Friction and Wear of Materials: Principles and Case Studies
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Lecture - 33
Sliding Wear of Silicon Carbide in Cryogenic Environment

So welcome back to this NPTEL lecture on friction and wear of materials. In last few lectures, I have discussed some of the case studies to explain the nature of friction and wear properties of metals and ceramics in cryogenic environment and cryogenic means more particularly liquid nitrogen environment and under high speed sliding conditions.

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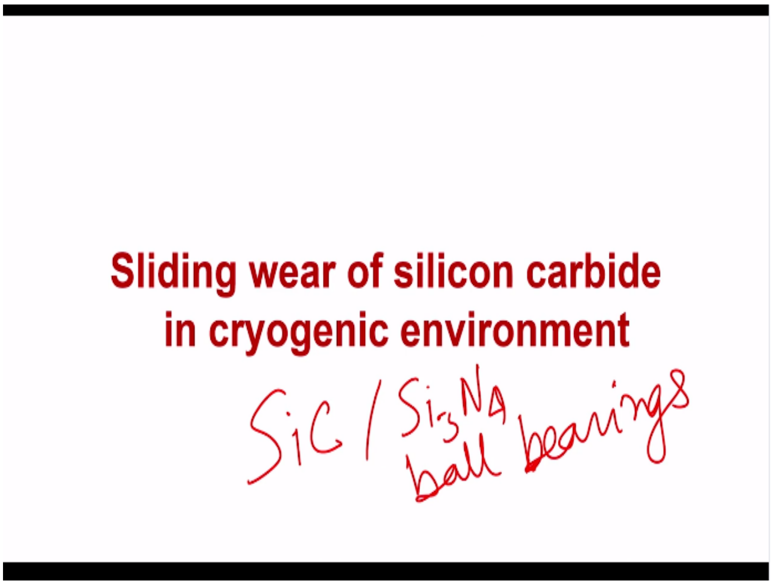

Sliding wear of silicon carbide in cryogenic environment

SiC / Si_3N_4 ball bearings

This is the last lecture on the cryogenic wear of materials and in this lecture I will describe one more case study again from our own research and that is on silicon carbide ceramics. Now silicon carbide and silicon nitride these are the two non-oxide ceramics which has received wider attention particularly for high temperature applications and silicon carbide and silicon nitride these are also used as ball bearings.

Because you can shape them to a balls of different sizes, so it was interesting for us to understand and investigate the sliding wear of silicon carbide in cryogenic environment with an objective to assess their suitability for ball bearings in Space Shuttle Main Engines.

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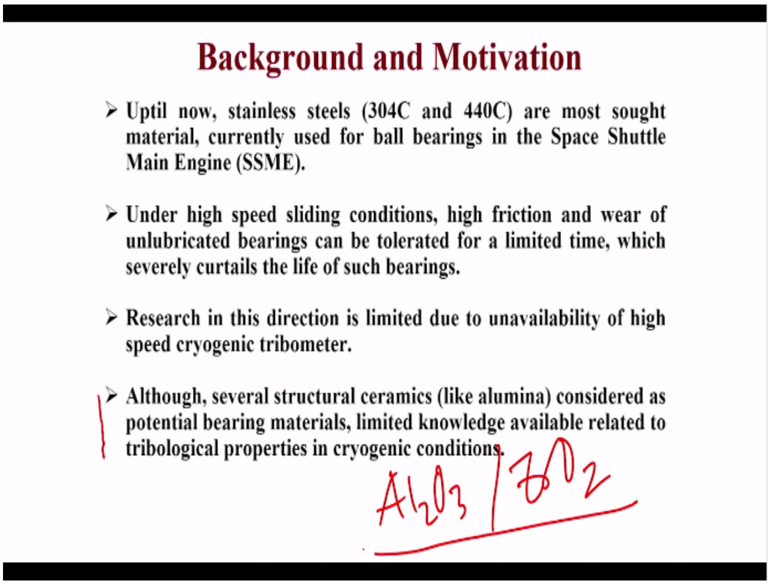

Background and Motivation

- ➢ Uptil now, stainless steels (304C and 440C) are most sought material, currently used for ball bearings in the Space Shuttle Main Engine (SSME).
- ➢ Under high speed sliding conditions, high friction and wear of unlubricated bearings can be tolerated for a limited time, which severely curtails the life of such bearings.
- ➢ Research in this direction is limited due to unavailability of high speed cryogenic tribometer.
- ➢ Although, several structural ceramics (like alumina) considered as potential bearing materials, limited knowledge available related to tribological properties in cryogenic conditions.

Al_2O_3 / ZrO_2

So this was the background, so as I have categorically mentioned in one of the earlier lectures that 440C martensitic stainless steels or 304C stainless steels, they are mostly sought material currently used for ball bearings in Space Shuttle Main Engine and under high speed sliding conditions, high friction and wear of unlubricated bearings can be tolerated but it severely curtails the overall life of the bearings.

And then in some of the earlier lectures, I have mentioned that how the cryogenic wear properties of these two oxide ceramics, one is that alumina and another one is zirconia, how they are different from their room temperature sliding conditions.

