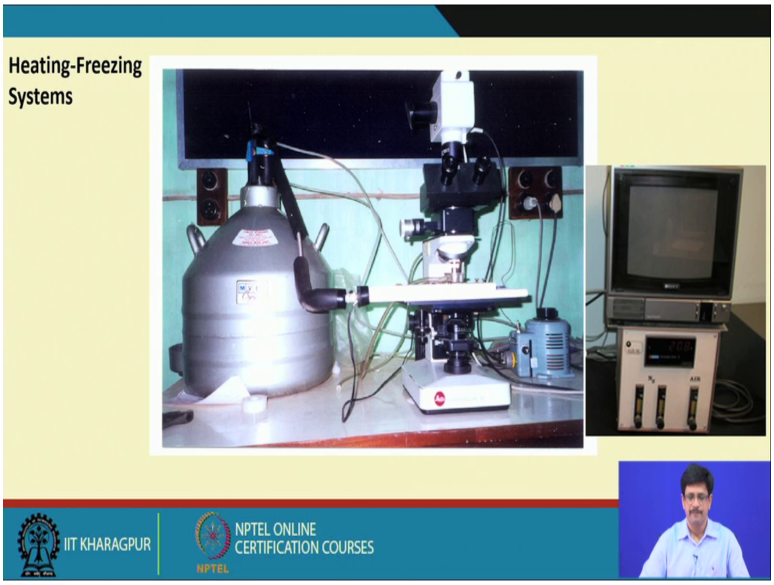
Fluid Inclusion in Minerals: Principles, Methodology, Practice and Application
Prof. M K Panigrahi
Department of Geology and Geophysics
Indian Institute of Technology, Kharagpur

Lecture - 07
Microthermometry: Principles

Welcome to the to the session of fluid inclusions in minerals. In this module we will be discussing about the principles of microthermometry. And, in the last class we discussed about certain specifications, requirements that these heating freezing systems must met must qualify for the for carrying out the microthermometry experiments on this fluid inclusions.

We discussed about their certain specification in terms of there range of operation, the thermal response is have operation changing from freezing to heating mode, and many of the other aspects which we discussed in the last class.

(Refer Slide Time: 01:10)



Will quickly have a view of a these microthermometric apparatus or which are the microscopic heating freezing systems which are mounted on microscopes.

There are many such commercially available models which will find in any of the research laboratories carrying out fluid inclusion work anywhere. And without getting into too much of the minor details about this equipment which one can experience when the when was starts working with them. So, this one which is here in this figure this picture it is this is a heating gas flow a heating compression system.

Originally designed by the United States geological survey and is adopted by the fluid incorporated in Denver Colorado united states of America. And the basic operation of this heating freezing system is that the freezing is done by a by cold nitrogen gas, the nitrogen gas is it comes from a commercially available nitrogen gas cylinder.

And this which is the container of liquid nitrogen there liquid nitrogen dewar within that there is a copper coil assembly and the dry nitrogen gas is made to circulate through this assembly and is controlled by the flow meter over here.

Which makes the nitrogen gas flow at a specific rate, and by which the gas nitrogen gas gets cooled and it can see well insulated delivery tube; which is connected to the heating freezing system, which is your ceramic stage and within which the sample hold assembly chamber is placed. Within that chamber the fluid inclusion are the vapor the w for is thin section in which we study the fluid inclusions is kept, and the heating is done.

So, within this there is a heating element, and the heating is done by passing comprised air and by applying desired current, the heating element gets heated up and the sample is the sample chamber gets heated. And the temperature is recorded by this device which is a temperature indicator or say to indicator. And so this the system the way it works that be the attachment for heating and freezing the different.

So, once the freezing is done or freezing is freezing work is completed, then the this cold nitrogen gas delivery tube is detached and is and the hot comprised air tube is attached with and the heating is done. And the temperature is temperature of any phase change as you can as we see through the microscope or if this microscope is fitted with a digital camera and on a computer screen or on a display video display unit. We can record the phase changes, and record the temperature from reading out from the value here.

