Fluid Inclusion in Minerals: Principles, Methodology, Practice and Application

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Lecture - 06
Microthermometry

Welcome to the second week of the lecture series on Fluid Inclusions in Minerals. In the introductory setup lectures, we got ourselves introduced to this technique of fluid inclusion.

Fluid inclusions are tiny microscopic, cavities, sealed within the solid lattice of the host mineral, there of different, there is a lot, there is wide diversity, in the way they occur in the inclusions in the minerals. And a that to be studied, they have to be understood, categorized, classified and then subjected to meticulous, micro thermometric and micro analytical studies, to retrieve important information which will be useful in synthesizing and addressing abroad and R processes like; O deformation, metamorphism, deformation, and many more.

So, to begin with we plan to in this particular week we plan to cover 2 important aspects of fluid inclusions studies. In the one is fluid inclusion petrography and we also intend to get ourselves introduced into the principles of micro thermometry, that is essential to and the remain is a background information essential background information, when we study them under the microscope or do micro thermometric experiments.

So, let us first try to understand, what is actually micro thermometry? What you understand by micro thermometry? So, micro thermometry is essentially what you mean is that, we see the inclusions under the microscope.

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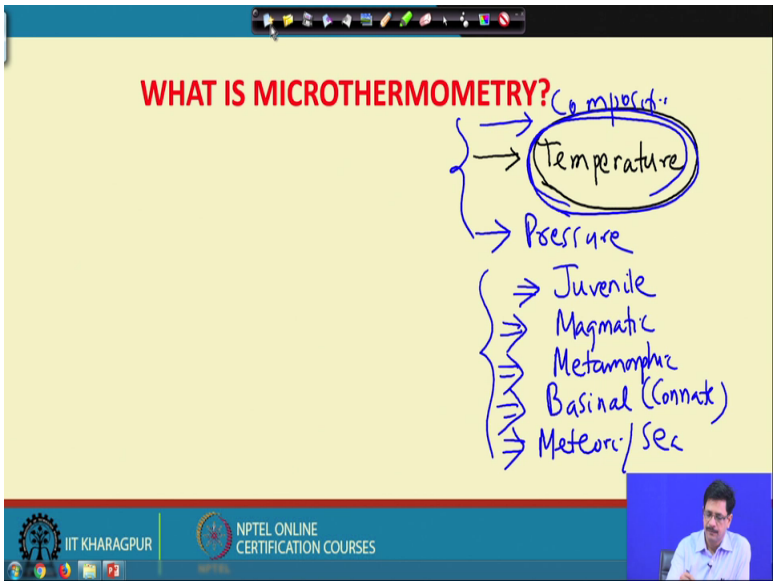

And primarily we are interested in the thermal characteristics; that means the temperature. We are interested to know what was the temperature of the fluid that give rise to this particular mineral? Because, we might ask yourself why temperature, because of the fact that the fluid that, we are interested in are essentially very diversely sourced.

This fluid could come from this fluid could actually could have come from I have much deeper part of the earth into their maybe from the mantle, which could be Juvenile fluid or could be a fluid, which have actually have been recycled into much deeper part of the mantle in through the metasomatism.

And, this type of fluids are essentially to be expected to be much hike in the temperature, the fluid could have been source from a crystallizing felsic magma, the fluid could have come from dehydration or devolatilization of sedimentary rocks with give them metamorphism. So, during the process of metamorphic devolatilization and we can call them as metamorphic fluid, they could have come from shalo circulating or kind of fluid which track with the sedimentary basins, they can be Basinal fluids or we call them as connate fluid. The fluid could be the fluid that is precipitated on the surface or sea water which flocculates to the deeper vision of the earth crust get heated up exchanges component with the rock.

So, this could be a fluid which could be meteoric or a sea water. So, all this fluids there the there this is a totality this the kind of fluids that very interested in, and each of these

fluids are very much characterized with a temperature and the compositional ranges. And so, we essentially try to retrieve this particular temperature and also the other parameter which is pressure which will be discussing later. So, temperature pressure and composition so, these are the 3 attributes that we are interest to work out of this week.

