Dealing with Materials Data: Collection, Analysis and Interpretation

Professor M P Gururajan, Professor Hina A Gokhale
Department of Metallurgical Engineering and Materials Science
Indian Institute of Technology, Bombay

Lecture No. 96

Case study 3: Calibration

Welcome to Dealing with Materials Data, we are looking at the Collection Analysis and Interpretation of Data from Material Science and Engineering.

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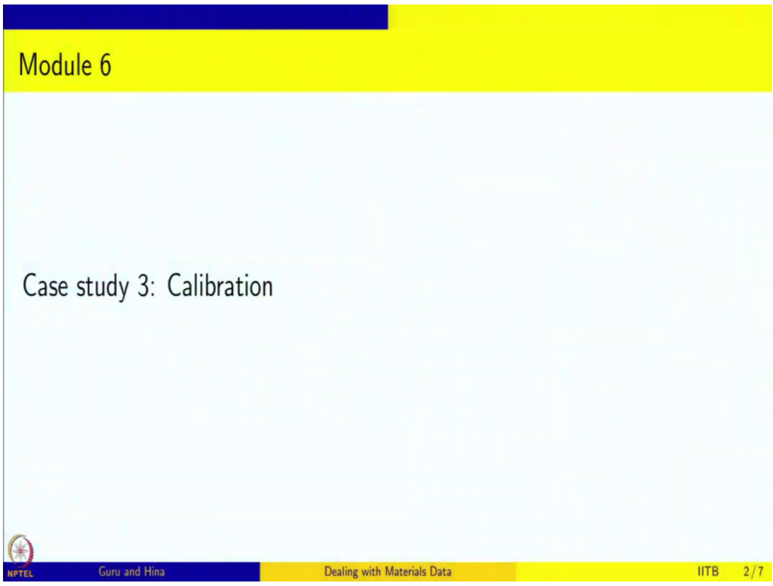

We rea in the module on case studies and this is a case study on Calibration.

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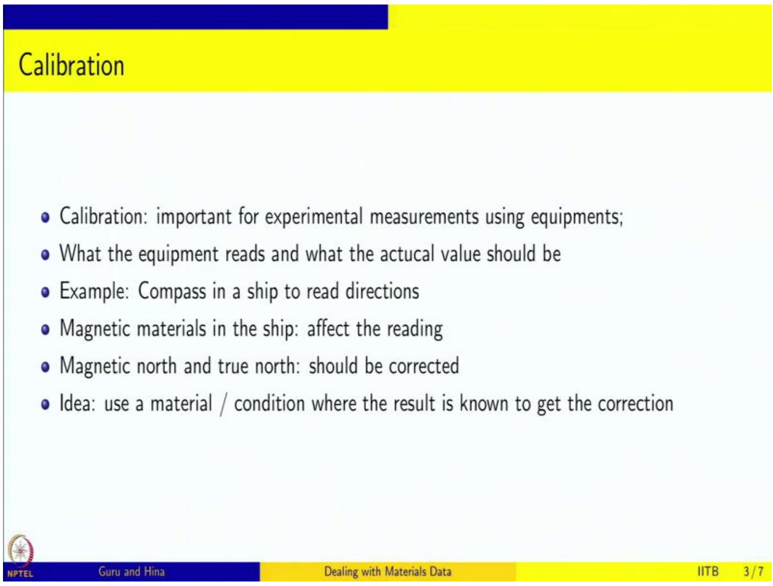

Calibration is important for experimental measurements using equipment and what the equipment reads and what the actual value should be, there could be sometimes discrepancies. The very well-known example that is also described in Berenson's textbook, for example, is compass reading in a ship to read directions, because there are a lots of magnetic materials in ship that affects the reading.

In addition, the magnetic north and the true north are slightly different. So you will read something in your compass, but is that the north that you actually see? Or should you correct for the values? That is given by the calibration. So if you have a calibration curve when your magnet, your compass read something then you can add the correction and actually know the true value.

So the idea is to use a material or condition where the result is known and use the equipment to make the measurement because we already know what the result should be, we will know what the error that the equipment is giving and keep an information of this error, so that we can correct it when we make a measurement on an unknown material or in a new situation, so that is the basic idea behind calibration.

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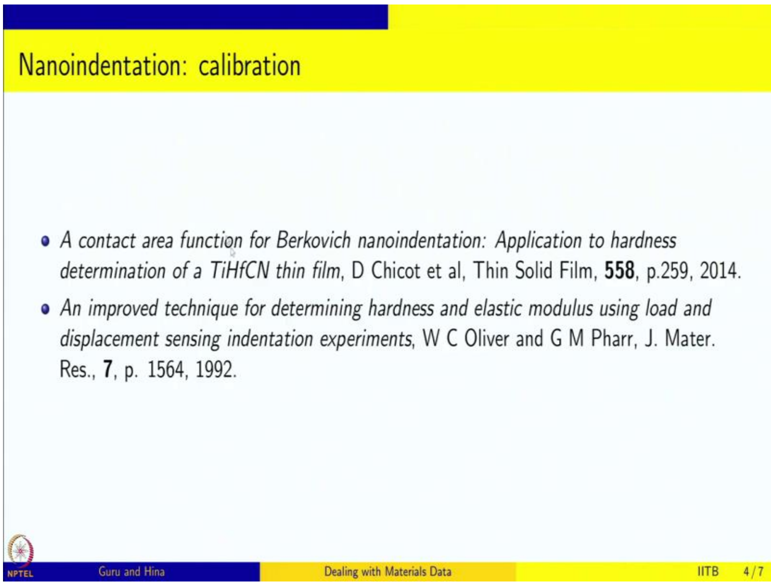

So we will look at calibration in the context of nano-indentation. There are two papers which I am going to use, I strongly recommend that you look up these papers, one is old paper and very well-known paper form Oliver and Pharr, this is from Journal of Materials Research, an improved technique for determining hardness and elastic modulus using load and displacement sensing indentation experiments.