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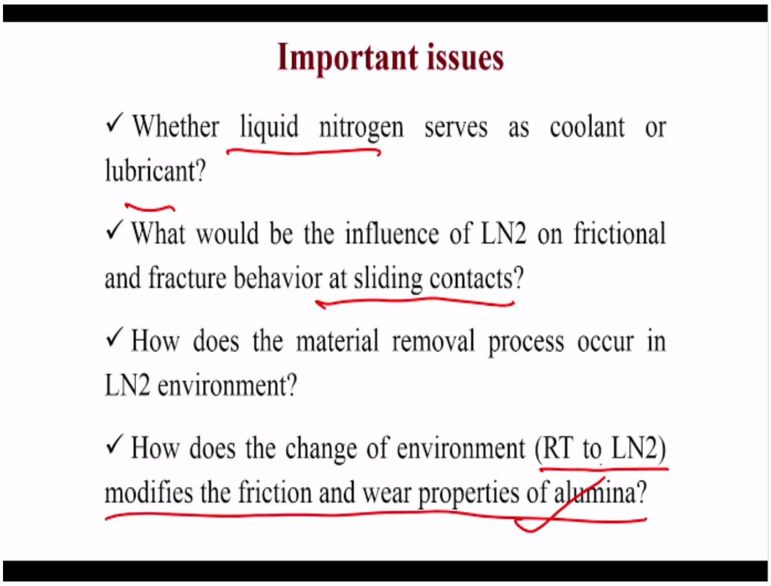

Important issues

- ✓ Whether liquid nitrogen serves as coolant or lubricant?
- ✓ What would be the influence of LN2 on frictional and fracture behavior at sliding contacts?
- ✓ How does the material removal process occur in LN2 environment?
- ✓ How does the change of environment (RT to LN2) modifies the friction and wear properties of alumina?

So here we would like to again address the similar issues that I have posed while discussing the earlier case studies and those are whether liquid nitrogen serves as coolant or lubricant and what would be the influence of liquid nitrogen on frictional and fracture behaviour at sliding contacts and how does the material removal process occur in liquid nitrogen environment and how does the change of environment room temperature to liquid nitrogen modifies the friction and wear properties of alumina.

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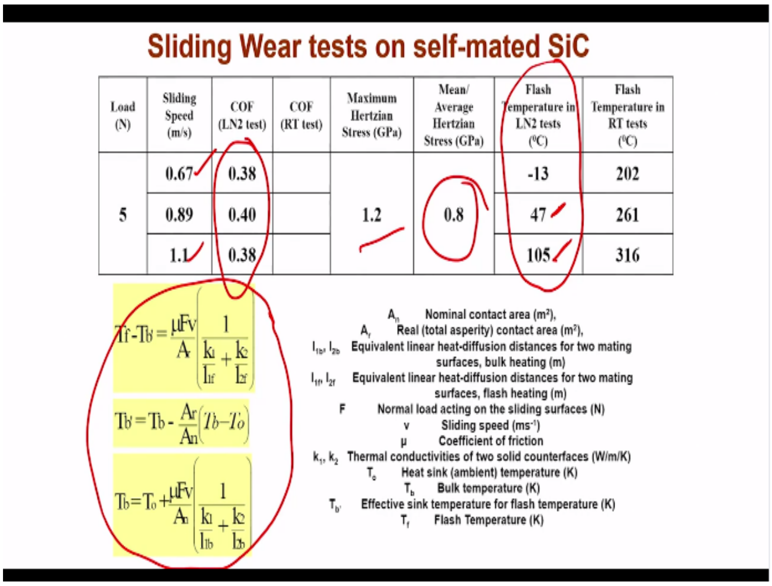

Sliding Wear tests on self-mated SiC

Load (N)	Sliding Speed (m/s)	COF (LN2 test)	COF (RT test)	Maximum Hertzian Stress (GPa)	Mean/ Average Hertzian Stress (GPa)	Flash Temperature in LN2 tests (°C)	Flash Temperature in RT tests (°C)
5	0.67	0.38		1.2	0.8	-13	202
	0.89	0.40				47	261
	1.1	0.38				105	316

$$T_f - T_{b'} = \frac{\mu F v}{A_r}\left(\frac{1}{\frac{k_1}{l_{1f}} + \frac{k_2}{l_{2f}}}\right)$$

$$T_{b'} = T_b - \frac{A_r}{A_n}\left(T_b - T_o\right)$$

$$T_b = T_o + \frac{\mu F v}{A_n}\left(\frac{1}{\frac{k_1}{l_{1b}} + \frac{k_2}{l_{2b}}}\right)$$

A_n	Nominal contact area (m^2),
A_r	Real (total asperity) contact area (m^2),
l_{1b}, l_{2b}	Equivalent linear heat-diffusion distances for two mating surfaces, bulk heating (m)
l_{1f}, l_{2f}	Equivalent linear heat-diffusion distances for two mating surfaces, flash heating (m)
F	Normal load acting on the sliding surfaces (N)
v	Sliding speed (ms^{-1})
μ	Coefficient of friction
k_1, k_2	Thermal conductivities of two solid counterfaces (W/m/K)
T_o	Heat sink (ambient) temperature (K)
T_b	Bulk temperature (K)
$T_{b'}$	Effective sink temperature for flash temperature (K)
T_f	Flash Temperature (K)

Similar to earlier case studies, here we have varied sliding speed from 0.67 meter per second to 1.1 meter per second and if we keep the load at 5 Newton we have found that coefficient of friction is around 0.38 so around close to 0.4 which is strictly relatively higher compared to some of the metals and Hertzian contact stress is 1.2 gigapascal, mean average Hertzian contact stress was 0.8 gigapascal and flash temperature is at 0.67 is subzero temperature at 0.89 is 47 degree Celsius and 1.1 meter per second it is more than 100 degree Celsius.

And flash temperature was calculated according to the equations that I have described at greater details in one of the initial lectures in this NPTEL lecture series.