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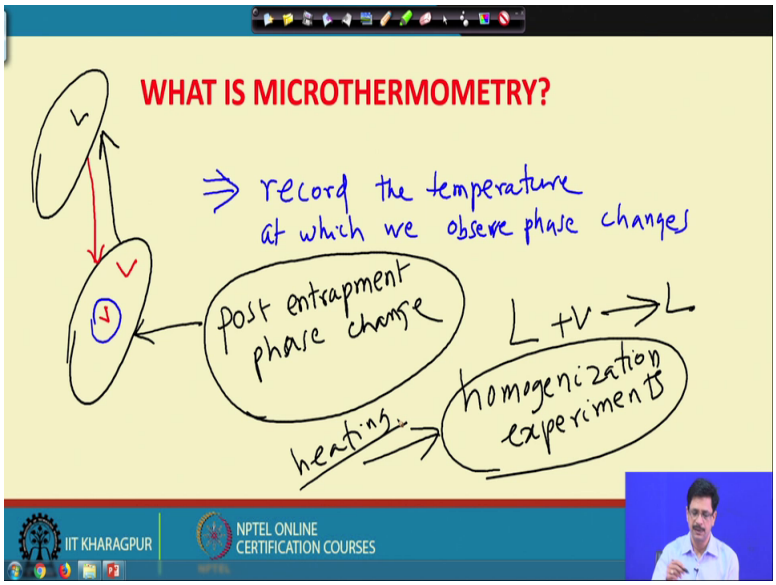

And so, if we first take temperature so, how, how we go about it? So, the micro thermometry means is the essentially it the process in which, we experiment on this fluid inclusions and record the behavior or record the temperatures at which we observe phase changes. Remember that we started with this the with so, before we do this with the what we discuss about the assumptions, the remainder the remain the basis for the micro thermometer study.

So, we go by the first assumption that the fluid was trapped homogeneous. So, if the fluid was trapped is a homogenous fluid, then when we see an inclusion as a this as a liquid and this as vapor. This inclusion definitely was where it was trapped; it was trapped as a homogenous fluid let us say it was a trapped is a homogenous liquid.

So, this homogenous liquid after it was trapped, when it underwent change in the pressure and temperature, there is something which happened, which we say that actually it underwent a post entrapment phase change. And, in this particular case a homogenous liquid got splitted up into a combination of liquid plus vapor. So, this our basic as on based on our the first assumption, that homogeneous entrapment; that means, when we

see them at room temperature is a combination of liquid plus vapor, we presume that this has this took place after the fluid was enrapt in the cavity they can have to sealed. And, this happened as a post entrapment phase change.

So, now if you recall all these combinations of the fluid that we show, that we saw through our sketches a liquid plus vapor, a liquid plus another liquid first one liquid 1 plus liquid 2 plus vapor or liquid plus solid or liquid plus more than one solid. All the situation there we observe at room temperature or basic assumption is that they all are a result of post entrapment phase change.

So, if this a process is actually the post entrapment phase change then it is quite natural that would like to go back or to go back to the original homogenous position, homogenous condition, the question is whether we are able to do that. So, if this particular thing is a result of post entrapment phase change, then our objective is to reach back to this homogenous state.

And, note down the temperature at which this inclusion the changed from a combination of liquid plus vapor to a liquid so L plus V to be coming L.

So, this kind of so, this is actually it would be worth file to introduce the term here. So, we generally call that this a homogenization and what we do here is actually homogenization experiments. And, essentially please note that we are interested in the temperature, means high temperature means we are only talking about the high temperature situation. Because, this the entrapment definitely took place at temperatures well about the temperature at which we see them, and beginning from anything between as we fix the range of the fluid from anything between 50 degree Celsius to 600 700 degree Celsius a temperature.

So, in respect to that what we say that we what we are conducting here we are contacting the homogenization experiments. So, we intend to bring back this particular inclusion into it is homogeneous state at which it was supposed to have been enrapt. The only difference here is that this particular entrapment process took place at a pressure, which is definitely higher than the pressure it which we doing experiments.

So, we are doing experiment at a room atmosphere one atmosphere pressure. Whereas this particular homogenous entrapment could have taken place anywhere in the earth

crust ranging from pressure as low as a 100 200 bars to going from 3 4 5 kilo bars and more than that, when you go to process such as the departmental when the diamond is forming it could be pressure well above 30 40 kilo bars and be more than that.