And this systems need to be calibrated with available standards. The synthetic fluid inclusion standards these there are available which can be a pure water or a carbon pure carbon dioxide or inclusion stapret any different pressure temperature, conditions or even there are certain other type of metallic standards with known melting point. Which can also be used to calibrate this equipment and this kind of period calibration is essential to record the fluid inclusion thermometric observations.

So, this is essentially known as the gas flow heating compressing system and this popularly known as the Renaults stage. Named on the by the by the person who has fabricated this equipment.

(Refer Slide Time: 05:50)

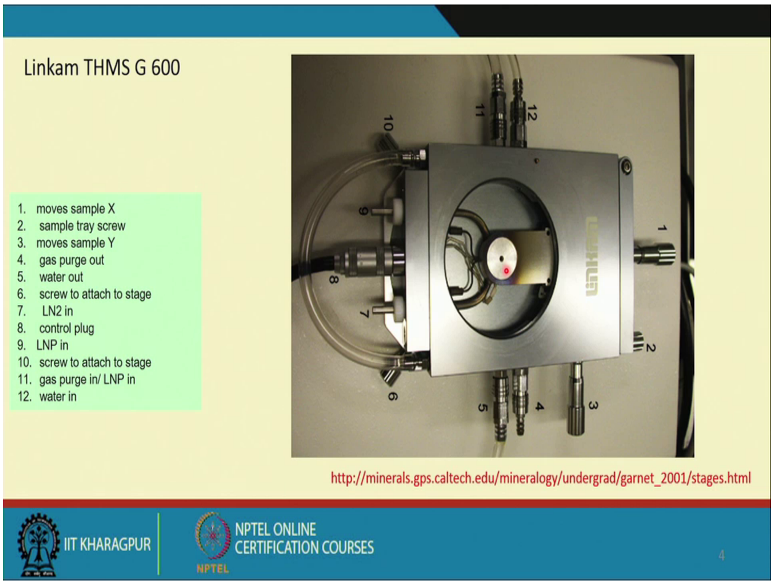
The other one which is also quite popular is the linkam THMS G 600 microscope heating the hot and cold stage. So, this particular Renault stage it works in the range of minus 196 degree, Celsius that is the temperature at which nitrogen boils at atmospheric pressure say above 196-degree centigrade or minus 76 Kelvin.

So, this and the depending on the specification on the capacity of this heating element by applying the current through this variable transformer the temperature range of operation can go to minus 196 to plus 700 degree Celsius. And there are there are devices which can be used to avoid the condenses enough moisture when the stage is cool to sub 0 temperatures as well as to also protect the objective when the temperature goes above 500-degree Celsius temperature.

So, this particular stage also works more or less in the similar manner, but the only difference is that it does not need to be the freezing and heating modes. Does not need to be any change over or connection or the disconnection of the tube. And here we can see the system in this totality.

So, this is a temperature controller, this is a liquid nitrogen pump. And this liquid nitrogen pump pumps slope liquid nitrogen from this dewar at any controlled rate as we desire. Depending on the rate at which we would like to decrease the temperature or freezing or during.

(Refer Slide Time: 07:49)



And the system here, it actually the heating is done by applying the by the co temperature controller by setting the limits of heating and also the heating rate. And this is a silver heating block, on which the sample holder as you can see is roughly about even less than about 2 centimeter in diameter. And having a central aperture for central hole for the light to pass through when it is mounted on a transmitter light microscope.

And this system also needs to be in it is the configuration in which the model in which it is shown here. It also needs to be the device for the anti-frosting; that means, the avoid avoidance are condensation a moisture on the glass windows are on the objective which will abstract the optical path for which the nitrogen gas is allowed to flow in the form of a jet so, that it dries of the moisture.

And, the temperature when it goes above 300 degree Celsius. It can be there would be water circulation system which can be designed or even sometimes there are better available device from the manufacturer which will do the water circulation. And so here we can make a gas that the size of the vapor on which we could do the microthermometric experiment would be limited. Although, the initially when we carry out the fluid inclusions study thunder a normal microscope.

We can use a larger size vapor or the w poly thin section that we discussed. And while bringing it to the stage hot and cold stage for taking the micro thermometric run, they need to be broken down to smaller pieces smaller sizes as we will see them later on. So, there are other make, other models of this of this a heating freezing system, and these there are many other such makes which are of multipurpose uses, like for material scientists and for biological purpose.