And based on this but slightly simplified one is given in Thin Solid Films, contact area function for Berkovich nano indentation and this is the fitting that I am going to do. If you read Oliver-Pharr, you will see that actual function that you have to fit is very complicated and off course one can do take data and do that fitting also.

But to simplify things and to give you an idea of how the methodology works, I am going to do a very simple fitting. We are still going to do ax plus b kind of fitting, linear fit, but it will tell you some of the ideas behind calibration and if you want a more complicated one, of course, you will be able to do because that is fairly easy for you now knowing all the regression and linear models and fitting and things like that, so it's easier to follow.

So what we are going to do is we are going to take some formulae from here but the fitting for the contact areas versus the contact depth will be done using very simplified module that is described in this paper.

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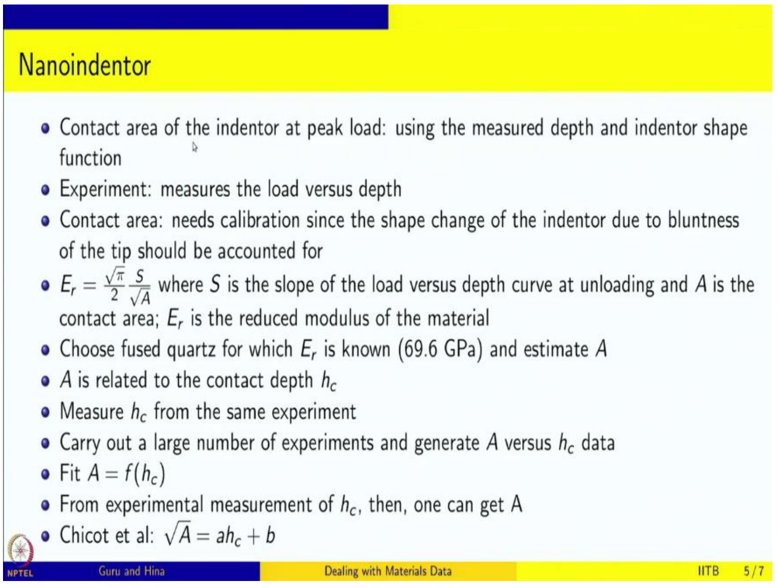

So the contact area of the indentor at peak load that we want to get using measured depth and the indentor shape function. So the depth measurement is made and we know what the indentor is, but sometimes the experiment is load versus depth, so we will know the load, we will know the depth and that is from where we are getting the depth.

And from this we have to somehow get some parameter which will help us get this measurement because this is not directly measured, so we want to make this function of the contact depth. But that contact depth area relationship depends on the indentor shape, we know the indentor shape, but the problem is the indentor gets blunted with use and or there are small changes in its shape and they can actually affect the contact area that you measure.

So we need to calibrate the area; that contact area that you get for a given measured depth and particular load versus depth curve that you obtain. How do you do that is what is described and that is the calibration part. Like I said for calibration, we always should have a material on which we know what the value should be and use that to back calculate what the equipment is actually reading and so we get.

So here is the relationship that connects the reduced modulus of a material to the slope of the depth, load versus depth curve which is the S and the contact area is A. So if you know the contact area and the slope of the curve, you can actually get the reduced modulus. So we take a material for which we know the reduced modulus which is fused quartz and that is 69.6 GPa. So we are going to use this value and so we are going to estimate A, or route A.

So we have a measure of A, but we want to develop a calibration curve in such a way that by looking at the contact depth, we will say what the area is which means you need to relate A to contact depth and the contact depth will be measured form the same experiment, so it should involve the same values that you measure.

And so you can generate a large number of experiments in which you will measure this hc and A using a known material and so you can develop a relationship between A and hc and then use that to do the analysis on a new material. And the idea is to carry out large number of experiment and generate this A versus hc data and you fit A to some function of hc and so from then an experimental measurement on a new material of this hc one can get the A.

And then you use it to get the mechanical properties of the material. Chicot et al give this root A is equal to a hc plus b so you have to determine the slope and the intercept so that is what we are going to do, but if you see Oliver-Pharr's paper, you will see that it is a very complicated function, it goes as some, with known constant hc square plus some eight unknown constants which go as h, h to the power half, h to the power one forth, h to the power a eighth etc. up to h to the power 128. So we are not going to do this very complicated fitting, we are going to use a very simple fitting to do the analysis.