So, that will discuss later. So, here so, the only difference is that we are conducting to get experiment at room temperature pressure room pressure condition that is atmospheric pressure, where is this particular inclusion must have been enrapt with the higher pressure.

So, if we keep the pressure thing for the time being we do not consider pressure, then if we only consider temperature, then this micro thermometry is essentially intended towards obtainment or obtaining of a homogenous state of any fluid inclusion that we are observing under the microscope, irrespective of what is the phase combination, whether it is a liquid plus vapor or there 2 liquids or 2 liquid plus vapor liquid plus solid plus vapor. Whatever maybe the situation, we would have to bring them to a state of homogenous condition and that is we are able to achieve by heating it by heating and.

So, we will see what exactly we do for that. So, it micro thermometry is essentially that file. So, at this so, the thing is not when we are conducting our heating experiment, it is very much when it is mounted on to the microscope we are observing, the fluid inclusion while we are increasing the temperature. And, then able to note down the temperature precisely with uncertainty of hardly plus minus 1 degree Celsius, that at what temperature is fluid become homogenous, which will we discussing now.

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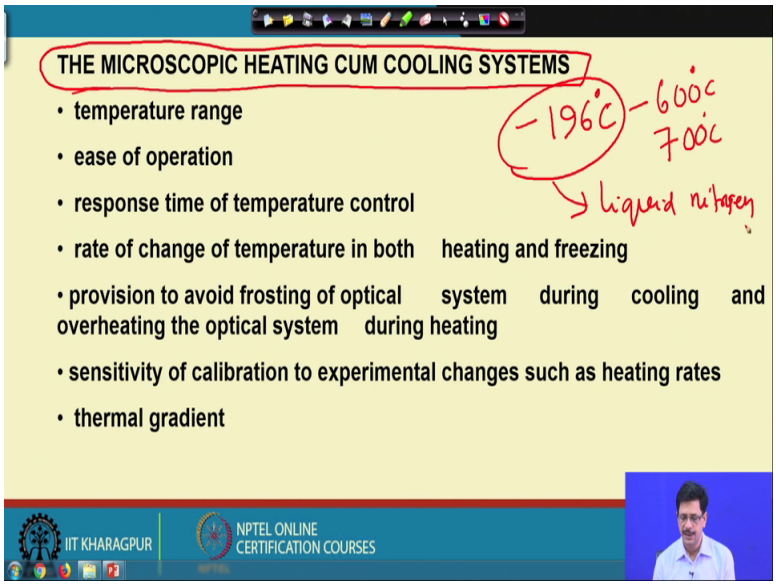

So, for the for contacting heating freezing experiments, we use there are devices which we call as the microscopic heating cum cooling or heating cum freezing systems.

So, these are the microscopic heating come freezing systems and there are many such commercially available equipment, where the heating freezing experiments are conducted, in the temperature range of minus 196 degrees Celsius to 600 or sometimes there are different systems in which the temperature could go to 600 or go to 700 depending on the design of the equipment. And, we are limited by this lower temperature is minus 100 and 96 degree Celsius, because it corresponds to the temperature of liquid nitrogen the boiling temperature liquid nitrogen at to atmosphere.

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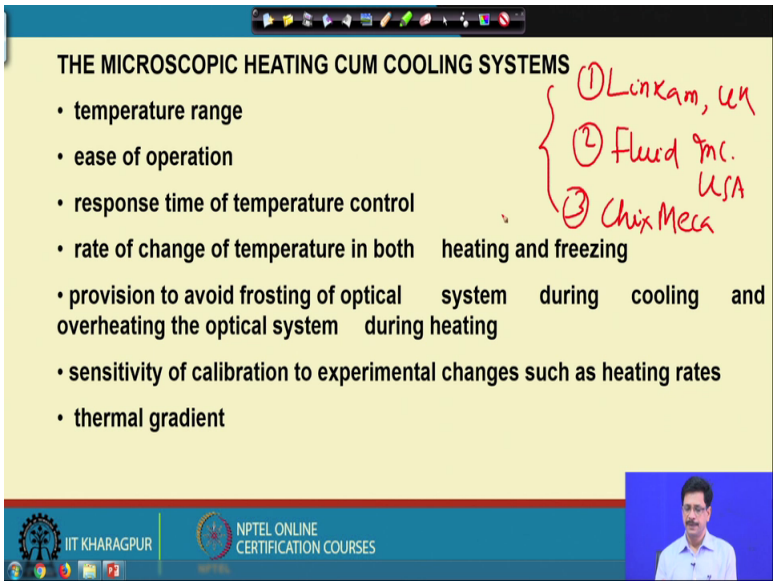

