## Rheology and Processing of Paints, Plastic and Elastomer based Composites Prof. Santanu Chattopadhyay Rubber Technology Centre Indian Institute of Technology Kharagpur

## Lecture 21 Practical demonstration on RPA

Welcome back to NPTEL online certification courses on rheology and processing of paints, plastics and elastomer based composites. Today we will be covering on RPA, there is a rubber process analyser. So as you have seen in case of ODR and MDR, we can analyse a rubber using a fixed temperature, a fixed RPM or a arc and also at a fixed time. So if we want to have a dynamic flavour to that experiment, we cannot go for the MDR and ODR. So that is why scientists have made the MDR evolved into a RPA. So the basic structure of the RPA is similar to that of MDR, but the difference is in its dynamic application.

That is we can change the temperature, we can change the shear rate, we can change the frequency and all those things. So detailed description about the machine and the software will be demonstrated by Mr. Rajesh De. Thank you Mr.

Debabrata Ganguly for your overview and demonstration of RPA. Now I Rajesh De will show you how to operate the instrument. Now in our facility we have Montech D RPA 3000 instrument. This is having a look of the instrument. The instrument needs single phase supply 230 volt AC and it has the pneumatic system which connects two biconical die to reduce the temperature of upper and lower plate heads.

In normal rheometer, the cooling capacity depends upon natural system, but in pneumatic system it is installed in this rheometer for faster cooling by compress air flow. We have connected two pneumatic system to the compressor and it required 5.5 to 6 bar air pressure. Whenever you start the machine you have to check the pneumatic supply is coming out or not. Otherwise the upper plate end will not go down.

The machine required two safety checks, one is electric supply and another is pneumatic supply. This machine has two pneumatic lines which is controlled by software, one is for main cylinder and other for cooling system. There is a motor inside which oscillates you can say a servo motor which can oscillates there is a torque transducer on the upper plate end. If we put the sample here, the upper plate end contains torque transducer which is connected through this line and this is these two pneumatic pressure line. This is temperature for upper plate end and this is for lower end. plate

We cannot change the temperature from here, we can change only temperature by software. There is three force switch, one is start, one stop, standby and errors. Also this machine is complied with this volumetric sample cutter. This volumetric sample cutter cuts the sample accordingly with this cavity volume. If we put the sample here applied a particular force 5.

5 to 6 bar, it creates 10 to 11 kilo Newton of force on the sample. So it is a basic for this kind of machine, it oscillates as a particular frequency and particular angle and give the data. The instrument main switch is in the back of the instrument. First we switch on the main instrument, then switch on the PC that is been software part. If we switch on the PC, automatically MonControl software will be opened.

Now I am showing the how to control this instrument. This is the MonControl software. This is the test, if you click the MonControl software, this type of display will come. Once we switch on the computer, the software will automatically start. We have wait for a couple of second, we can see the temperature from here, this is the temperature.

This is the test time, this is angle, this is temperature and this is frequency. There are some icons is here, if I click on this icon, the communication active. This is green means its communication is active right now. This connects the software to the instrument. This is temperature stabilizer.

Now we set by default 177. Now it is already coming 177 that is why this is temperature stabilizer, stable temperature is now stable. This is for sealed up and down. Right now the sealed of the instrument is up that is why it is not green and this is cylinder up and down. Cylinder also this time is that is up is why it not green.

This is the motor, motor is on or off, motor is right now on that is why it is green and this is test off or on. So this rheometer here some three icons, there is a device called additional function over here. If you click on it switch instrument type, if you click on it please choose the option. Here we can choose the option, it is now rheometer mode to change the mode click dynamic mode, it already changed to dynamic mode. We cannot make the Mooney testing in this machine because Mooney have separate rotor which already demonstrate in other videos.