And the stages that are used for the microthermometric experiments for fluid inclusions are the once which we just briefly discuss here. So, there are this kind of systems here the as you can see here the sample chamber here can be moved in the x and y direction from the knobs which are shown here is one and 3, this sample chair can chamber can be opened for inserting the sample chamber or taking it out.

And this is the this is connected to a platinum register which reads the temperature. And the advantage of this particular linkam stage is that the temperature at any point of time can be held by the controller at that exactly at that value whether it is depending irrespective of whether it is below 0 or above 0.

And these are kind of stages are also can be calibrated by using synthetic fluid inclusion standards or any other standard material whose melting points are known. And it works on a 3-point calibration below 0 degrees are below 0 degree Celsius at 0 degree or anything any temperature higher.

So, generally this kind of calibration are stored in the temperature in the controller and they can be periodically checked or can be re reset for to ensure that whatever data we are obtaining from this stages are correct and also reproducible. So, with this much of little bit of idea on the instrumentation there are other makes like, high make which is a French heating compression system that is also used in many of the research laboratories and.

So, with this and the basic idea about the functioning of this equipment and the specifications, and what are the features that we must this we should look for while going for such kind of stage heating freezing system is as we discussed before. Well, with this the brief introduction we will now get into the proper topic of the fluid inclusion the microthermometry, the basic principles of microthermometry.

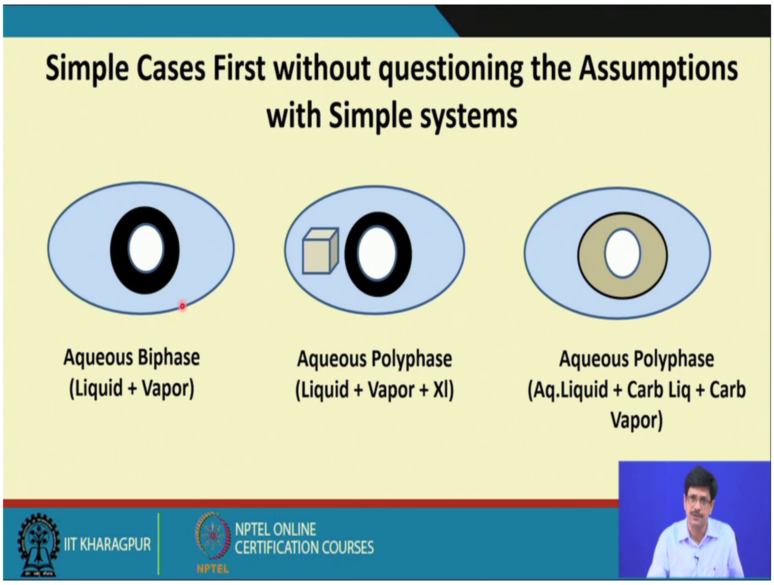
And let us first begin with the simple cases, because as we discussed there could be many questions many uncertainties many questions and many of the areas in which the fluid inclusion research is actually progressing, but for a beginner we can always start with this and going by the very simple cases and avoid. And the assumptions, the very basic assumptions that we made before getting into the topic of fluid, above the fluid inclusions and before going for any study further study of this fluid inclusions.

We have to we have to first consider this very simple assumptions. And if we recall the number one assumption is that the entrapment is homogeneous means, the time the condition during which their fluid is encapsulated in the solid lattice of the host mineral the fluid was homogeneous.

And then they fluid once trapped behaves as a close system without any subtraction or addition of material from with the surrounding; that means, with the host mineral are anything in the surrounding. And also it has followed a constant composition constant density path during it is for the revolution after the entrapment of this fluid in fluid in the host mineral.

And then the other assumption is that that we are actually that is basically the way we are defining our problem, that we are actually sampling the fluid depending on exactly the way we are addressing the problem whether it we are trying to study and over deposit are a metamorphic craft and we think that the fluid that we have we are sampling is actually representative of the geological condition in that prevailed during the formation of this main role.