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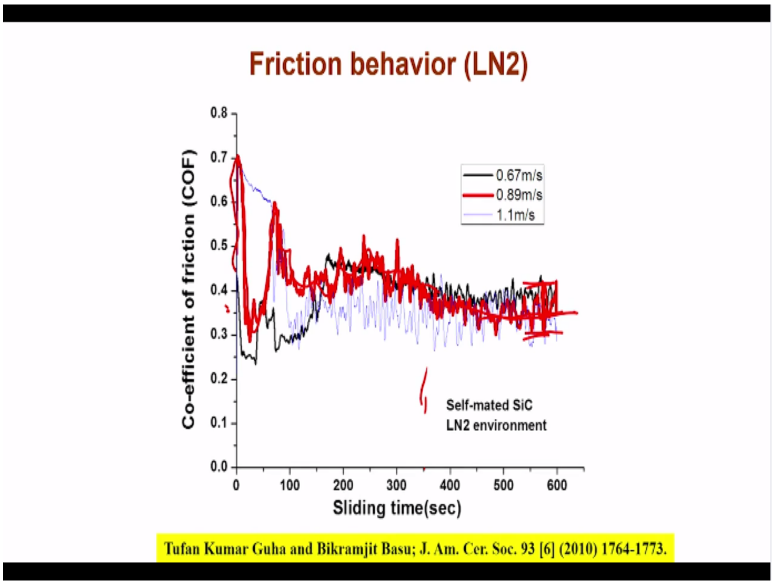

Friction behavior (LN2)

Tufan Kumar Guha and Bikramjit Basu; J. Am. Cer. Soc. 93 [6] (2010) 1764-1773.

And flash temperature also we calculate in the room temperature environment, now this is how the coefficient of friction evolves with time for the tests that we have conducted up to 10 minutes that is up to 600 seconds. So what you see here that this coefficient of friction goes of very high value and then it goes through some kind of unstable region and then finally it reaches the steady-state after the 5 minutes.

And then it gets very much flattened and if you compare the steady-state coefficient of friction, we do not see much change because it varies over the window of 0.3 to 0.4 but there is lot of undulations there if you loop through this very closely that frictional behaviour of self-mated silicon carbide.

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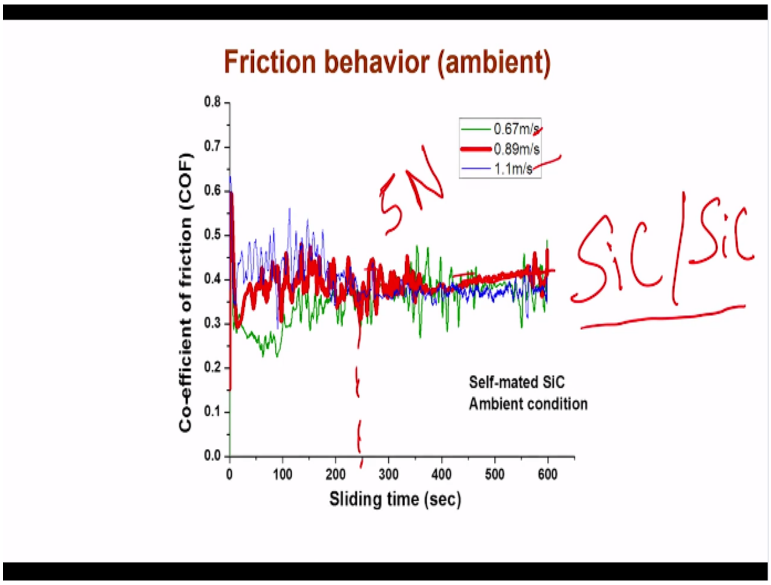

Friction behavior (ambient)

How about frictional behaviour of self-mated silicon carbide that means silicon carbide versus silicon carbide so here the flat material is silicon carbide and ball material also silicon carbide and under the similar sliding conditions with variation in speed from 0.67 to 1.1 meter per second and the load is around 5 Newton we see not much difference in terms of the steady-state coefficient of friction.

Only thing is that in room temperature conditions that this couple reaches the steady-state to match earlier times scale of up to 3 to 4 minutes but whereas in liquid nitrogen we see lot more unstable region in the frictional behaviour. So if you go back to this particular table where these things are kind of fill in the blanks and you can now fill in the blanks because there is not much difference in terms of the steady-state coefficient of friction between room temperature and liquid nitrogen tests.

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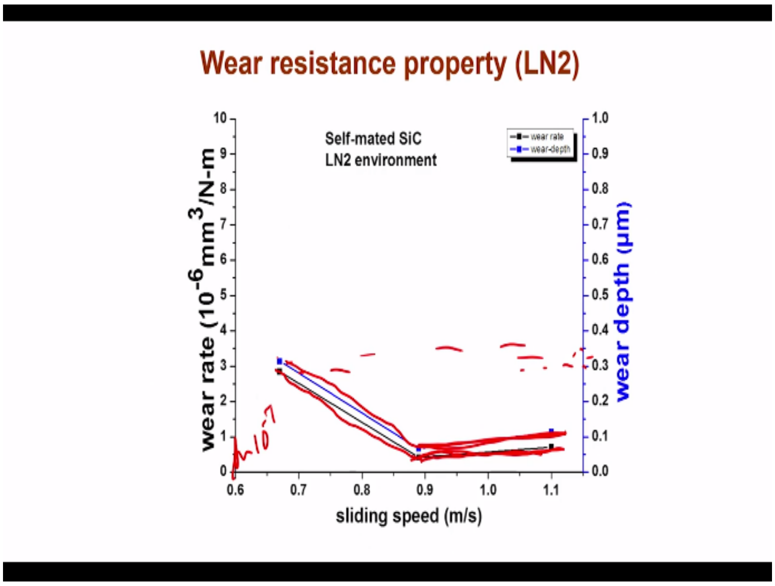

Wear resistance property (LN2)

Now as far as the quantitative data on the wear resistance is concerned, this is the results we have obtained in liquid nitrogen environment based on the laser surface profilometric analysis of the sliding track what we got that wear depth is fairly small so wear depth is <0.4 micron okay, this is the wear depth and wear depth decreases as you increase the speed and it reaches almost like a steady-state value of around 0.1 micron.