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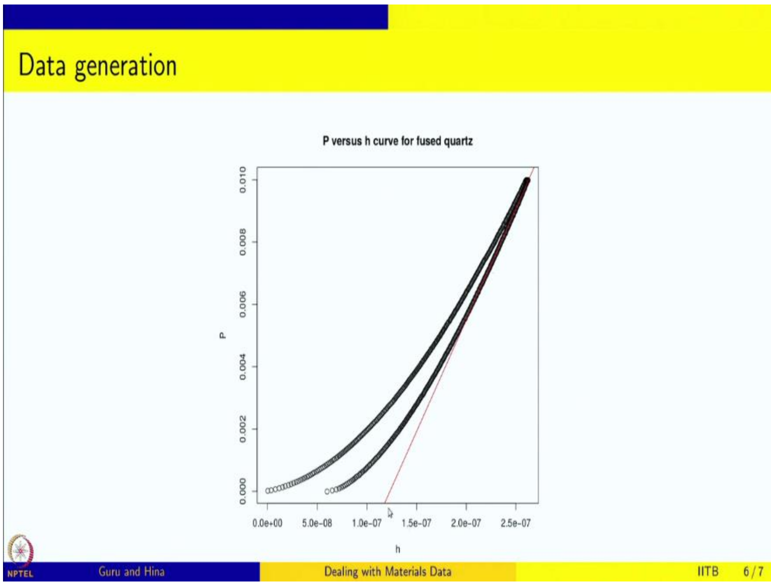

So this is how the load versus depth curve looks. So this is the loading part and then it is held for a while and it is unloaded, this is the peak load and the depth at peak load is this quantity and we are going to fit the unloading part to a straight line and we are going to fit slightly after this max, we come a little bit down and take the data and do the fitting and the intercept actually gives the depth that you get from this slope line.

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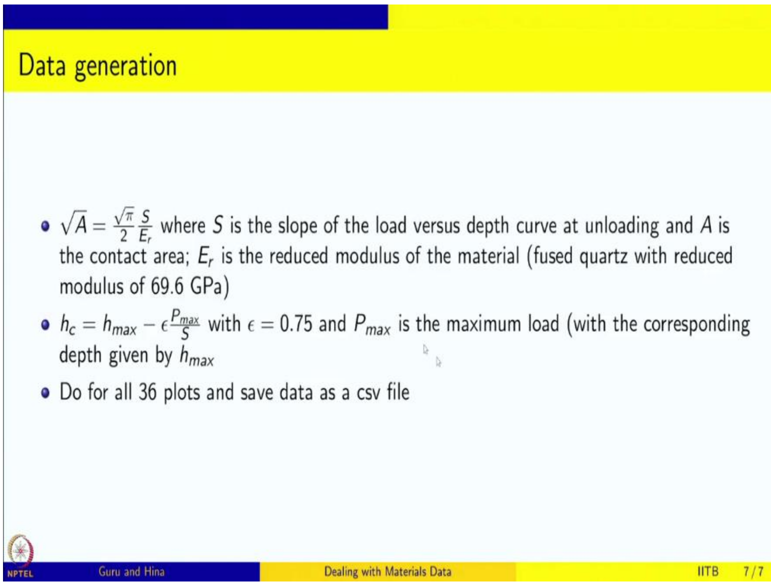

There is a relationship. So we already know this, so root A is root pi by 2 S by Er,, so pi by 4 S squared by Er squared is actually giving you the A because we know the Er and we can measure the slope from the this curve. So this is basically the slope of this red line you see here.

So you can get this quantity, so a is known and the contact depth h is given by hmax minus epsilon Pmax by S.

Epsilon is 0.75, Pmax is the maximum load, and the corresponding depth is hmax, so we know hmax, we know Pmax, we know S, all these come from the data itself, so by multiplying with this epsilon you can get a hc. So we have hc and A measured from the curves, so we do it for all 36 plots and save the data as a CSV file and then do the analysis on the CSV file to get the calibration curves.

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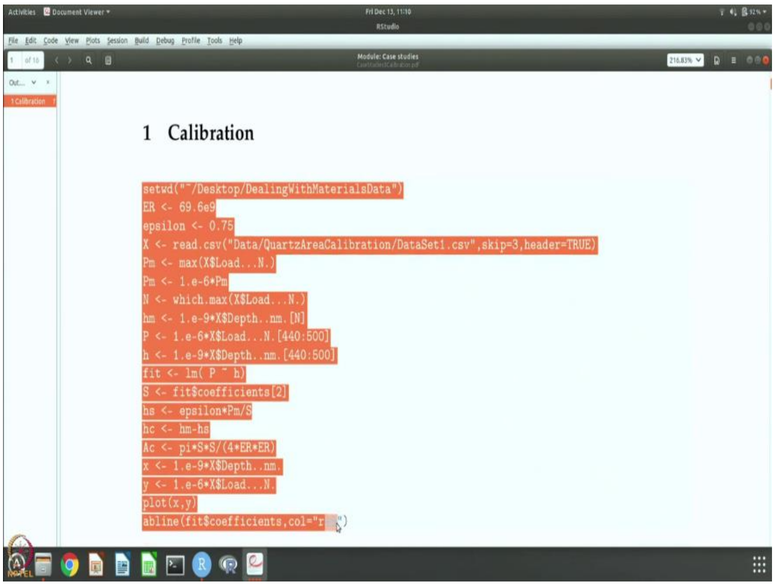