And, there are heating freezing systems which are available like the one which is available from Linkam U K, there is the stage which is available from fluid incorporated in a company in USA, Colorado in USA. And there is to be Chix Meca French. And these days there are many other devices which are you manufactured by many companies with multiple applications to material sciences as well as geology.

But, our primary requirement is that the we should be able to the sample the doubly polished sample or the vapor that we have prepared, we should be able to put it inside the system which is our microscopic heating cum cooling system. And, should be we should be able to observe them through the transmitted light or in case of a opaque mineral through then through an ith light source. And, would be able to observe I mean life so that we could record any of the temperature any of the phase changes taking place at any different temperature.

So, we will see some of the features of them, but it would be for rather more appropriate to talk about the attributes of this kind of heating freezing systems, in terms of their functionality in there usability. So, the temperature range we have already have considered. So, that is minus 1962 600 or 700 degree Celsius.

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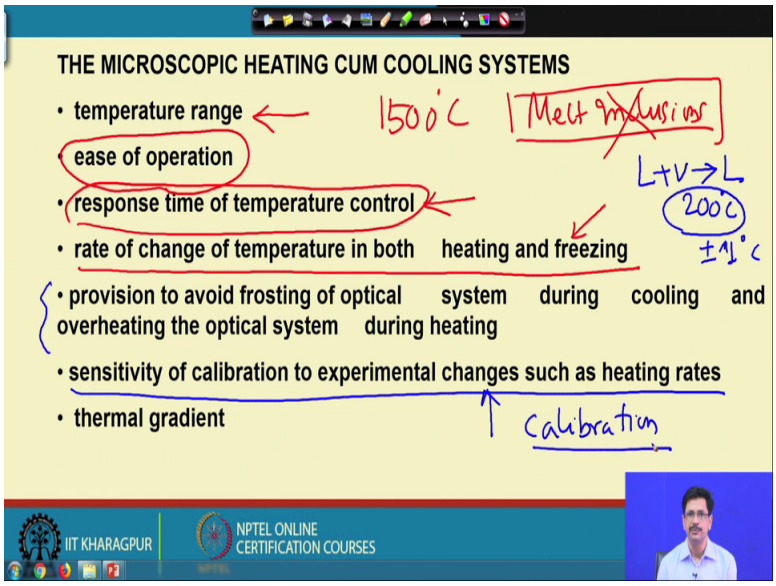

Even there are hot stages, which goes up to 1500 degree centigrade there, which has specifically designed to study melt inclusions, which we are not considering in this particular course, but there are hot there are hot stages where the temperature can go to as high as 1500 degree centigrade. These of operation is definitely is one which is essential for us, like should be able to easily put the sample inside the a device, and will be able to increase or decrease the temperature at our wish, the way it will be convenient for us to do the experiment and the response time of the temperature control.

So, all these whenever it comes to heating, the heating has to be done definitely there is electric current that is needed to heat a particular object. They must be some element through a through which the electric current to be passed and it will get heated up. And this thing has to be controlled by specific control unit. And a here as the as non-specialist on this kind of device, we can only talk about it that the response time of the temperature control must be very good. Means, if we want to increase the temperature in we just increase the voltage. So, the temperature to which the desired temperature to which we want to, wanted to increase must happen instantaneously.

So, it depends on a combination of the material, which is actually used for making such devices and the control unit. And so, this response time of the temperature control and the rate of the rate of change temperature in both heating and freezing.

For example, if you want to observe the phase changes very carefully and it is much more important for us, that the observations to be reproducible. Giving it suppose for example, one particular inclusion that we have observing is as a liquid plus vapor say for example, only one inclusion which was liquid plus vapor has become a liquid by homogenization and this takes place in at 200 degree Celsius, that we observe. Now, if we if your rate the rate at which we are heating the sample is too fast, then reproducing the value at 200 degree centigrade be very difficult.