As far as the wear rate is concerned, it is 10 to the power -6 millimeter cube Newton meter and again it changes from 3 10 to the power -6 to <1 10 to the power -6 millimeter cube per Newton meter for self-mated silicon carbide at 5 Newton load. So less than 0.4 micron depth at such a high speed sliding conditions and under high contact stress is really a good number.

So what I point out here that it is 3*10 to the power -6 goes to <10 to the power - <1*10 to the power -6 means that once you go down the scale here so here you can approximate it as millimeter cube per Newton meter. So essentially what I am trying to point out here that it is possible to decrease the wear rate by one order of magnitude.

Or it is possible that self-mated silicon carbide can experience one order of magnitude less wear rate at higher sliding speed of 1.1 meter per second compared to that what the self-mated silicon carbide experiences at 0.67 meter per second, so all in all this wear rate and wear depth both the data if we consider together it really leads to the conclusion that self-mated silicon carbide experiences very high wear resistance or less wear rate in liquid nitrogen environment.

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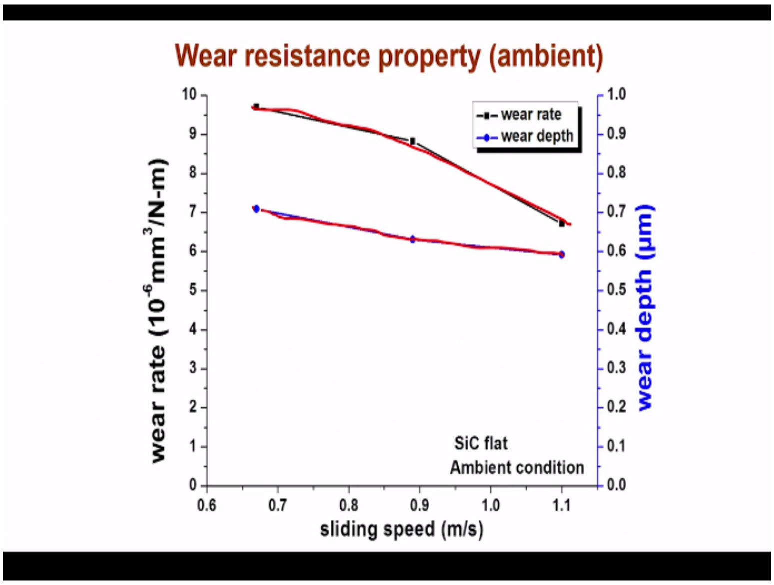

Wear resistance property (ambient)

Now if you look at that similar data of wear rate and wear depth, after self-mated silicon carbide at slided at ambient conditions that is room temperature and this is that wear rate that was measured on the silicon carbide flat and what you see again that this wear rate decreases and almost like linear fashion as you increase the sliding speed from 0.67 to 1.1 meter per second the same is true for the wear depth.

But both the numbers here if you compare to the liquid nitrogen conditions sliding conditions because both are y-scale at the same if you notice here and if you compare now the y-scale values then you are convinced that room temperature sliding conditions leads to more wear of the self-mated silicon carbide under these particular sliding conditions (()) (10:23) this tribometer.

So there is a very clear signature that wear resistance on a whole or as a combination of wear rate and wear depth is much reduced in liquid nitrogen sliding conditions compared to that on that ambient condition.

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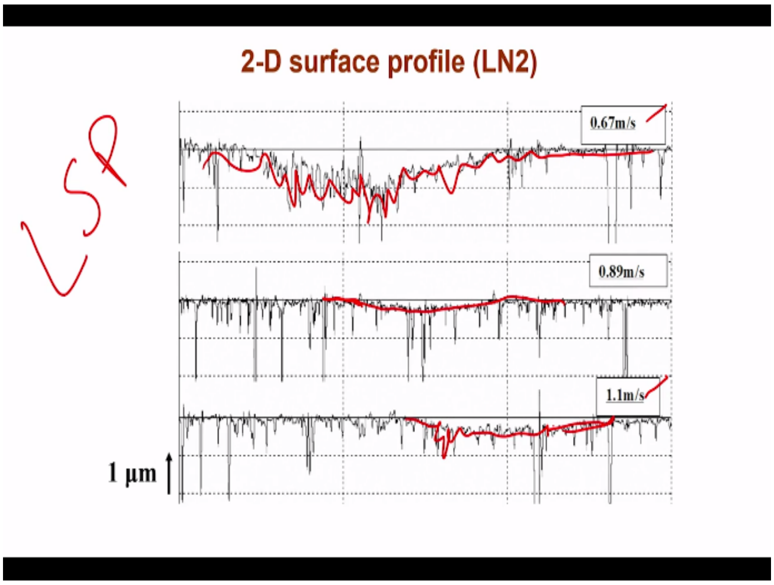
2-D surface profile (LN2)

Now this is the typical 2-D surface profiles what we have measured in laser surface profilometer, LSP stands for laser surface profilometer so you have the sliding speed like 0.67 meter per second to 1.1 meter per second and you can see very clear sharp peaks and valleys in the 2-D surface profile. What it means is that these are the signatures of the differential wear that means this wear profile or 2-D surface profile is not very smooth.

And it shows multiple surface groups of different heights and also different shape so essentially these are the signatures of the differential wear behaviour.

