Creep Deformation of Materials Metallurgy and Materials Science Creep Testing Methods Part 2

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Creep testing

- Force application and removal procedure
	- A small fraction of the test force, usually not more than 10 % for materials that yield immediately like stainless steel and 15% for materials that show a linear elastic region, can be applied
	- This usually increases the axiality of force application by reducing the displacement of the specimen and the load rods due to lateral forces from furnace packing and thermocouple wires
	- Apply the force in a manner that shock forces or excess forces due to inertia is avoided.
	- The time for applying the force as short as possible within these limitations

So temperature is one aspect the second aspect is the force, so you are going to apply some force under which the creep test is carried out and typically the force application or the removal of the force at the end of the test there are certain methods or approaches that have been defined or specified by the ASTM standard, so a small fraction of the test force, usually not more than 10 percent for materials that immediately yield like tensile stainless steel and about 15 person for materials that show a linear elastic region can be applied.

So basically you can apply a small stress right at the beginning before carrying out your creep test and what this small test force does is that it increases the axiality of the force application by reducing the displacement of the specimen and the load rods due to lateral forces from furnace packing and thermocouple wires. So once you have your sample inside the furnace and you have provided some insulation material, packing material et cetera. this could lead to displacement of the sample or cause some loss of axiality, so in order to prevent the loss of axiality you could make your sample slightly taut by applying an initial test force and the test force should not be greater than 10 percent for materials which tend to yield immediately and it should not be greater than 15 percent for materials that show a linear elastic region and also please remember when applying the force that the force should be applied in a manner that shock forces or excess forces due to inertia are avoided and the time for applying the force should be as short as possible within the limitation that have been defined.

Creep testing

- Strain measurement during test
	- For adequately defining the creep curve, the strain measurements must be carried out at frequent intervals.

E

q

 $1000k$

 $= 10k$

 1 %

- Readings must be made more frequently in the primary creep stage than in the secondary creep stage
- The interval for strain measurement should not be more than 24 hor 1 % of the estimate duration of the test, whichever is longer

 $11 = 1006$

In the primary creep region, the strain rate of deformation in the primary creep region is higher than the strain rate of deformation in your study state, so that is why you should make measurements more frequently in your primary creep region, so your measurement should be more frequent in the primary creep region and then in the steady-state region they can be more spaced out.

The important thing to remember though is that the interval for strain measurement should not be more than 24 hours or 1 percent of the estimated duration of the test whichever is longer, so if you are planning to carry out a test for 1000 hours, then 1 percent of 1000 hours will be 10 hours, so here in such a case the duration between measurement should not be more than 24 hours, however if your tests and duration of the test is 10,000 hours then 1 percent becomes 100 hours, so in this case you can have a measurements taken every 100 hours, so that is what the standard is specifying.

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Creep testing

• Extensometers for tensile creep tests

Now the measurement of the strain can be carried out by extensometer, so here I am showing some typical extensometers, so you are going to have a metal-based extensometer or you can have a ceramic-based extensometer. The metal-based extensometer are typically used for a test carried out up to 850 degrees centigrade and when tests are carried out at higher temperature such as 1100 degrees centigrade you could use ceramic-based extensometers. You could also use LVDT's for making your strain measurements.

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Creep testing: Test specimen

- Tensile test specimen can be as per ASTM E8
- These modifications are however recommended for elevated tests
	- Tighter dimensional tolerances
	- Larger ratios of length to diameter of reduced section may be desirable to increase accuracy of strain measurement
	- For coarse threaded specimen of limited ductility, the size of the thread must be 7/4 the diameter of the reduced section
	- For sheet and strip, rectangular cross-sections can be used; otherwise circular cross-sections should be employed

So you have your furnace, you have your loading system then you have your strain measurement system ready now. Now the other important thing to remember is your sample, so if you are doing a tensile test then the tensile test sample can be prepared as per ASTM E-8 and the ASTM E-8 provide certain recommendations for test carried out at elevated temperatures. Now the ASTM E-8 is for a normal tensile test to be carried out at room temperature and the same dimensions with some modifications can be used for your tensile creep sample.