So, we need a very optimum or heating and the freezing rate. So, that we should be able to reproduce this particular value to plus minus 1 degree centigrade we to be acceptable. And, the provision to avoid frosting of the optical system due to cooling and overheating of the optical system during, heating; so, this things are also very nicely taken care of by the device which are available these days. When, we are working in a laboratory where the temperature is room temperature of the order of 20 between 24 degree Celsius are so, and there we are going to even though in a very small chamber of the device, where the temperature is going down to the level of minus 100 or minus 120 of minus 196.

There it is very likely that the moisture of the air will always convents on the device and will be able to that it will affect the visibility. And, the there should be a the system must be operating in such a way that the in the lower temperature ranges of operation, the visibility also should be maintained because those are the time those are the temperature ranges where proper observation the clarity of the observation is very important.

The sensitivity of calibration to experimental changes such a heating and heating rates wherever we do any as we all know that whenever we are doing an experiment and we take the reading. So, the dependability of the accuracy of the temperature values that we are getting from the device is very much dependent or it can only be obtained, if we have a good calibration.

So, such heating freezing systems which are used in all our laboratories, they need to be calibrated and for that calibration we need to have some non-standards.

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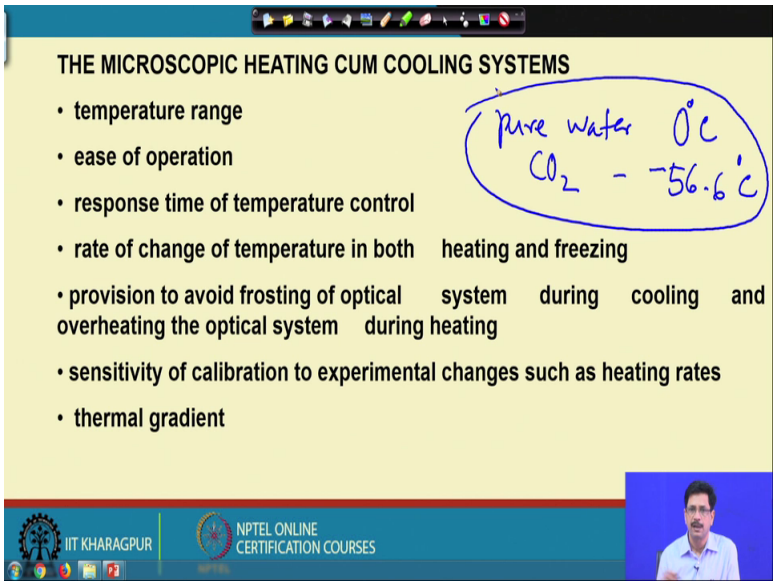

For example, we know that water if it take pure water, the ice and water equilibrium will be corresponding to a 0 degree Celsius.

If, we take up all the will be discussing a little later, if it take carbon dioxide. We know that carbon dioxide triple point, where the solid liquid and vapor coexist is minus 56.6 degree Celsius. And there could be many other so, these days when with very good quality synthetic fluid inclusions been prepared in many world class laboratories. And, there the fluids it is of non-composition are being trapped a secondary inclusions in this minerals, synthetic fluid inclusions, those we though the very well are used in all laboratories and standards, where such kind of temperature values could be reproduced.

Sometimes the higher temperature supposed in any particular synthetic inclusion the temperature of trapping is more in temperature homogenous is known. Those can be used as standard for our heating freezing runs when we take in our own mission.

So, thermal gradient is of course, one situation where we are even though irrespective of the small size of the sample the small area in which we are conducting this heating of freezing heating freezing experiments. They could still be minor thermal gradient; that means, when we are we are suppose for example, we take vapor and this is our vapor and within that vapor there is one inclusion which is here.