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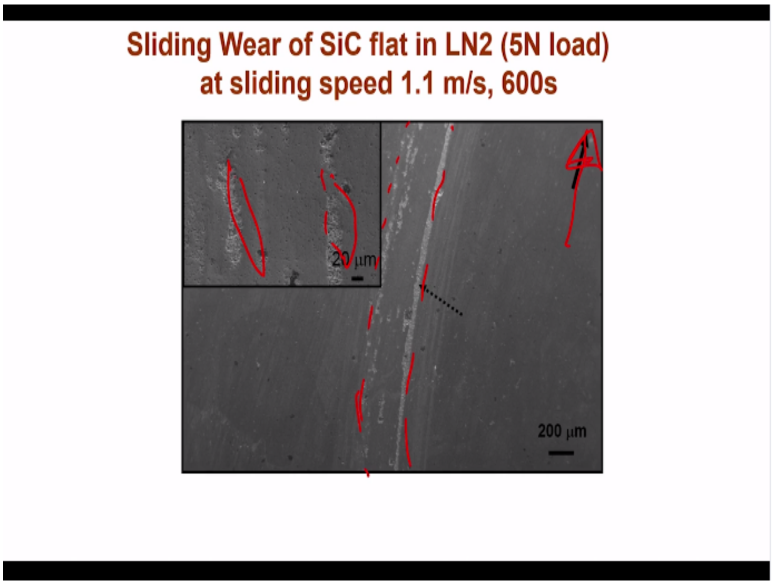

Sliding Wear of SiC flat in LN2 (5N load) at sliding speed 1.1 m/s, 600s

Now if you look at this scanning electron microscopy images so this is your sliding track and this is your sliding directions and if you look at little bit closely, if you look at this particular region, so there is some contrast difference showing that perhaps some of the silicon carbide can be oxidized to silica.

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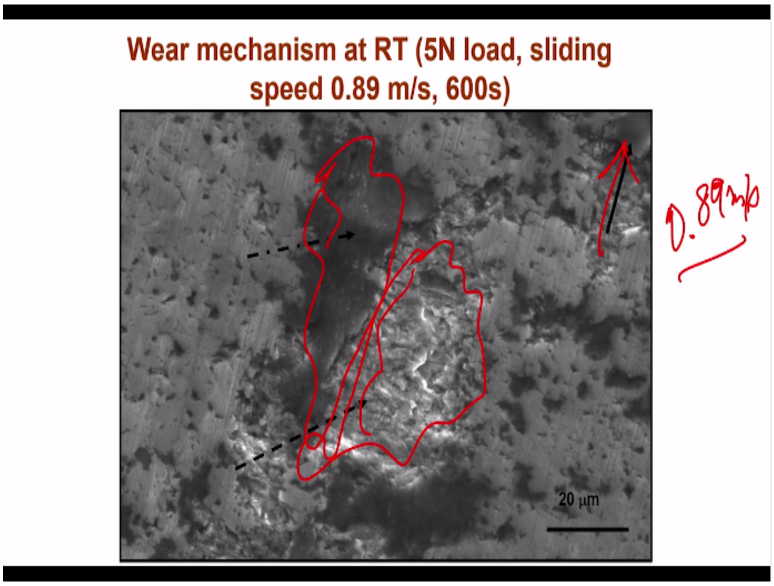

Wear mechanism at RT (5N load, sliding speed 0.89 m/s, 600s)

Let us look at much more details into what is happening at intermediate sliding speed of 0.89 meter per second. After the sliding is over for 10 minutes okay, so this is your sliding directions and if you look at this particular region or this particular region, here you can clearly see this back scattered electron image it appears in different contrast compared to this particular region of the sliding surface.

So that means the tribochemistry plays a role and also there is tribochemical reactions that is taking place under these sliding conditions are 0.89 meter per second.

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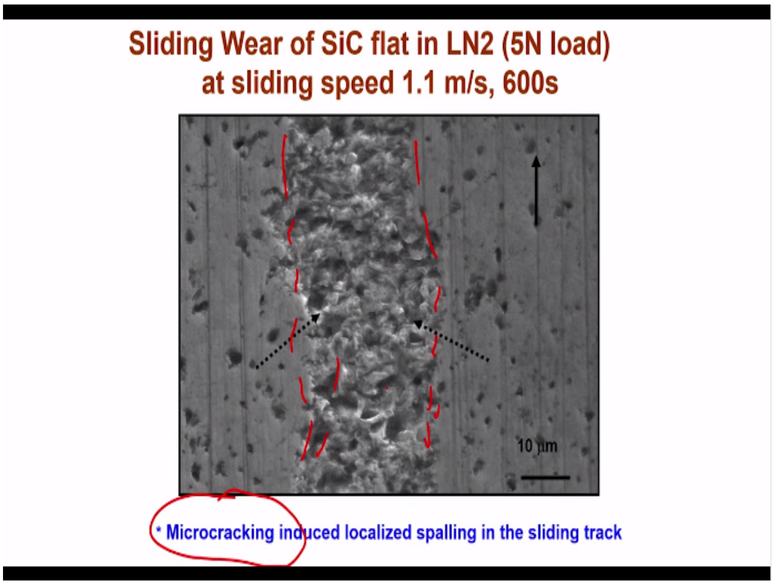

Sliding Wear of SiC flat in LN2 (5N load)
at sliding speed 1.1 m/s, 600s

* Microcracking induced localized spalling in the sliding track

This is at the high sliding speed conditions and you can see this is your wear track and this wear track a closer look at this particular region clearly shows you that there are multiple or there is numerous microcracks so this microcracking is playing an important role in this particular case and that is the high sliding speed of 1.1 meter per second. Unlike this alumina or zirconia in silicon carbide, we have fixed the normal load of 5 Newton.

But ideally using the same tribometer one can do the experiments with variation in the normal load but here we are more interested to understand that if you increase or if you vary the sliding speed over similar window what would be its influence on the wear resistance and wear mechanisms of self-mated silicon carbide rather than low dependent wear behaviour of self-mated silicon carbide.