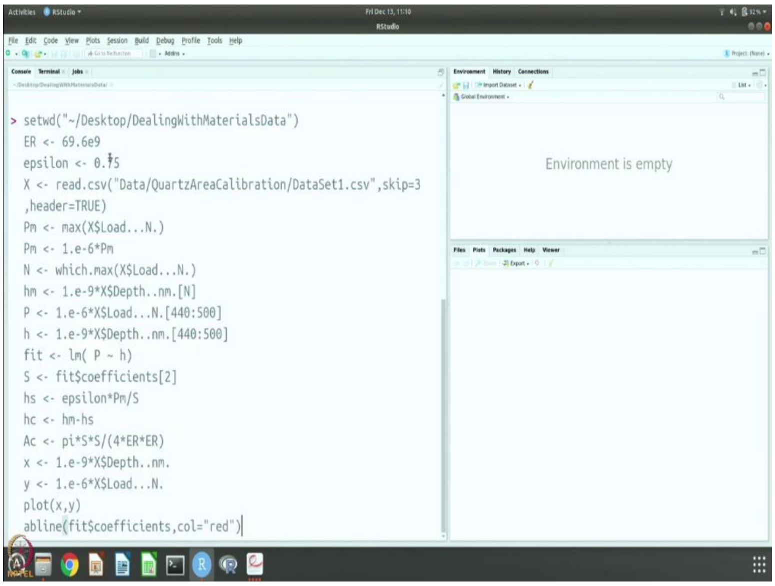

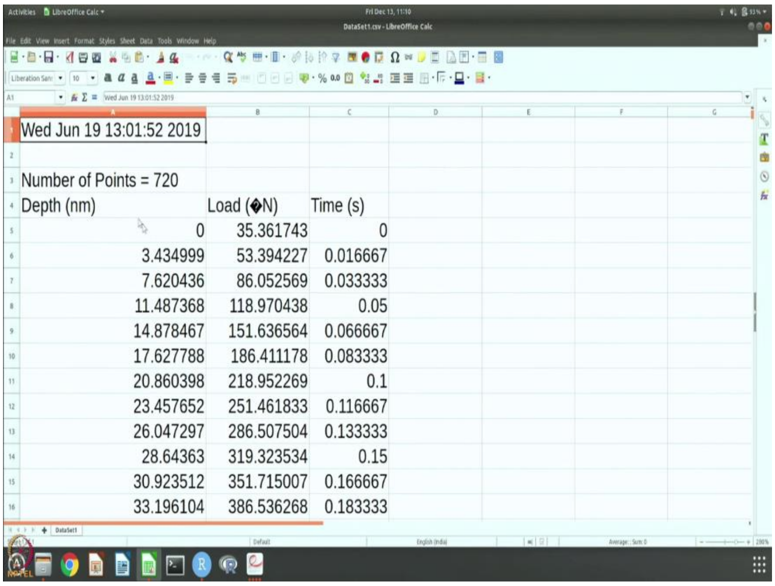

So let us do that as usually, lets open R and start doing this analysis. So the first one that we want to do is that we will take one of the data and do this fitting exercise. Just to give you an idea of what is happening. Okay, so we know the Er, we know the epsilon and we read the data and in this QuartzAreaCalibration directory, there are 36 data sets. First 3 lines are skipped because if you open the data set, you will see that the first 3.

So it gives some data off when this measurement was made and number of points etc. So we are going to skip these there steps and then the lines and then we are going to read the header and then we are going to read the data. So that is what it does, skip three lines and then read the next one as header and then the rest is data and Pm is nothing but the maximum of the load and it is micro newton, so we are going to multiply it by 10 power minus 6.

And N at which point the maximum load is measured, the corresponding depth we want, so which max actually gives the line number, so if you take that line number and measure the depth that is hmax and that is in nanometre so we are multiplying by 1 e power minus 9. And then you can go to the data and see somewhere about 440 to 500 is where we are doing the fitting and that is because that is the point.

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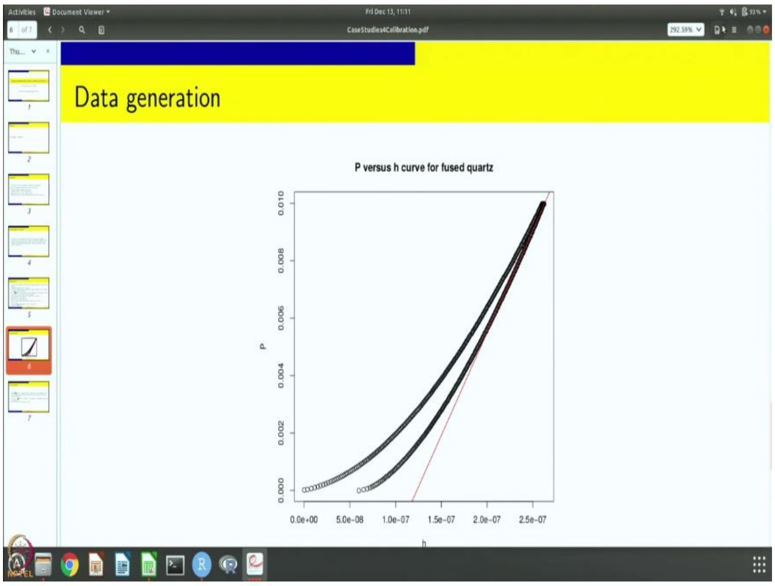

So let us go back to this curve, so the 440th to 500 data point is somewhere here and this is what we are using to do the fitting. So with the max, we come slightly below and so I am just doing it to show how it works, actually you have to take 5 percent or 10 percent off from the Pmax and from there we have to choose, of course, when you do this analysis yourself you can also try those things but in this case I am going to take for 40 to 500 to be slightly below the hmax and that is here we are taking the data and we are going to do a linear fit.