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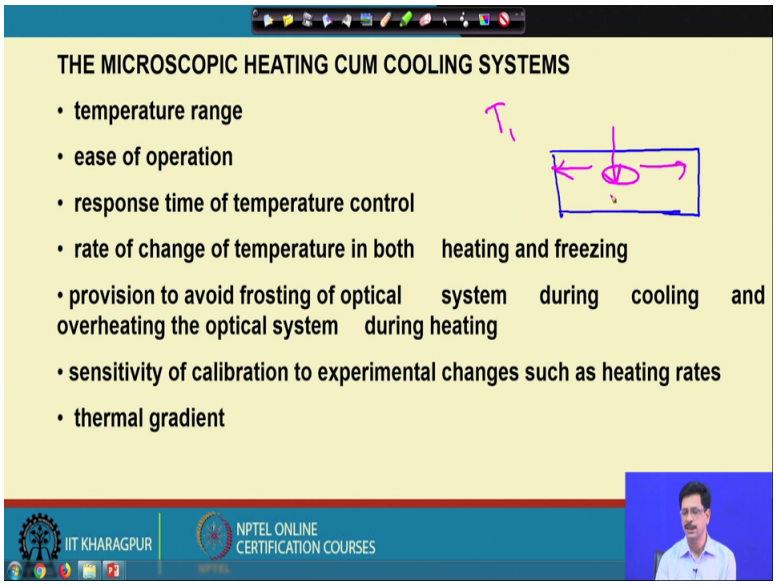

And this particular inclusion, we are doing the homogeneous experiment. So, we are we are definitely. So, this particular this is the vapor which is kept inside the instrument. And instruments temperature is being sensed by some kind of thermocouple mechanism. So, some kind of like a paternal number register or some kind of chromel alumel thermocouple and is depending on the voltage and current it is calibrated the temperature, and it is indicating a temperature let us say any value let us say temperature T 1.

Now, the thing is that whether in this entire sample the temperature could be T 1 and we are more interested in exactly knowing the temperature, it is exactly is what here. So, it may be just about few a millimeters have a that this side and that side or so, here do within that there should be there should not be any thermal gradient. So, that the temperature that I have that is recorded here and is being sensed as a whole for this particular area by the device that should not be any discrepancy.

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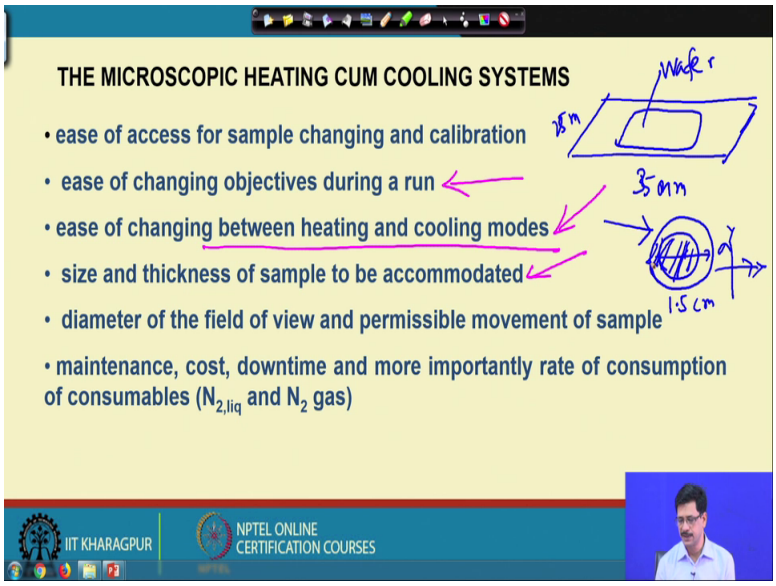

And, the ease of access for sample changing and calibration, means the whenever we are changing the sample, the sample holder this thing should be very well designed. So, that there be a good ease of operation. The ease of changing of the objectives during a run this is not that very important, but sometimes it is required that, during our heating phasing experiments we or relocating the inclusion, the one the inclusion that we took freezing run some time and we want to get it back and take a heating run. We may have to change the objective to a smaller magnification and then so, this kind of change of objective while, the microscope stages actually fitted with the hot and cold system.

So, this also is an important criteria for the hot and cold system that will be using and, the ease of changing between the heating and cooling modes. For example, even though we have not considered the low temperature part we are talking about temperature only, but as we know that we need to do a freezing experiment on these inclusions also for knowing there composition. And, then we want to change the mode from the inclusion that we froze, we want immediately want to take a homogenization experiment of homogenization run on the particular inclusion.

This kind of switching of between the heating and the freezing mode should be very easy for us. And, the size and the thickness of the sample to be accommodated is another criteria, that that should come in evaluating how good or how bad or how effective or how good and device could be for our purpose. And, the diameter of the fluid of you the

permissible movement of the sample, because generally it would be good to have a bit of a better an idea that what kind of dimension that we could be using.