(Refer Slide Time: 14:50)



Well so, we will go by the very simple cases. So, the this one is an aqueous biphase inclusion which you have already described. These are the most a common type of inclusions that we find in minerals host, minerals like aqueous and all other host minerals that we discussed calcite fluoride appetite, metamorphic minerals like garnet cordinite or even ore minerals like cassiterite wolframite sphalerite and so on.

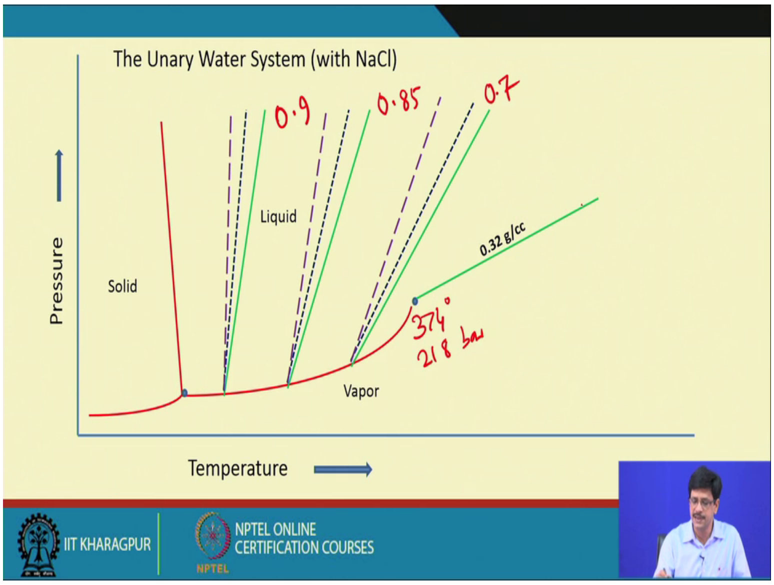
So, this is inclusion which is an aqueous biphase inclusion, where there is a vapor bubble and a aqueous liquid and it is boundary is quite distinct with respect to the solid host mineral. So, this is the liquid solid interface and this is the liquid vapor interface.

And so these are the most common type, the simplest type where we know that it could it is it is water. And this water the only come only thing which you could possibly accommodate, right now is that this water is not a pure water. It has some dissolved electrolytes in it. The other type of inclusions which also a very common as we aqueous polyphase inclusion in this sense that they have liquid plus vapor plus a solid host mineral which is inside it.

And as per our assumption this host mineral twisted the precipitated, after the after the fluid was encapsulated in the host mineral, and this crystal precipitated out of this liquid when it attend it is saturation concentration ah. So, we call them they aqueous polyphase, liquid plus vapor plus crystal, and the third most a common type of inclusions are the mixed water and gas or water and carbon dioxide inclusion, where we have a liquid reach this liquid aqueous liquid, this is a carbonic liquid and is a carbon dioxide vapor where we see that the meniscus the interface between the vapor carbon dioxide and the liquid carbon dioxide is not that very dark it is another pretty sharp and prominent.

So, these are the inclusions which are very easy to identify when we see them in the inclusion in the host mineral that we are studying. So, we will try to understand the principles of microthermometry by asking this or by trying to understand that how this possibly could have happened. Means, what are the conditions in which we can expect the fluid the crustal fluid a fluid that is anywhere in the subsurface could have been in what kind of a state so that the mineral that will go from it and will encapsulate the fluid.

(Refer Slide Time: 17:43)



So, here this is a familiar so we can we can attempt to understand the situation in which fluid inclusions could be interrupt and in what condition. So, as you see here this is the unary water system, it go it could initially we could think of it is a pure water at a on a pressure temperature diagram.

Here is this is the, this line is the with a negative slope is we know that water increases in this volume is it sorry decreases in volume is it a solidify. So, there is a negative dp by dt slope and this is the solid liquid or the melting line. And this is a solid and vapor this is the sublimation curve and this is the curve on which there is quite distance of liquid and vapor.

So, if you look at this situation, the there is a huge field in which the water is stable is a one phase liquid. So, if we imagine that a that the condition in the in the crossed anywhere in the below the surface, could be even though we have not put the exact values of the pressure. But we know that there is a vast stability field of liquid water; which is capable of existence in the liquid form and can also deposit minerals depending on when the particular mineral is attending saturation with respect in the in the in the liquid. This is the field of vapor.