So the creep sample requires tighter dimensional tolerances and also the larger creep sample requires larger ratio of length to diameter or the reduced section and this is to increase the accuracy of your strain measurement, so the reduced section is basically, so if this is your sample, so this is your reduced section, so here the length to diameter should be higher for creep samples and also if you are using core threaded sample and if the sample has limited ductility then the size of the thread must be 7 by 4 times the diameter of the reduced section.

So if that is your sample so the diameter of the thread d, d should be at least 7 by 4 times the reduce section diameter d_0 , so another specification of the ASTM standard and for sheet and strip rectangular cross-section can be used, however whenever if you have a sheet or a strip from which you are planning on making the sample then rectangular cross-section can be used otherwise circular cross-section are generally recommended.

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And circular cross-sections the sample would look like that and then you have a rectangular sample then the sample will look like that. Here g is the gauge length and a is the length of the reduce section and l is the total length of the sample, b is the length of the grip section and c is the width of the sample and you can get more details about the dimensions and the ratio of g and a and all that the values of g and a and all that from ASTM E-8 or you can also refer to this websites where some of this details are provided.

Creep testing

- The tensile creep testing method is less suitable for studying ceramics
- This is because brittle materials such as ceramics are very flaw sensitive and ceramics usually have a population of flaws
- It has been observed that ceramics tend to creep faster in tension than in compression
- Cavitation contributes significantly to creep strain during tension while it is almost absent in compression.
- The tension creep rate increases linearly, at low applied stresses, whereas it is increases exponentially at higher applied stresses
- In compression tests, the creep rate increases linearly with applied stress

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So that was about how to carry out a conventional creep test and some of the things to remember while carrying out your creep test. One other important point is when you are employing the creep, when you are carrying out the creep test and if you are doing the creep test for brittle material such as ceramics then probably the tensile creep testing method is not that suitable.

This is because brittle material such as ceramics are very flaw sensitive and ceramics usually have a population of flaws, so when you tend to do a tensile creep test then what you will notice is that the material tends to creep faster in tension than in compression, so the higher rate of the creep in tension is because the flaw population contributes significantly to creep strain during tension while it is almost absent in compression.

So if you recall when tertiary creep, if you recall the strain rates of deformation are very high and we said that in tertiary creep the strain rates of deformation are very high because there is growth of defects and when these defects such as cracks or voids tend to grow they tend to bring down the effective cross-section of the material or the effective load-bearing section of the material and this leads to higher strain rates of deformation.

A similar issue is observed when you are doing tension test of ceramics because ceramics invariably have cracks and voids and things like that, so when you are doing your tension test, so the cracks and voids tends to grow and when they grow the effective load-bearing ability of the material comes down but if you are doing a compression test the cracks and voids et cetera what they do is they tend to get closed.

So they tend to get closed, so when they get closed the chance for their growth is minimised that is why the creep rate of deformation of ceramics in tensile test will be greater than the creep rate of deformation in compression and also tension creep rate increases linearly at low applied stresses, whereas it increases exponentially at higher applied stresses. However in compression test, the creep rate increases linearly with applied stress.

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So this is a creep test of a Hot Isostatically pressed silicon nitride, so NT154 is a silicon nitride and creep tests were carried out on this material, so if you see the strain rate versus stress profile, so if you see the data seems to be linear at lower stresses and then it takes an exponential shape at variation at higher stresses whereas in compression test it is more or less linear.

So when you have an exponential change in strain rate versus applied stress then the data analyses become difficult because generally we relate exponential dependence on stress because it as power law breakdown, so you are in artificial power law breakdown regimen is being observed on account of the growth of voids and cracks in attention test. So probably you will misinterpret the creep behaviour of the material and on account of the tension test more reliable and accurate creep performance of the material can be obtained if you do your test in compression. So this is the difference between tension and compression test for brittle material such as ceramics.