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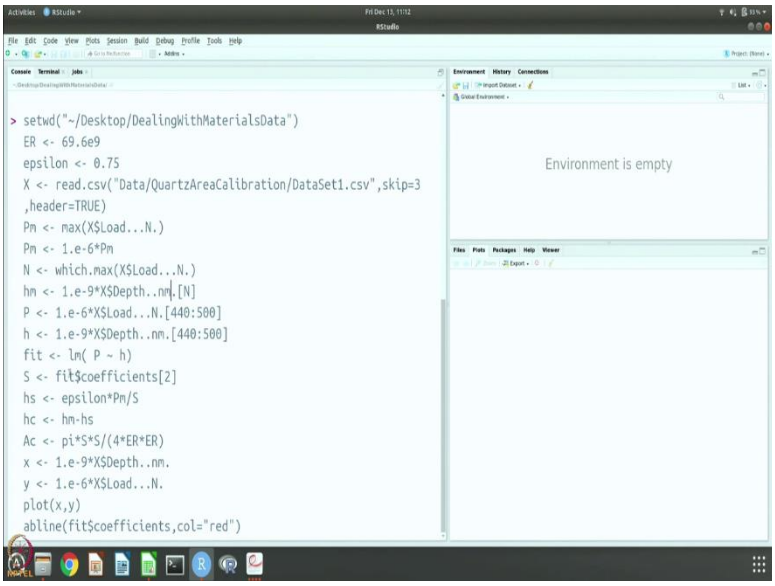
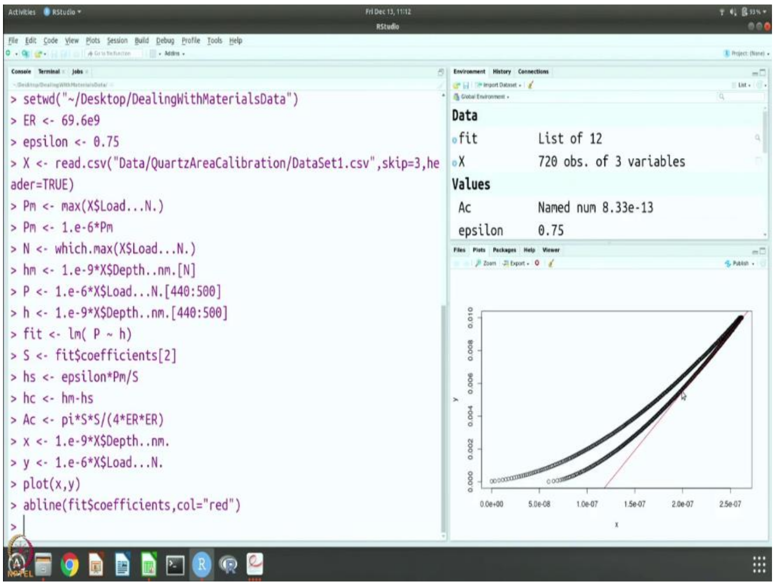
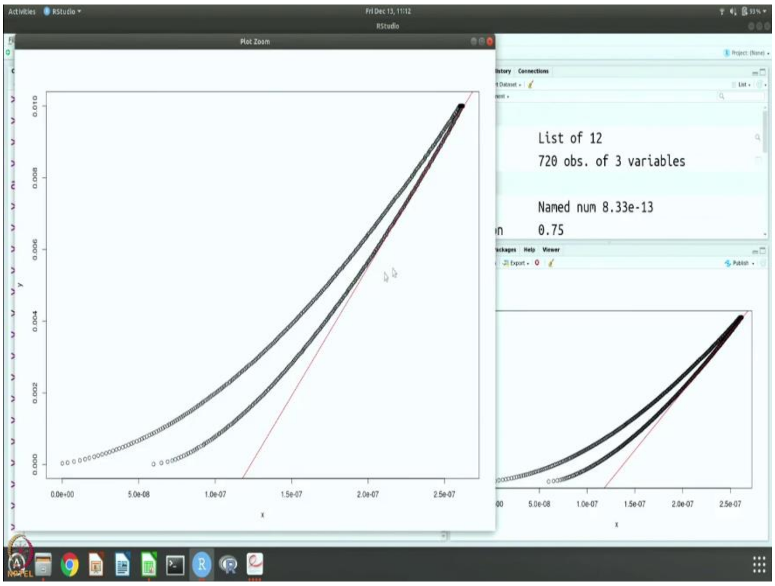
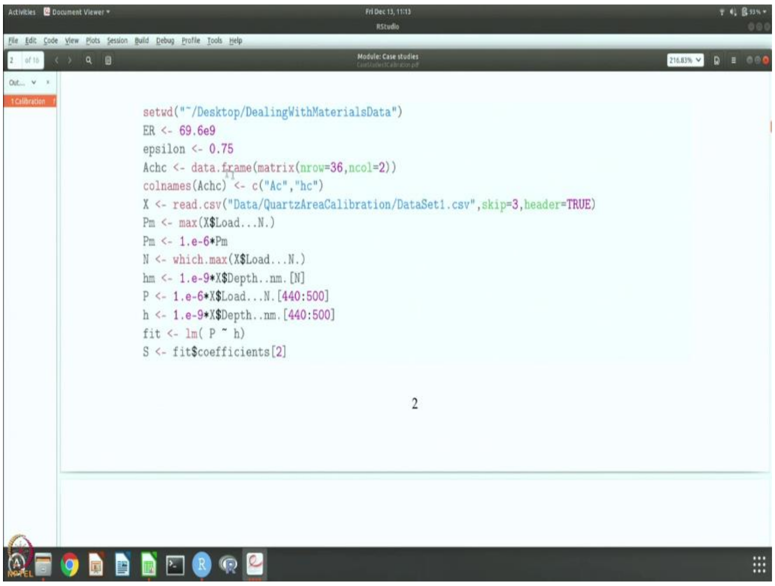
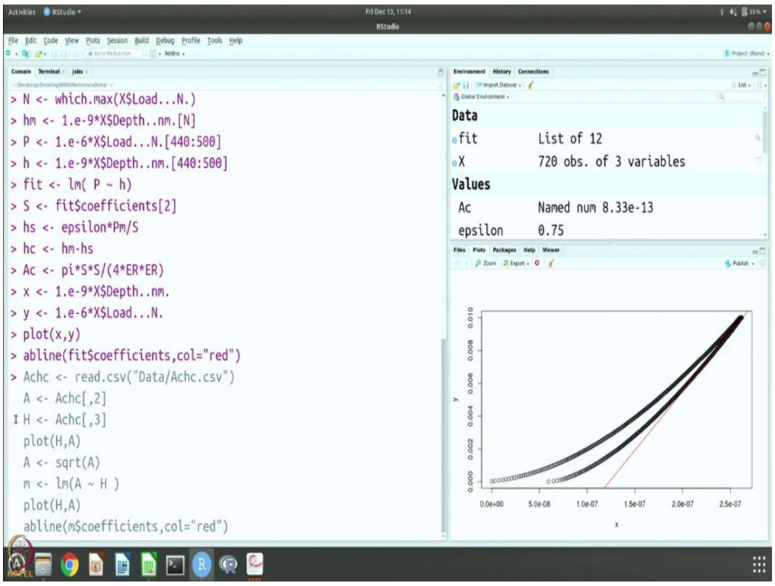