So, the only condition which will give rise to a inhomogeneous or heterogeneous condition is on the locus of this curve which we call is the boiling curve where the liquid and vapor would coexist. So, let us try to first see to well. So, the factors that within this vast liquid field or also for example, in the vapor field. Anywhere any point on this curve will represent a certain percentage or certain volume percent of a liquid and vapor. And will uniquely define a value of density of that particular fluid.

And this fluid in this region where it is homogeneous liquid, and there will be points on this pretty field which will be corresponding to the densities exactly the similar density values and we can join them with line. So, this these lines actually a representing the values of a consentience; the let us say this could be a value of a say 0.9, it could be a value of say 0.9 this could be a value corresponding to possibly, let us say 0.85 or this is 0.7 just for the sake of value I am just putting here.

So, that means, this diagram the this lines which are plotted here. The did not there they represent, same value of density and that density will be also the same where the bulk density that is calculated on the basis of the ratio of the liquid plus vapor. So, as we move along this particular line, the density will be less and less. And what we observe here is that the slope of this isodensity line will also become Shelwar has not when the density value will keep on decreasing.

Now, so this from pure water, let us move onto a water which will content some dissolved sodium chloride in it. And will be moving from the pure water to a system where it is just water plus NaCl. And the reason why you are considering NaCl let me tell it at the beginning, the sodium is the most common a most abundant of the cationic species that we see in natural in crustal fluid. And chloride is also the dominate amongst the anions. And the crustal fluid the aqueous fluid which actually we encounter the most of the environments will have sodium chloride exist dominant species.

And it has been the system which is been very extensively and widely studied adjust were volumetric properties are concern. And we still I represent the crustal fluid as a fluid within the belonging to the water NaCl system. So, let us say that this dotted lines represent a value say let us say 5 weight percent NaCl in that particular liquid. So, then by virtue of the concentration of the electrolyte is density will be little higher.

So, the isochore will become stripper as an when we go from pure water which is represented by this green line to the dotted line which. And still if we dissolve more of sodium chloride unit let us say this is 10 percent sodium chloride than the density will be still be higher and the slope of the isochores will be will become stripper. So, this is a general rule.

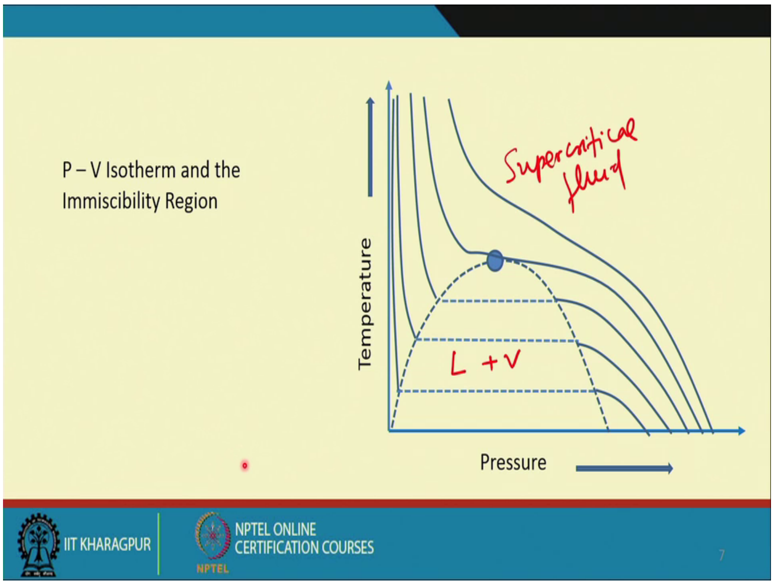
So, in the context of the fluid inclusions this understanding will play a important role, because will be considering the inclusion the inclusion fluid to have been entrapped in the one phase liquid field. And, will be having some dissolved constituent which can be expressed in terms of the weight percent. So, on weight percent NaCl, and the isochore the density line because the inclusion is going to evolve of the when the pressure and temperature changes the inclusion is going to follow a that this isodensity line, because of a basic assumption that that you have already made.