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Flexural testing

- Another disadvantage of tensile creep testing of ceramics is specimen preparation and specimen loading.
	- This is especially so in ceramics where common machining operations for specimen preparation are not easy to carry out.
	- Small misalignment of specimen in the grips can cause fracture/cracking because of their brittle nature

Flexural testing is an alternative and more simpler technique for creep testing of ceramics

Now brittle material such as ceramics, a better way of probably carrying out a creep test is the flexural testing because one of the disadvantages of tensile creep testing of ceramics is specimen preparation and specimen loading. Now this is so in ceramics because common machining operation for specimen preparation are very difficult for ceramics, so if you want to prepare a dog bone sample for a ceramic it is a lot difficult compared to making just a simple rectangle apart, so and also dog bone sample because of radius of curvature involved here and things like that.

Even if you have a small misalignment in your loading during loading that can lead to easy cracks in the tensile sample, so that is why a rather better way of carrying out the creep test is to go for a flexural test method, so flexural test is an alternative and more simpler technique for creep testing of ceramics because ceramics are not flaw tolerant whereas when you do when you study metals, metals are more flaw tolerant so that is why you will not face a problem when you are doing tensile creep testing of metallic materials.

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Flexural creep testing

- Advantages
	- The samples are easy to prepare
	- The experimental design is relatively simple
	- Overall cost of testing is low
- Two types of flexural creep testing is possible
	- Three point bend testing
	- Four point bend testing

So the flexural creep testing the advantages are the samples are easy to prepare because they are bar samples so these are easy to prepare and the experimental design is relatively simple and the overall cost of testing on account of easy preparation of samples as well as the simplicity of the experimental design, flexural creep testing can be carried out in 2 ways you can do a three-point bend testing or you can do a 4 point bend testing.

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And here the specimens are rectangular and without notches and the three-point bend testing you can have something like that, so this is three-point bend testing and your load is acting there, so the spacing between the loading points is L by 2, L by 2 and then in 4 point bend testing, so this is 4 point bend testing and this is three-point bend testing.

So the inner loading span this Li and the stress generated is 3P L minus Li by 2 bh square so L is the length of the sample b is the breath and h is the height or the thickness of the sample so that is the…if you are applying a load P, if P is the applied load so that is the stress generated within the material and Li is the length of the loading span, the inner length of the loading span and the strain generated because of this is given as Epsilon is equal to 6 hx by L minus Li into L plus 2 Li.

So this is the, epsilon is the strain generated in the outermost fibres for the measured deflection where x is the deflection, so if x is the deflection so that deflection leads to a strain like that and so the strain rate of deformation Epsilon dot is basically dependent on the deflection rate, so this can be written as some constant times the height of the sample into the deflection rate so using these equations you can determining the strain rate of deformation during creep carried out under flexural testing mode.

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So that was about tensile creep testing, some general information about tension creep testing method and why tension creep testing method may not be the best or most suitable testing method for ceramics, so we talked about the flexural creep testing method, so these are all conventional creep testing method, so now I am going to talk about a slightly nonconventional creep testing method which is the impression creep testing method, so the impression creep testing method is known as a small scale testing technique and this testing technique was mainly developed for the nuclear industry to understand the ageing of the materials or components used in the nuclear industry.

So it is very important to carry out health monitoring of your components or the materials being used in the nuclear industry because the nuclear industry is a very sensitive industry in terms of a kind of hazards that it can cause for the kind of environment damage as well as loss of human life that any accident in the nuclear industry can cause, so that is why it becomes very important to regularly monitor the performance of the components or material being used in the nuclear industry, so in order to do that there were several many small scale testing technique that was developed.