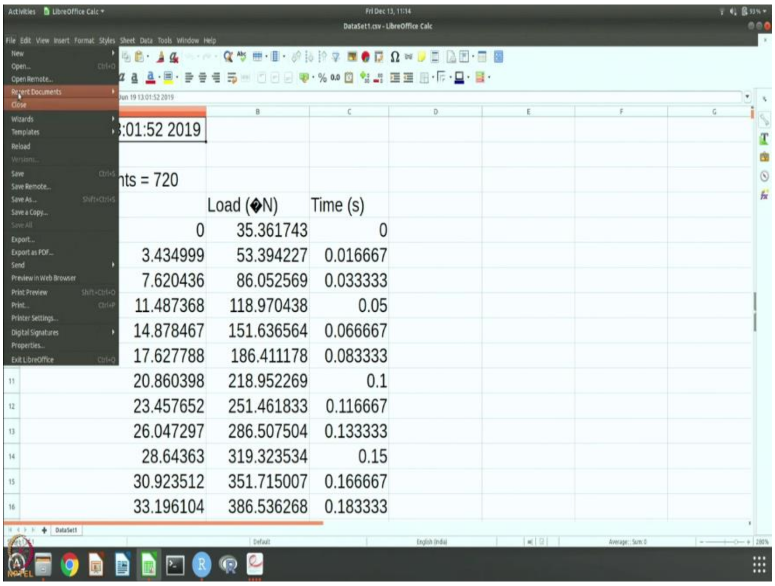

> setwd("~/Desktop/DealingWithMaterialsData")
> ER <- 69.6e9
> epsilon <- 0.75
> X <- read.csv("Data/QuartzAreaCalibration/DataSet1.csv",skip=3,header=TRUE)
> Pm <- max(X$Load...N.)
> Pm <- 1.e-6*Pm
> N <- which.max(X$Load...N.)
> hm <- 1.e-9*X$Depth..nm.[N]
> P <- 1.e-6*X$Load...N.[440:500]
> h <- 1.e-9*X$Depth..nm.[440:500]
> fit <- lm(P ~ h)
> S <- fit$coefficients[2]
> hs <- epsilon*Pm/S
> hc <- hm-hs
> Ac <- pi*S*S/(4*ER*ER)
> x <- 1.e-9*X$Depth..nm.
> y <- 1.e-6*X$Load...N.
> plot(x,y)
> abline(fit$coefficients,col="red")
Data
fit List of 12
X 720 obs. of 3 variables
Values
Ac Named num 8.33e-13
epsilon 0.75


Plot Zoom
List of 12
720 obs. of 3 variables
Named num 8.33e-13
0.75


Module: Case studies
setwd("~/Desktop/DealingWithMaterialsData")
ER <- 69.6e9
epsilon <- 0.75
Achc <- data.frame(matrix(nrow=36,ncol=2))
colnames(Achc) <- c("Ac","hc")
X <- read.csv("Data/QuartzAreaCalibration/DataSet1.csv",skip=3,header=TRUE)
Pm <- max(X$Load...N.)
Pm <- 1.e-6*Pm
N <- which.max(X$Load...N.)
hm <- 1.e-9*X$Depth..nm.[N]
P <- 1.e-6*X$Load...N.[440:500]
h <- 1.e-9*X$Depth..nm.[440:500]
fit <- lm(P ~ h)
S <- fit$coefficients[2]
2