When, we take a sample and we prepare a section the sample, let us say it could be something like a 35 mm to 25 mm; that means, 3.5 centimeter to 2.5 centimeter. And, on which we have mounted a section of our sample that we that we wanted to stay do the fluid inclusion study. And, this will be of the order of say for example, 2 centimeter by 3 centimeter or to 1.5 centimeter by 3 centimeter and of a size and this is the this is a doubly polished thin section or the vapor, that was prepared to study the fluid inclusion.

So, this vapor which is mounted on a slide by using some adhesive like a Canada Balsam has is basically be taken out and will be put into the hot and cold stage.

But, the stage the sample holder or the area could possibly be just about a one 1 centimeter or at the most 1 5 1.5 centimeter diameter circular sample holder on which the sample could be put. And, then even if this sample is put inside the sample holder like this is possible suppose this is the sample, which is put inside the sample holder and has to be again smaller and it is dimension.

So; that means, the vapor which was prepared of a particular dimension has to be broken into smaller pieces or chips and will be loaded onto the sample holder of the device. And, now once it is loaded into the sample holder the device also should allow a sufficient amount of the movement in the in the X and Y direction.

So, that as much of area of this particular vapor should be visible to us. So, I will come back to the discussion of the on this particular aspects that is to what should be the size minimum size or the maximum size of this vapor, which will be loaded into the sample holder of hot and cold system. This also is very important, where we plan our micro thermometric experiments, which will be coming back to this particular point again later.

And, then the situation is that this devices they work on liquid nitrogen or till so, far we have been able to use liquid nitrogen, we have not been able to use any other gas which would give us lower temperature than that, so, that is how we are limited to a temperature ranger minus 196 degree Celsius.

So, we need to have constant supply of this liquid nitrogen. And other kind of maintenance of the equipment that it is necessary for keep it in a running condition are these are for example, the consumption of the consumable part, we will might just look a little bit details into one particular type of hot and cold stage, and what kind of consumables are the exteriors peripherals that we use there or you may get it from there from the from standard literature as well. And, then the peripherals like the quality of temperature control at any temperature, which actually we were.

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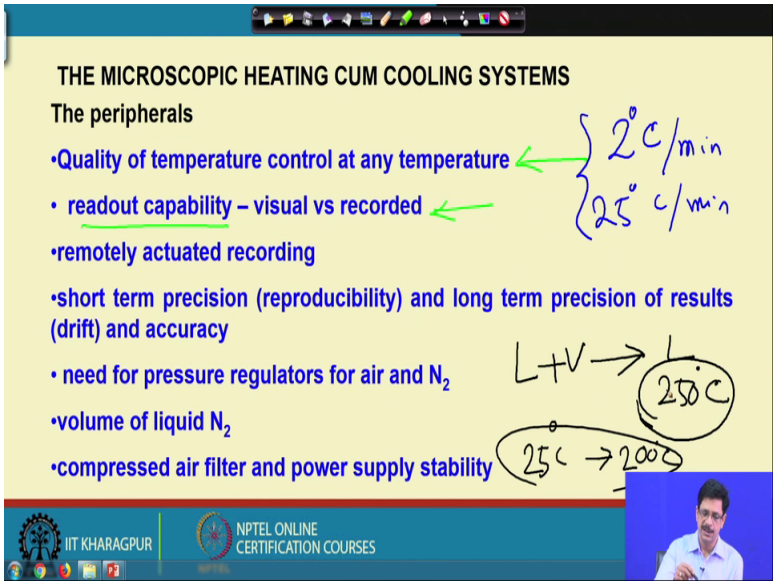

So; that means, the design of the a control devices such that the quality of the temperature control should be to the best possible for us. And, the readout capability verses visual record in today's the today's situation most of the equipment are computer controlled. And, the microscope is attached with CCTV and the images are very well displayed on computer monitors with good graphics card. And, we can get a very good image very good very good visual effect of this particular inclusions, which we were studying and taking micro thermometric measurements while observing I am under the microscope.