Some of those were developed for the nuclear industrial study so you can use the hardness as the way of checking the strength of the material and impression testing is another such method. People also developed shear punch testing technique, disk bending testing technique and things like that, so these are some of the small scale testing technique that were developed for the nuclear industry. In recent years automated ball indentation, Nano indentation and mini tensile testing techniques are also some of the other small scale testing techniques which have been developed but the focus of our lecture is on the impression creep testing technique, so how is this technique useful for carrying out or understanding the creep behaviour of material.

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So the impression creep testing method is a useful method for extracting the creep properties from a small quantity of material, so say if you are trying to develop a new material and you are interested in knowing whether this material is useful for the application that you have in mind and because material development or product development can involve several cycles of trial and error so you have to go through several iteration to finally arrive at your material of choice.

So if you have to rely on conventional test techniques then you will have to make dog bone samples each time and dog bone sample making is not that easy it is more involved than making a simpler sample may be like sheet sample or may be a simple cube, so that is why people thought that the impression creep testing method can be more advantageous because you can make smaller samples and also extract data from small samples and the sample preparation is also simple, so all types of materials can be tested or studied under the impression creep testing methods you can study metal, ceramic, polymers, glasses et cetera.

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So the impression creep testing method in a way is very similar to a hot hardness test, now the hot hardness test was what people have developed earlier but it has a certain limitation and the limitation was that the material tends to exhibit a continuous reduction in hardness with time, so for the same load the material shows reduction in hardness as a function of time that is because the same load at higher temperatures, the imprint that the material provides is growing in size.

The dimension of the imprint so say if you have a circular imprint generated from a ball now the same load the size of the imprint is going to increase as a function of time and hence what happens is the stress that the material is experiencing is not a constant and this prevents the attainment of a steady state of creep deformation, so if you recall for analysis of creep data you need a steady state of creep deformation, so Epsilon dot minimum and this should be constant over a significant duration of time.

So in a hot hardness test this is not happening so this limitation was overcome by using a tool with a constant cross-section such as a cylinder, so if you have a punch which is cylindrical in nature, so when this punch penetrates the sample the area of contact or the imprint area is going to remain a constant it is going to be pi d square by 4 and this technique was extensively developed by JCM Li and co-workers and in India Professor Sastry and other coworkers made important contributions to the development of this technique because the area is going to be constant this means the stress that the material is experiencing is also going to be constant and so you can get a constant strain rate of deformation.

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So the methodology is very simple the testing involves using a flat bottomed cylindrical punch of diameter D and then the depth of penetration of the punch is recorded as a function of times, so the depth of penetration Delta is recorded as a function of time and the impression creep is obtained by plotting Delta versus time.

And the impression creep apparatus, simple apparatus is shown here, so you have a load that is getting transmitted to the sample through the cylindrical indenter and then you have a furnace to maintain a constant temperature and then the stress that the material is experiencing can be calculated. The impression stress is Sigma is equal to 4P by pi D square where P is the applied load and D is the punch diameter and the impression creep strain rate is calculated from the following relation, so at the end of the day for analysis of the creep data you need Epsilon dot and Sigma.

So you need to find out Epsilon dot and the way to do it is you can use this relation v_s over d where v_s is the steady state velocity of the punch during impression creep and d is the plastic zones size under the impression. Now what people have noticed is the small d is roughly found to be equal to that of the punch diameter D and hence the impression creep strain can be represented as Epsilon dot is equal to v over D where v is equal to v_s is the steady state velocity.

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So the steady State velocity can be obtain from the slope of this line, so if you notice the slope of the line is basically Delta by time t, impression depth is Delta, Delta over t is the slope of the line and that is equal to the punch penetration rate, so that is the steady state velocity of the punch and the red lines here basically indicate that the steady-state slope which is v_s it tends to increase with the applied stress.