> N <- which.max(X$Load...N.)
> hm <- 1.e-9*X$Depth..nm.[N]
> P <- 1.e-6*X$Load...N.[440:500]
> h <- 1.e-9*X$Depth..nm.[440:500]
> fit <- lm(P ~ h)
> S <- fit$coefficients[2]
> hs <- epsilon*Pm/S
> hc <- hm-hs
> Ac <- pi*S*S/(4*ER*ER)
> x <- 1.e-9*X$Depth..nm.
> y <- 1.e-6*X$Load...N.
> plot(x,y)
> abline(fit$coefficients,col="red")
> Achc <- read.csv("Data/Achc.csv")
A <- Achc[,2]
H <- Achc[,3]
plot(H,A)
A <- sqrt(A)
m <- lm(A ~ H)
plot(H,A)
abline(m$coefficients,col="red")
Data
fit List of 12
X 720 obs. of 3 variables
Values
Ac Named num 8.33e-13
epsilon 0.75



DataSet1.csv - LibreOffice Calc
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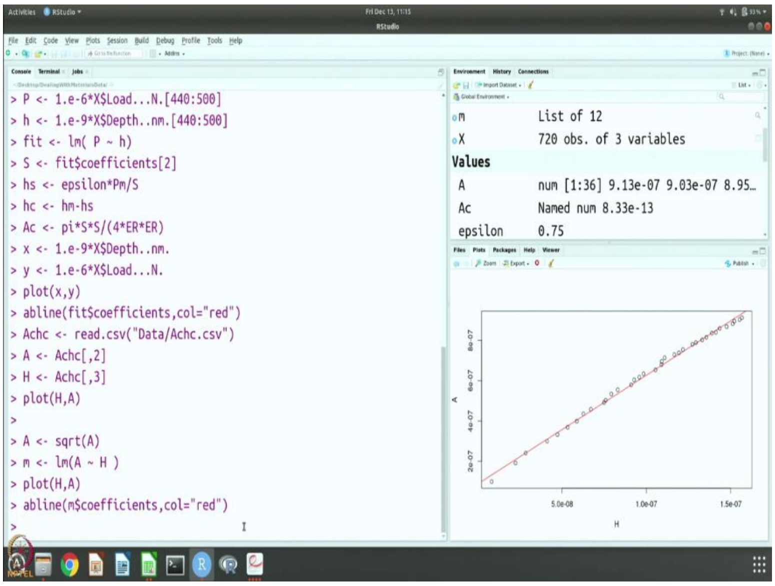
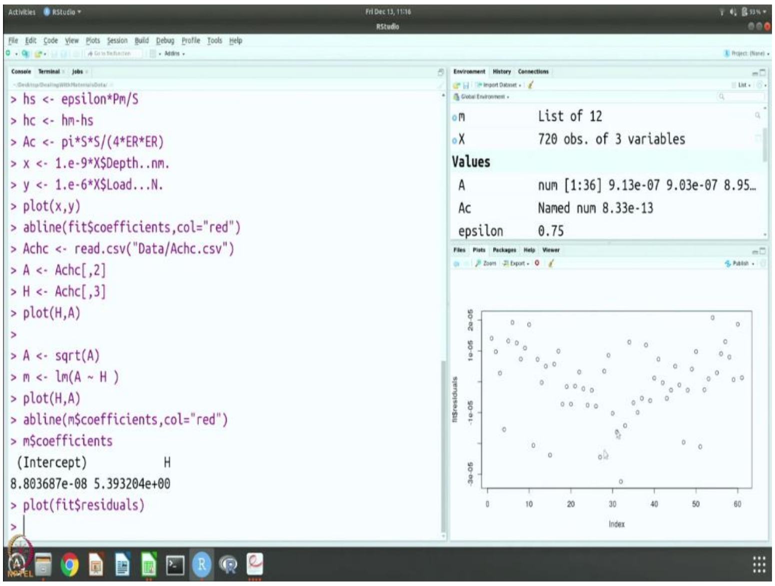
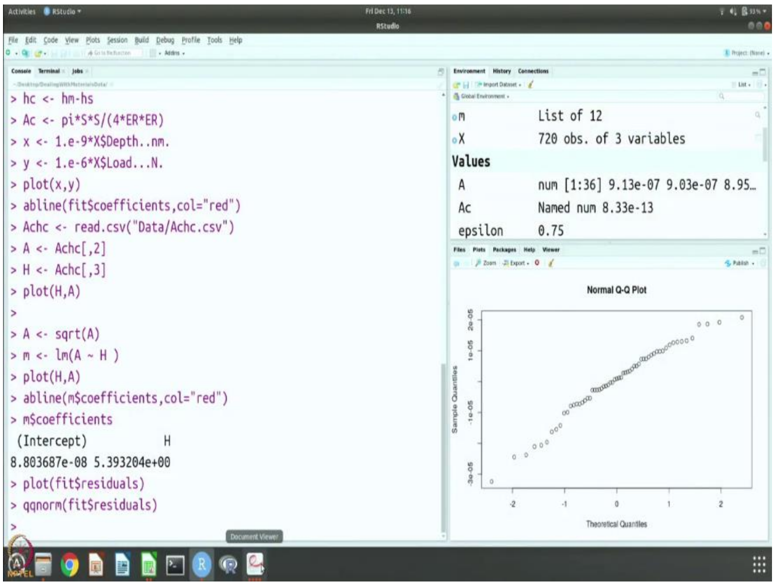
nts = 720
Load (�N)
Time (s)
0 35.361743 0
3.434999 53.394227 0.016667
7.620436 86.052569 0.033333
11.487368 118.970438 0.05
14.878467 151.636564 0.066667
17.627788 186.411178 0.083333
20.860398 218.952269 0.1
23.457652 251.461833 0.116667
26.047297 286.507504 0.133333
28.64363 319.323534 0.15
30.923512 351.715007 0.166667
33.196104 386.536268 0.183333