And as we all know this is the critical point of pure water at 374 degree Celsius, and 218 bars corresponding to that. So, this is 300 and this correspond to 374 degrees Celsius and 218 bars pressure. And this 0.32 grams per cc among we express density in unit of grams per cc. At this is the critical density that is a 0.32 grams per cc, and this is the this is the a critical isochore.

So, the situation that we consider that the fluid could have been entrapped by the by the growing crystal could be anywhere here. Could be a situation corresponding supercritical state or could be similarly when we have a vapor situation corresponding to the aqueous a liquid is actually that much lower pressure weight high temperature situation. There are such situations possible in the subsurface conditions.

And then still it can be entrapped by a growing crystal by some mechanism and the vapor could be entrapped. Similarly, the vapor will also have in the in the vapor field it is also possible to have such kind of the lines of isodensity lines which will be intersecting the boiling curve at different pressure temperature conditions which will see them later.

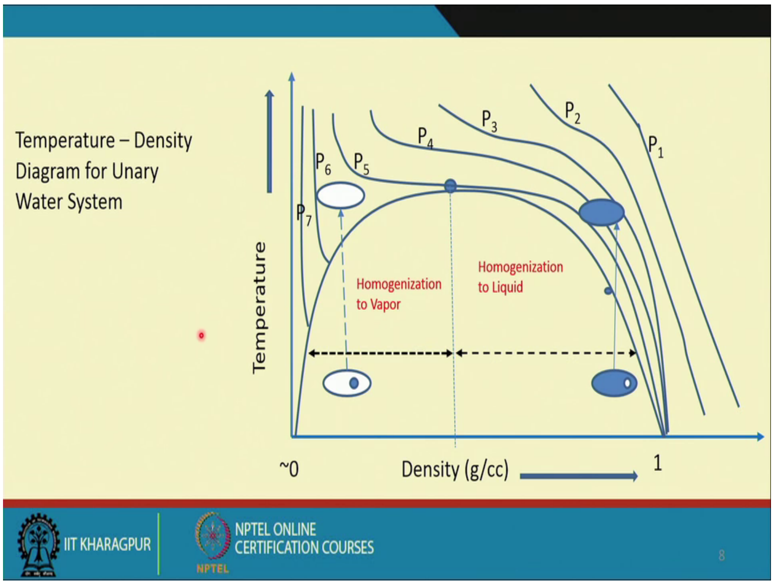
(Refer Slide Time: 26:25)



The similar diagram can be explained on the basis of the what we know is from the fundamental so, P-V isotherm. And these are the different pressure and this is the critical temperature of water. And this dotted curve here represents the situation in which there will be coexistence. There will be coexistence of liquid plus vapor, and this region will correspond to correspond to a supercritical fluid.

And as we all know that within this the pressure which is below that if we increase the pressure than the vapor will be liquefied. And it is not possible to further decrease the volume in the after we cross this boundary; where it is say it is defined by liquid plus vapor field separating from a one phase supercritical fluid. So, this is the previous diagram which was a pressure temperature translation of this kind of a P-V of the temperature pressure diagram in which we can.

(Refer Slide Time: 27:56)



The same situation could be understood in the situation which you we discuss the boiling curve. And the this is the critical point of water, and this even though this is not exactly a temperature composition diagram, but here the situation is shown that from a pure vapor which to a pure liquid or anywhere in between when it could be a mixture of liquid plus vapor as we move towards a right this will be the vapor will be smaller and proportion. And here it is a largely a vapor with a negligible proportion of liquid. And this roughly this dotted line will separate the 2 regions where the little be a, which will be corresponding to the boiling curve that we showed on a pressure temperature space in the first diagram.

And here the situation is that if you originally think that this inclusion was trapped over here. As a temperature will decrease the phase separation that we see inside the inclusion will be resulting in a dominantly at aqueous liquid plus a vapor or every inclusion is trapped here and the temperature is decreased, then will have a dominantly a liquid with a small proportion of vapor. We will be discussing a little bit detailed about the implications of such a situation. And so will continue our discussion on the principles of microthermometry with the help of such a simple phase diagrams will.

Thank you.