This software is common for all the three instrument and that is why it is given by default switch to Mooney mode, but actually this is not valid in this instrument. If I change to rheometer mode a green color will change here, now it is rheometer mode. This is the window over here, test time, angle, temperature will show here. What we have set the time show here, angle should be here, temperatures will show here and frequency will be hereafter reading is coming. So this is the temperature graph, this is the temperature graph andthisisthemaingraph.

The scale of the graph is auto fixing, as the temperature increases the range of the graph changes. There are bottoms here start, stop, standby, error. Now green color start button is blinking means we have create a program, but the machine is not ready for test. This left side window is pull a list, whatever program we will create it comes to be tested. To create the program we have to click the test configuration.

First then we go to create type, compound name, additional information and recipes after status active we can create the program. In this static mode of rheometer angle and frequency always constant, in the static mode angle always 0.5 degree as like MDR and frequency always 1.67 hertz. So now we have to select the data point that is results are asking for these compounds.

So we required S prime minimum, S prime maximum, scorch time, TS1, TS2, TC90, cure rate index, ETC. So again I change the it to dynamic mode. If you in dynamic mode this type of display on the screen for initialization, temperature for initial start and stabilized time slab range, ETC we have to provide. Isothermal test for normal cure test. Here time, temperature, frequency, angle have to put.

Frequency sweep, in frequency sweep how many cycles are required we have to set here and frequency from beginning to end we have to put here and initial temperature angle also we put here. Temperature sweep it is a very important sweep of this RPA. This is here temperature time is needed and this is relaxation. Relaxation after certain strain applied how much relaxation will coming from this rubber it will be measured from here. Hold time in if we select multiple test in a same program then each after finishing each of the program hold time it is required.

Non isothermal test is also frequency angle program type temperature it has to be filled. And this is amplitude sweep that is when strain sweep normal strain sweep how many cycles are required and what is the angle is to be provided. Multi sweep, multi sweep we can select isothermal frequency amplitude can be set simultaneously in a same program hysteresis test and retardation. This is the for dynamic mode of testing. This is the volumetric sample cutter.

This is the cutting chamber and this is two switch left and right. If we put the sample like that and press the bottom simultaneously it cuts. So, it is 5.5 grams sample required for each test.

This cavity volume is 5 cc. So it required 5.5 gram. Here you can see two pneumatic line is attached with this instrument. One is for main piston and another is for cooling the upper and lower platens.

And all pressure is always 5.5 to 6 bar. You can see here the rotor is oscillating at 3 degree angle. We can change the rotor oscillation. Now it is 3 degree angle. After cutting the sample from volumetric sample cutter the sample is cover this type of plastics and put inside the rotor at center portion.

Then we can start the test. Its shield is go down and the platen also is coming down. Now the test is started. After close the platens this type of gap is coming that is when test is started already. This is the temperature it set 177 and this is the torque is coming S prime.

It will take minimum 30 minutes. So we will present and discuss with you already completed analysis data because each test required some time to finish. So you can see here isothermal cure for static mode of rheometer. It is the isothermal cure experiment are the most common quality control test of rubber and elastomeric processing with over 3500 data point available on this one control software. All characteristics including minimum, maximum, elastic torque, scorch time, TC90 cure time, reaction rate, preciously calculated, pass or failed status and tolerance gate can be easily set and evaluated with each test. You can see here the green color is curve for S double prime and S prime is this red color is the main cure characteristic torque.

Here TC, TS2 is scotch safety and TC90 is optimum cure temperature, optimum cure time. Similarly this is for non-isothermal cure. Montake MDR and RPA can be programmed to follow any non-isothermal temperature profile to simulate mixing, milling, extrusion and compression molding, injection molding and storage condition. Final test sequencer executed in a single test can be included with other dynamic test for the most accurate data discerning material behavior. You can see the temperature now approximately 40 degrees.

It starts from 40 degree and it is final temperature 160. If I set some temperature rates, it is say it is 5 degree or 10 degree per minute, 10 degree per minute, then the temperature is generally rising and cure curve also be computed like that. This is the non-isothermal cure test. This test is possible only this RPA because MDR, ODR does not facilitate this type of test. This is cure with simultaneous sponging, foaming, blowing reaction.