> P <- 1.e-6*X$Load...N.[440:500]
> h <- 1.e-9*X$Depth..nm.[440:500]
> fit <- lm(P ~ h)
> S <- fit$coefficients[2]
> hs <- epsilon*Pm/S
> hc <- hm-hs
> Ac <- pi*S*S/(4*ER*ER)
> x <- 1.e-9*X$Depth..nm.
> y <- 1.e-6*X$Load...N.
> plot(x,y)
> abline(fit$coefficients,col="red")
> Achc <- read.csv("Data/Achc.csv")
> A <- Achc[,2]
> H <- Achc[,3]
> plot(H,A)
> A <- sqrt(A)
> m <- lm(A ~ H)
> plot(H,A)
> abline(m$coefficients,col="red")
m List of 12
X 720 obs. of 3 variables
Values
A num [1:36] 9.13e-07 9.03e-07 8.95...
Ac Named num 8.33e-13
epsilon 0.75





And the slope is actually our S and we know that hc is hmax minus hs which is epsilon into Pm by S. This is a Pmax and this is the slope that you read from the curve. So we now, and AC is pi S squared by 4 Er squared, so that also we know and so you can do that and in this case we are also going to plot this points and then we are going to also fit the line that we are doing in red which will basically give you the curve that you see in this presentation.

So this is the curve that we got. So this is the data and this is the max and then it comes down and then we are choosing some points here and then we are fitting a straight line, we are taking the S and we can calculate all the values. So you have to now do this for all the 36 data points, right? And that is what is shown in this.

So I am not going to do redo this here, but this shows you that you can take data set 1 and do this and we also generate a data frame which has 36 rows and 2 columns and the 2 columns are

Ac and hc and we are going to do this for every data set, we store the data. Then we do it for data set 2 and that is the second line of the data. We do it for data set 3; that is the third line of the data and so on and so forth.

So you do it for all 36 of them, okay. It is the same code, just that there is no plotting and you can do it for all 36. Once you do all 36, we are going to right the data as a CSV file. This is something that we have not done so far, you can also like read CSV, you can use write CSV and write the data file. So now that we have the data, we are going to do the other analysis, so that is the analysis that we will do let us do this.

So what is analysis? So we read the data and once you read the data, then you say that okay, the second column is A and the third column is H. That is because if you look at the data, the data also has the line number, so let us open the tab. Okay. So the data when it is written, it also has the line number, so I want to skip the line number part and it is Ac and hc, so that is what we are reading.

So we plot this h versus A and we take square root of A and we fit it to a straight line. Square root of A is some m h plus c and so we plot this data and then we plot also the line. So let us just do this part first. So this is the data that we are getting and now let us do the square root A and plot and then plot the square root A versus h and then the fitting that we have achieved. So this gives you the fitting and if you look at m dollar coefficients.

So this is basically telling you that if you can measure a contact depth, 5.393204 times contact depth plus this intercept some 8.8 and 10 power minus 8 is basically the contact area that you measure, so now that you have this relationship for any material you just need to measure this quantity and you can get the contact areas and you can see that the fit is good. Of course there are other ways of checking that the fit is good.

So let us plot the residuals and you can see that residual is equally distributed and randomly distributed, so the error is normally distributed. There is another way to check that it indeed is normally distributed which is Q-Q norm and you can see that this curve is also a sort of straight line indicating that we have a good fitting and all the error is normally distributed so it is basically noise and so we have done the calibration.

So to summarise, calibration is very important. Whenever you use any equipment, it has to be frequently calibrated, this we have discussed when we have discussed error and ways of avoiding systematic errors and so many cases calibration leads to either calibration table or fitting so that you will get a calibration curve. Here is an example where we actually fitted it to get a calibration curve.

And this curve actually connected the contact area to contact depth and contact depth is what is measured and we have to get the contact area. In order to do this calibration, we use a material of known modulus and using that we actually calculated the calibration curves themselves, and I have given the complete process of doing it as well as the data set will be available for you to experiment with.

So we are going to stop at this point. I will share this data set. I recommend that you go through this and the example of the compass reading and how to correct for it that is given in Berenson, that data is also available online, so you can look it up or you can take it from Berenson's book and try to do the calibration curve yourself, so to understand it better. So with this we will conclude this case study and we will look at two more case studies design of experiments and hypothesis testing in the sessions to come. Thank you.