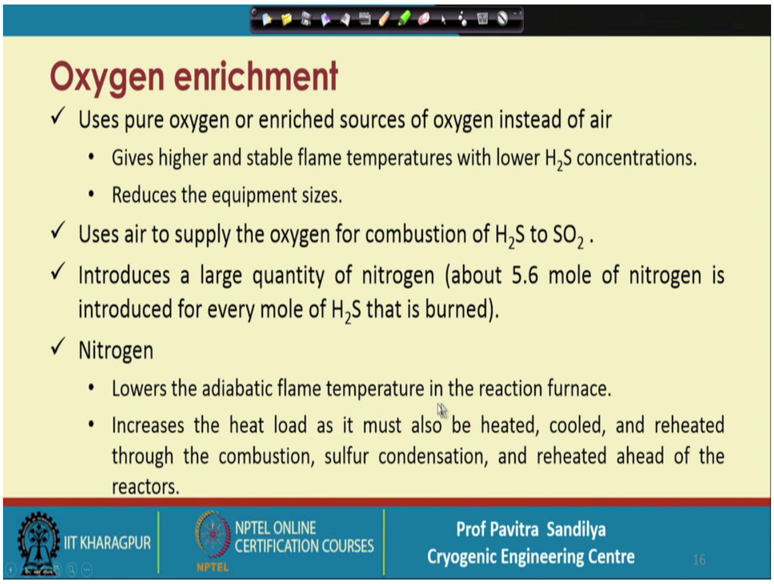

Oxygen enrichment

- ✓ Uses pure oxygen or enriched sources of oxygen instead of air
  - Gives higher and stable flame temperatures with lower H_2S concentrations.
  - Reduces the equipment sizes.
- ✓ Uses air to supply the oxygen for combustion of H_2S to SO_2 .
- ✓ Introduces a large quantity of nitrogen (about 5.6 mole of nitrogen is introduced for every mole of H_2S that is burned).
- ✓ Nitrogen
  - Lowers the adiabatic flame temperature in the reaction furnace.
  - Increases the heat load as it must also be heated, cooled, and reheated through the combustion, sulfur condensation, and reheated ahead of the reactors.

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Now, we also may use oxygen enrichment in this means that we are using pure oxygen all enriched oxygen that is not the ordinary air which contains about 21 percent oxygen, we can also use enriched air that means it can enrich to 50 to 80 percent oxygen or pure oxygen. And this gives higher and stable flame temperature with lower H 2 S concentration that means, even if the amount of H 2 S is less, because, we are using quite pure oxygen that means with less dilution from nitrogen because that is the another major component of air. So, what we find because of less dilution by nitrogen, we are able to get higher and stable flame temperature with lower concentration of H 2 S.

And this reduces the equipment size, but on the other hand getting purer oxygen involves more cost. Now, this air is used to supply the oxygen for combustion of H 2 S to S O 2. And then introduce a large quantity of nitrogen that means that is why we need to enrich with oxygen and nitrogen lowers the adiabatic flame temperature in the furnace, and into the heat load that is why we use the purer oxygen so that we can avoid the dilution effect by nitrogen.

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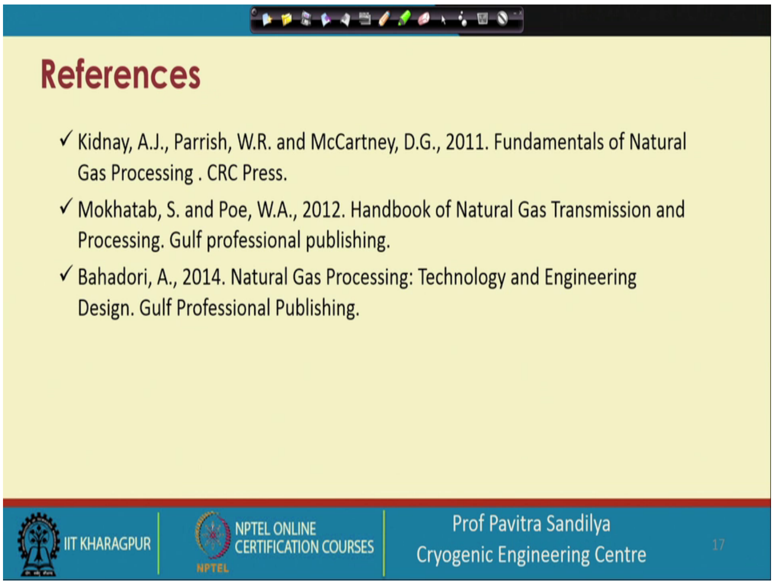

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And these are the references which gives more detail on this sulfur recovery processes.

Thank you.