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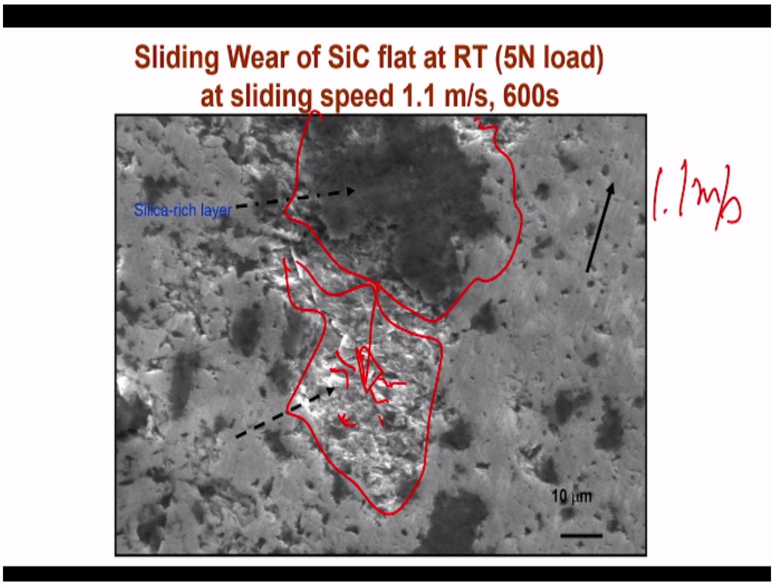

Sliding Wear of SiC flat at RT (5N load) at sliding speed 1.1 m/s, 600s

So you will see that similar behaviour at 1.1 meter per second at some part of the tribolayer and you can see this is the region where it clearly shows that this particular region should have different chemical composition or different chemistry compared to this particular region. So this region certainly is very rough and this roughness of this worn surface can be attributed to cracking and localized poling from the tribolayer.

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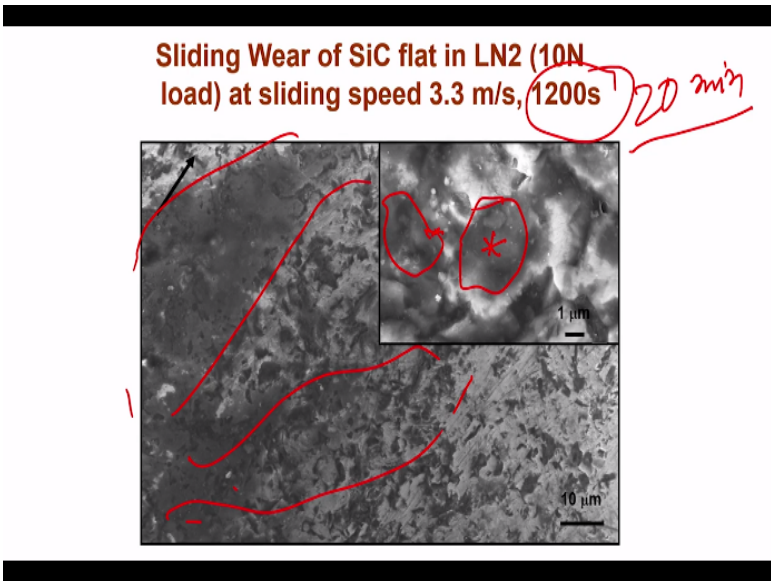

Sliding Wear of SiC flat in LN2 (10N load) at sliding speed 3.3 m/s, 1200s

So this is given that higher sliding speed of 3.3 meter per second at 10 Newton load and here again we have done the experiments up to 1200 seconds so 1200 seconds means up to 20 minutes. So we cannot do one of the things, I think I might have mentioned in the earlier lecture that there are two major difficulties with using this cryogenic sliding wear experiments.

The first one is that that with this tribometer it is extremely important to fix or place the ceramic disc inside the tribometer in a very stable manner. If there is certain difference in the top surface or the leveling of the surface then what would happen, ceramics being brittle materials when it will experience high sliding speed against the ball when ball is stationary and then disc is rotating that is every chance of ceramic disc will be broken into two pieces.

But here we are more interested to know how does a ball behave, it is not about the disc but in the ball but to have the self-mated conditions essentially you are trying to understand that under the self-mated conditions when the ball will be slided against the same material of similar composition what would be the influence on its friction and wear behaviour. So from that point of view, we have conducted all these experiments.

So the first difficulty is that placing the ceramic disc and that is very important. Second one is that we cannot carry out this high speed sliding wear experiments in cryogenic environment for up to longer time period like up to 30 minutes continuously because it causes lot of undulations and because of the high speed sliding conditions and in the Space Shuttle Main Engine also as far as the bearing life is concerned, the 440C martensitic stainless steel bearing ball used to have a life of less than half an hour.

So if we can do these tests up to 20 minutes or so on that is good enough for these particular applications in mind. Now coming back to this particular scanning electron microscopy images, again you see this large region here it is a tribolayer and also here and if you blow up this particular region some of the region you can focus then you can see that there is a very thin tribolayer which is covered on this individual silicon carbide grain okay.

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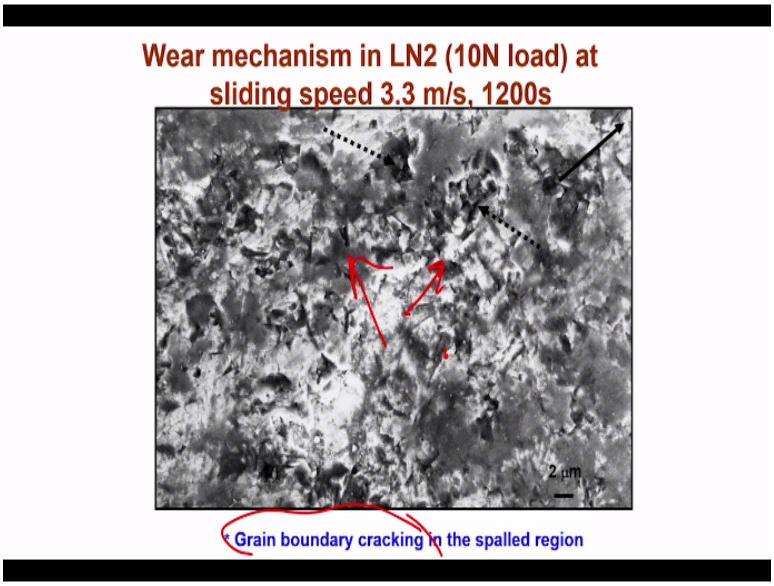