So if you see when the applied stress is 61.8 MPa you have lower slope compared to the slope that you get at higher applied stresses, so like I mentioned the impression creep punch the interaction zone is roughly equal to the diameter of the punch, so D is the diameter of the punch and the plastic zone created under the impression punch is small d and small d is roughly equal to the capital D and so that is why you can carry out Epsilon dot you can determining Epsilon dot by doing v over capital D.

So the impression creep people have found that it provides data which is similar to that generated by a tensile creep method, so the advantage of impression creep machine is that the data that your generating from the impression creep machine from the impression creep test it maps very well onto the tensile creep data, so here is an example so this is test carried out on magnesium and what they found out is the tensile creep data represented by the open circles maps onto the impression creep beta and which means the impression creep testing technique can be an alternate to the tensile creep testing method.

However for the mapping you will have to go you have to use a conversion, the sample is getting deformed under an applied stress of Sigma i which is P over pi D square by 4 and what people have noticed this that the mapping works out well when you convert Sigma i into an equivalent tensile stress, so the equivalent tensile stress that the material is experiencing is Sigma i over k where k is roughly equal to 3 for the impression creep test, so whatever stress you are applying in your impression creep test that stress has to be converted into an equivalent tensile stress and that equivalent tensile stress can be obtained by dividing Sigma I by constant 3, so once you do that then you can plot Epsilon dot versus this equivalent tensile stress and then what you will find out is your impression creep greater matches very well with the tensile creep data.

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Equivalence between impression creep and tensile creep methods

testing are similar in values to those determined by conventional tensile creep testing

So this equivalence between impression creep in tensile creep method was studied for variety of materials, so here is an equivalence shown for zinc and cadmium, so your stress exponent n determine for zinc using the impression creep is similar to the stress exponent determine using the tensile creep. Similar behaviour is seen also in cadmium 5.7 is what you get as n for cadmium using the impression creep machine and you get in n values in the similar range using the tensile creep machine. Similarly the activation energy that you determine using your creep test they also bear a very similar values in impression creep as well as tensile creep testing conditions, so what this tells is that indeed the impression creep testing method is a good alternate to the tensile creep testing method.

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So some of the applications of the impression creep testing method are as follows, so you can use the impression creep testing method for evaluating the effect of crystallography orientation on creep behaviour, so you can carry out studies on the RD, TD and ND plane of a sheet sample, so when you are preparing a rolled sample you have an RD, TD and ND and because the impression creep testing method requires a small quantity of material.

So you can carry out tests on samples along the 3 direction, so you can take samples with planes perpendicular to RD direction, so you can do a test on samples where the RD directions, the plane is perpendicular to the RD direction or you can take samples with plain perpendicular to the TD direction or you can take samples with plain perpendicular to the ND direction which is the plane of this sheet, so you can take samples from all 3 directions and you can understand how the creep behaviour is varying as a function of the direction.

Similarly you can study the role of grain boundary characteristics, so the impression creep test is dependent on the diameter of the punch, so the punch diameter D will tell you the strain rate of formation, so if you want to study the effect of a grain boundary and if you want to know what is the behaviour of the material close to the grain boundary you can carry out test within the bulk of the grain, so you can choose a punch which has a diameter D significantly smaller than the grain size, so say if D is less than the average grain size then you can carry out test within the bulk of the grain and you can also carry out a test close to the grain boundary and this will tell you how creep behaviour is influenced by the grain boundary of the material. Similarly you can employ the impression creep testing method for studying the creep of weldments.

Advantages of impression creep technique

- · The impression creep testing technique offers the following advantages¹
	- 1. A small quantity of testing material is sufficient which provides an inexpensive method of developing new and advanced materials.
	- 2. Constant stress can be obtained with a constant load.
	- 3. The temperature and stress dependence of the steady state (or minimum) creep rate can be obtained on a single specimen, thereby making more valid the assumption of constancy of structure, necessary in the estimation of thermal activation parameters for deformation such as activation area and activation energy.