So, visual recording is almost like there was a time when the make the phase changes used to be recorded through the I P s and then the temperature at which is to be recorded as to manually recorded. Now, this particular situation is improved a great deal. And readout capabilities like that, the display device of that particular heating freezing

system. That means a temperature at which a particular phase change is occurred has to be recorded effectively and sometimes also can be put into the memory and so, that the data can be utilized.

So, this is also one of the things which is important and there has been significant improvements on this particular area were the last one decade, where there has now very remotely operated or touchpad type systems, where the hot and cold stage could be operated for decreasing or increasing the temperature, the rate of the rate at which the temperature to change, sometimes we can even go up to as low as this kind of this kind of so, hot and cold stages. They operate in a situation where the temperature change rate of change could be as low as about 2 degrees centigrade per minute to even as high as 25 degrees centigrade per minute.

And, if we are taking the it is doing the micro thermometric experiment on any inclusions. And would like to go to temperature and then this particular gradient or this temperature the heating and cooling rate also could be very variable. For example, i if a particular inclusion, which was a liquid plus vapor at room temperature. And a homogenization experiment is taking place is being conducted and suppose this particular liquid plus this homogenous, this inclusion homogenous to liquid and a temperature 250degrees Celsius.

So, we were interested in a precise recording of 250 degree Celsius not actually much concerned about, how many times we can change the heating rate? To go to do a to make a compromise between the time taken to have such micro thermometer experiment done on and because it can be very well visualized, that we have to do micro thermometric experiments in each and every individual inclusion and in 100s in numbers. Sometimes it actually depends on what is the dimension or the what is the area on which the work is taken up and what exact problem we are actually will addressing.

So, their number of inclusions that is required to be studied for micro thermometry or micro analysis is quite variable. So, when I have to measure the temperature record the temperature in 250 degree Celsius, going from room temperature of 25 degree Celsius to almost like 200 and 200 degree Celsius, this could be done at a much faster rate even going up to 20 degree centigrade per minute or been more than that, but as a as the temperature of homogenous in would be approaching for a proper reproducibility and

proper the recording of the temperature without introducing any error. There the rate of heating should be as low as possible. In the heating cycle the low cool with the rate could be a little higher, but in the freezing cycle the could be the minimum possible, which will be discussing.

So, there should be short this remotely actuated recording the short term precision the reproducibility and the long term precision of the result that is the drift. So, this as we discussed is coming from the periodic calibration of the equipment, where there is. So, that there is no long term drift of the equipment if the so, the any hot and any hot heating freezing system has to be periodically calibrated.

For example, if I calibrated today I may need to calibrate it just about a month later even a week later depending on how the equipment is actually behaving. And sometimes many of the situation that we observe that the it does not actually shift much, but the it is a routine procedure to check or to re calibrate the instrument with the standards and so, that the did you could be taken care of. And the pressure the since, it operates on liquid nitrogen, which is volatile and it is actually gas at the room temperature.

So, you need to have a proper circulation system. So, that is in built into the system how the liquid nitrogen will be circulated in the systems like the Lincoln stage. The liquid nitrogen circulation is done by a liquid nitrogen pump, which can pump the liquidated any controlled rate. Manually or could automatically set and the another important aspect of the hot and cool system is the volume of liquid nitrogen that would be required, in systems like the fluid incorporated adopted the fluid incorporated hot and cold the system, heating cum freezing system, there the volume of liquid nitrogen required is more and in case of Lincoln stage the volume of liquid nitrogen required is less. Because, in a fluid incorporated hot and cold microscopic heating freezing system, there is no liquid nitrogen pump it actually just is circulated by using dry nitrogen gas.

So, that it carries the cold nitrogen gas to the sample and can an cooled cool set. And, sometimes the is in case of the fluid incorporated adopted stage hot and cold stage. The heating is done through heating coil and bypassing compressed air to the heating coil, but this problem this the system the heating system in Lincoln stage does not require any compressed air.

So, these are some of the consideration. So, some of the characteristics, attributes of the microscopic heating cum freezing systems, that are utilized for conducting micro thermometric experiments on fluid inclusions. So, we conclude our discussion today. And, we will continue with the principles of micro thermometry in relation to the entrapment of various types of fluid inclusions with correspondence to their phase behavior at in high temperature as well as the low temperature regions. So, we will continue the next class.

Thank you.