Wear mechanism in LN2 (10N load) at sliding speed 3.3 m/s, 1200s

* Grain boundary cracking in the spalled region

So essentially these are the places where silica rich layer perhaps have formed. So 3.3 meter per second indeed is very high sliding speed and under these conditions the only way that the silicon carbide is worn out it is because of the grain boundary cracking and you can see that at various locations like these locations as well as these locations. There are small microcracks which are formed and presence of these microcracks essentially indicates the occurrence of the intergranular cracking.

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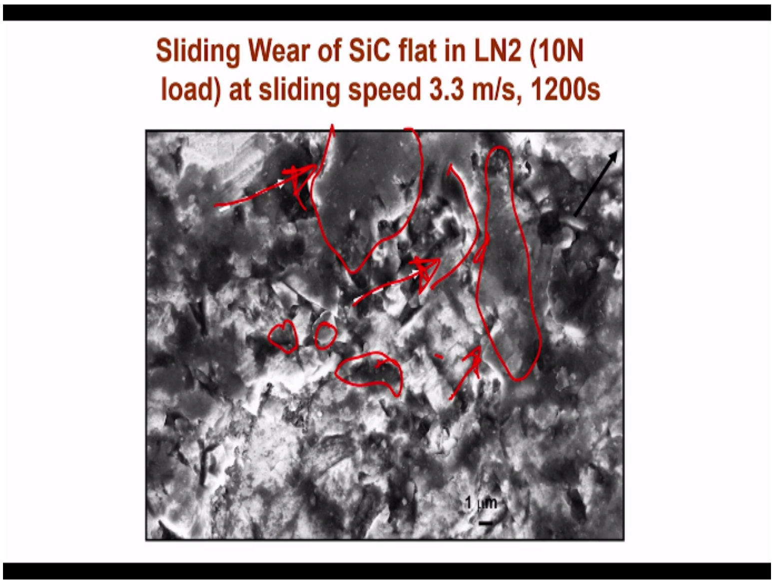

Sliding Wear of SiC flat in LN2 (10N load) at sliding speed 3.3 m/s, 1200s

The same is true for little bit higher magnification SEM images just to illustrate that how these silicon carbide wear is influenced by high speed sliding conditions. So this is that one arrow and this is another arrow as you can see that these are cracking also it shows and also there are small pockets of the pull out region that you can clearly see here on the worn tribological surface.

And also you can see this is the layer of the tribology tribolayer and this is again in the region that where the microcracks takes place. So what you have seen earlier in case of alumina and zirconia and what you see now in case of silicon nitride I am sure that you would agree with me that sliding wear of silicon carbide is governed by more tribomechanical wear and partial tribochemical wear.

If the tribochemical wear would have played a major role, then entire tribolayer would be covered with the tribochemical layer and that is certainly not the case. Second thing is that despite the dominance of the tribomechanical wear what you see the tribosurface is relatively smooth not extremely rough what we have seen earlier in case of alumina particularly in case of the alumina which is again a model brittle ceramic.

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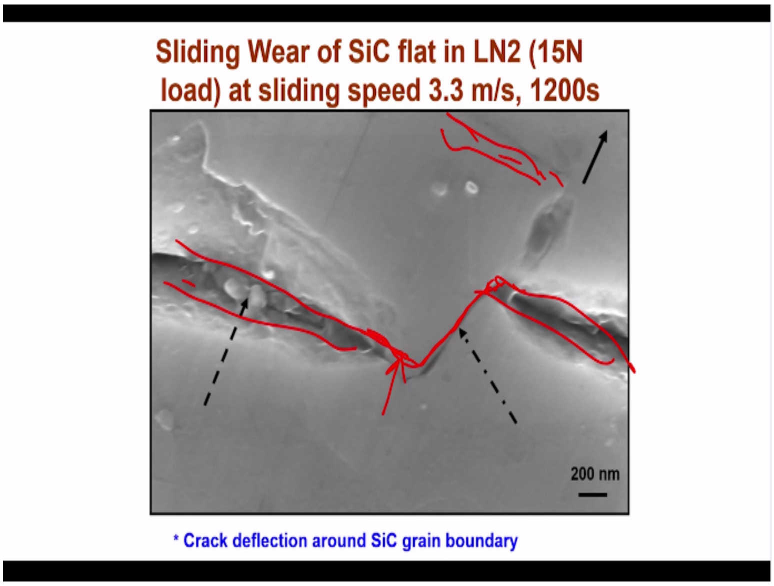

Sliding Wear of SiC flat in LN2 (15N load) at sliding speed 3.3 m/s, 1200s

* Crack deflection around SiC grain boundary

Now if you look at the wear at the grain level or the grain scale, now if you see this is the two silicon carbide grains which is tabular in nature and you will see that sorry this region is again another region where you can see cracks so there is microcracks, grain boundary cracks and these cracks sometimes goes deep into the material. So this cracking is taking place but the cracks are getting deflected around the grain boundary region.

And if you look at the crack deflection along the grain boundary region so essentially that crack deflection leads to delayed wear in case of silicon carbide. So if the grain boundaries are the parts for the crack propagation or the crack deflection certainly it will help in increasing the wear resistance of these materials. So this is what we have seen at 3.3 meter per second for 20 minutes of the sliding speed.