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So just summarise the advantages of the impression creep testing method, so the impression creep testing method offer the following advantages one it requires a small quantity of testing material, secondly constant stress condition can be developed throughout the test, so in a conventional creep test in the tertiary creep regime your stress may not remain constant because the material has deformed considerably and there is a reduction in the cross-sectional area of the sample in the tertiary creep regime. So your stress can get enhanced but in the impression creep testing method you do not have a problem like that, your stress can remain constant throughout the creep test and also the temperature and stress dependence of the steady-state creep can be obtained from a single sample, so instead of making multiple samples you can use a single sample, apply a load P1 determine the strain rate of deformation for P1 then increase the load from P1 to P2.

So that will give you the strain rate Epsilon dot 2 you can increase the load to P3 that will give you the strain rate of deformation Epsilon dot 3. So what you can now get is Epsilon dot versus Sigma where Sigma 1 is for P1, Sigma 2 is for P2 and Sigma 3 is for P3, so from single sample you can get the strain rate versus stress plot and from that you can determine the stress exponent, so this is the advantage of the impression creep testing technique.

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Advantages of impression creep technique

4. Absence of tertiary stage of creep makes the deformation more stable a test is, therefore, better suited for investigating near brittle materials.

5. The test is eminently capable of yielding information in some investigations, which is not possible or extremely difficult to obt conventional creep testing. Some examples are investigation of the behavior of individual zones in a weld structure and understanding the grain boundaries in determining the rate controlling mechanism of p metals.

Another advantage is that you do not have any tertiary creep stage and so the deformation is more stable and hence this is more suited for investigating brittle materials and finally the test is capable of yielding information in some special investigations which is not possible or extremely difficult to obtain in conventional creep testing, so special investigation such as behaviour of weld zones or understanding the role of grain boundaries because you can selectively carry out a creep test in a single grain close to a grain boundary and understand the performance or the creep performance of this grain boundary and its contribution to the overall creep behaviour of the material, so these are the advantages of the impression creep technique.

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Limitations of the impression creep technique • Friction between the punch and the specimen at $+ \sqrt{ }$ higher depths of penetration could introduce errors Cupstrate during analysis of data

• The specimen thickness must be at least 3 times the punch diameter. The substrate on which the specimen is resting could resist the evolution of the plastic zone under the punch if the thickness of the specimen is small

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Creep rates
D = punch diameter
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However there are a few limitations as well, so one of the key limitation is that when the punches penetrating the sample there is going to be some amount of friction between the punch and the sample and this frictional force tends to grow at higher depth of penetration and this frictional stress can lead to errors during analysis of data. Similarly you cannot choose all kinds of specimens for your test, so if your specimen is very thin with respect to punch diameter then you can have contribution of the substrates, so if you have placed your sample on steel block as an example, so this is your substrate and this is your sample let us say zinc and you are carrying out this impression creep.

You can have resistance coming from the substrate also, That resistance can lead to lowering of creep rates, so hence in order to avoid such kind of interference from the substrate your specimen thickness should be at least 3 times the punch diameter, so if you are diameter of the punch is D then for a good impression creep test your sample thickness t must be greater, sample thickness t must be at least greater than 3 times the diameter of your punch.

In that case you will not have any contributions from your substrate and you can be more sure about the data that is getting generated. So that is because when you have impression creep happening you have a plastic zone that gets plastic zone that gets developed under your punch and like we said this plastic zone typically has a diameter D similar to the diameter of the punch and if your sample thickness is such that the plastic zone starts interacting with your substrate then the substrate will resist the plastic zone and if it raises the plastic zone then your creep rates will come down and the actual performance of the material will not be captured.

So that was about the impression creep technique its advantages as well as its limitations and in this particular lecture we have covered creep test in general, what other precautions or to be taken while carrying out a good creep test and what is the difference between a tension and compression creep test when you apply it for ceramic and how flexural test are very good for ceramics and then we ended it with impression creep testing technique.