Actually taking place during this cure process, foaming reaction produce cellular membrane like structure within mixed and are vital part of the compound development.

This cellular matrix created during the foaming reaction reduce density, increases thermal and acoustic insulation and affects the thickness of the matrix. Montake cryometer are optionally equipped with the precision normal phase transducer in the die cavity. This advanced transducer reveals interrelation between the simultaneous cure and foaming action. This green color is pressure and this is the S prime deci Newton meter.

And here the graph is showing is advanced cure kinetics modeling. The test data from similar statistic or dynamic test sequence executed as a different temperature are evaluated and modeled for an advanced cure kinetics analysis. Information acquired including reaction rate, order of reaction, rate constant, activation energy and incubation time. Here you can see 140 to 180 degree centigrade varied a sample. So in general 140 degree centigrade the cure rate is very low, this type of, scorch time is more and in 180 the cure rate is very low, this type of, scorch time is more and in 180 the cure rate is very low.

So this is advanced cure kinetics modeling. Frequency sweep material analysis. Isothermal frequency sweep provides details analysis of the molecular weight distribution, crossover modulus and average molecular weight crossover frequency for any elastomeric compound. Based on the frequency and given temperature during a test, mechanical properties can be easily predicted. This instrument has incorporated additional advanced testing capabilities such as time temperature suspension principle TTS. This type of rheometer for WLF can be used to master modeling. cut

So predict material performance at temperature and frequencies outside from normal range. This graph is for structural characteristics and processability. Structural characteristics of elastomeric compound influence material behavior during processing and final product performance. In order to simulate various process method or evaluate material states, tests are performed in the linear or non-linear viscoelastic range. This dynamic rheometer conduct frequency sweep over a large shear range to reveal substation material characteristics pertaining directly to processability.

Here NR 1, NR 2, NR 3, three different type of NR from three different countries. Structural characteristics and possibility can be displayed in this graph. Here NR 1 and NR 3, the moniviscosity is almost equal. That is why these two points almost same position.

Non-linear material response at high strain. Dynamic oscillatory shear rates commonly known as small amplitude and large amplitude. Oscillatory shear rates are effectively method for measuring viscoelastic properties of rubber compound for polymers and integral part of the discerning material response in processing operation. This rheometer can be equipped with high speed data acquisition system. This enables Fourier transformation analysis of periodic data including full raw data access for research into viscoelastic behavior. By using Laplace testing, the materials trace response is easily quantified enabling a fully outstanding of filler content structure and polymeric architecture.

Linear branching can be detected from here. And this is isothermal curing at various strains. If we change the strain, that is been a degree of oscillating, this type of graph will show the results. The rheometer provides precise test results at variable oscillating range angles for ideal strain amplitude optimal single to noise ratio while avoiding any structural breakdown or slippage of the sample in the die cavity. The variable oscillation angle can be set according to these needs of the compound. For example, a higher oscillation angle may better distinguish different between branches and soft materials.

This is for structural breakdown of rubber compound process simulation. Processing simulation is a powerful tool that can be used to shorten R and D times and aid with mixing quality control. This rheometer provides simultaneous capabilities for simulation providing data for developing rubber compounds by simultaneously manufacturing process and environments. And this is a very interesting test for this RTA strain sweep or filler loading.

It can say pain effect filler filler interaction rubber. The pain effect testing measured the stress strain behavior of test material. Physically the pain effect can be attributed to deformation induced changes in material microstructure. That means to breakage and recovery of weak physical bond linking adjacent filler cluster. By discerning the relationship between modulus and strain, it is low strain or high strain areas. Users can quantify filler loading dispersion and filler filler interaction.

The resulting materials characterization directly impact dynamic stiffness, damping behavior and final product performance.