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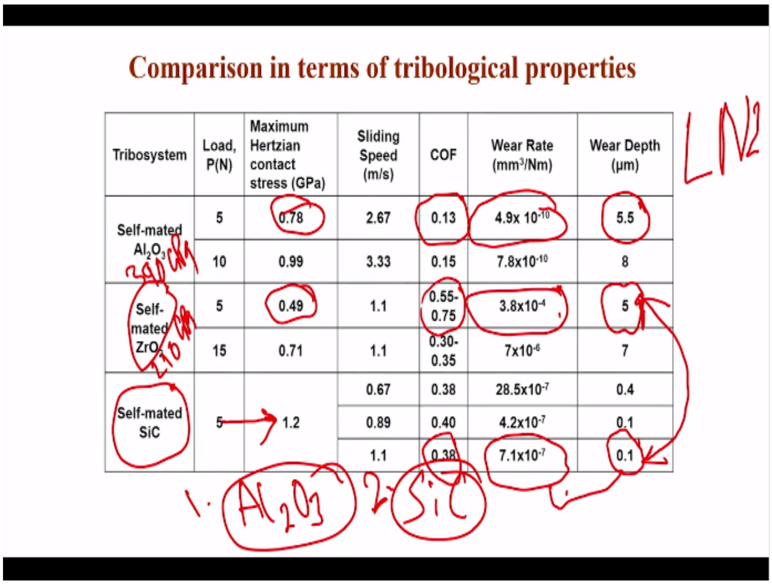

Comparison in terms of tribological properties

Tribosystem	Load, P(N)	Maximum Hertzian contact stress (GPa)	Sliding Speed (m/s)	COF	Wear Rate (mm³/Nm)	Wear Depth (μm)
Self-mated Al_2O_3	5	0.78	2.67	0.13	$4.9x\ 10^{-10}$	5.5
	10	0.99	3.33	0.15	$7.8x10^{-10}$	8
Self-mated ZrO_2	5	0.49	1.1	0.55-0.75	$3.8x10^{-4}$	5
	15	0.71	1.1	0.30-0.35	$7x10^{-6}$	7
Self-mated SiC	5	1.2	0.67	0.38	$28.5x10^{-7}$	0.4
			0.89	0.40	$4.2x10^{-7}$	0.1
			1.1	0.38	$7.1x10^{-7}$	0.1

Now if one look at the if we compare this is fairly an important slide so let us spend some time in comparing the numbers which are placed in this slide. So in last two lectures, we have seen that self-mated alumina, self-mated zirconia in this lecture I have described wear behaviour of self-mated silicon carbide. Now if you want to be extremely fair in making this comparison, it is important to fix the load.

Because the load is varied in this 3 studies but one load is very common for alumina, zirconia and silicon carbide that is 5 Newton load and 5 Newton load depending on what is the kind of elastic modulus of the materials that leads to difference in the Hertzian contact stress. For example, oxide ceramics their elastic modulus is relatively lower compared to silicon carbide. So therefore in the oxide ceramics here like alumina is 390, so therefore gigapascal is the elastic modulus.

So this is the elastic modulus value I am talking about, zirconia is 210 gigapascal and silicon carbide is little higher than alumina also. So 5 Newton load it leads to 1.2 gigapascal as a maximum Hertzian contact stress which is 0.78 in case of alumina and which is 0.49 in case of zirconia. Now typically the wear rate in case of alumina is around 10 to power -10, in case of zirconia which is 10 to the -4, 10 to the -4 and 10 to the -10.

And in case of silicon carbide is 10 to the -7 so if you look at that wear rate, alumina is certainly highly wear resistance with 10 to the -10 followed by silicon carbide 10 to the -7 and zirconia is least wear resistant at 10 to the -4. Zirconia also experiences high coefficient of friction about 0.5 to 0.75 whereas silicon carbide coefficient of friction is around 0.38, alumina it is the lowest.

So looks like alumina is the better wear resistance materials as per as the cryogenic sliding conditions, remember all these data that we summarized in this table are obtained under liquid nitrogen sliding conditions not in the room temperature. However, only one point that I must note here that wear depth wise alumina has larger wear depth 5.5 whereas silicon carbide is 0.1 extremely small and alumina and zirconia has relatively similar like 5 to 5.5 micron.

So 5 micron versus 0.1 micron it is order of magnitude difference right, so I just what I wanted to compare this two values, 5 versus 0.1 it is order of magnitude difference in the wear depth, so therefore what I believe that if you compare these two like say 10 to the power -7 and 0.1 micron wear depth looks like silicon carbide is not a bad choice either so therefore what I believe that although alumina is a good choice but silicon carbide is also equally the second choice.

So number one choice is alumina, number two choice is silicon carbide and we have also established a close correlation between the wear mechanism and the wear rate and where we have found in self-mated silicon carbide it is a limited tribochemical reactions and more of tribomechanical wear with grain boundary cracking, localized poling as a major wear mechanism.

Zirconia it is the transformation in this cracking and if you remember correctly in case of zirconia I have shown that tetragonal zirconia undergoes phase transformation to orthorhombic zirconia during the cryogenic sliding conditions and the reason that I have provided while explaining this intriguing observations was that tetragonal zirconia to monoclinic zirconia, this particular transformation that one is the intermediate product that is the orthorhombic zirconia.

Since we are doing these experiments for relatively shorter time 10 minutes or so, we are giving the system we are not giving enough time to the system to come back to the equilibrium transformation phase that is the monoclinic zirconia and therefore orthorhombic zirconia forms and in case of the alumina what happens it is that thermal conductivity effect is also very important.

And we have seen the occurrence of both intergranular and transgranular fracture, cleavage steps and so on and that leads to wear of self-mated alumina as well.

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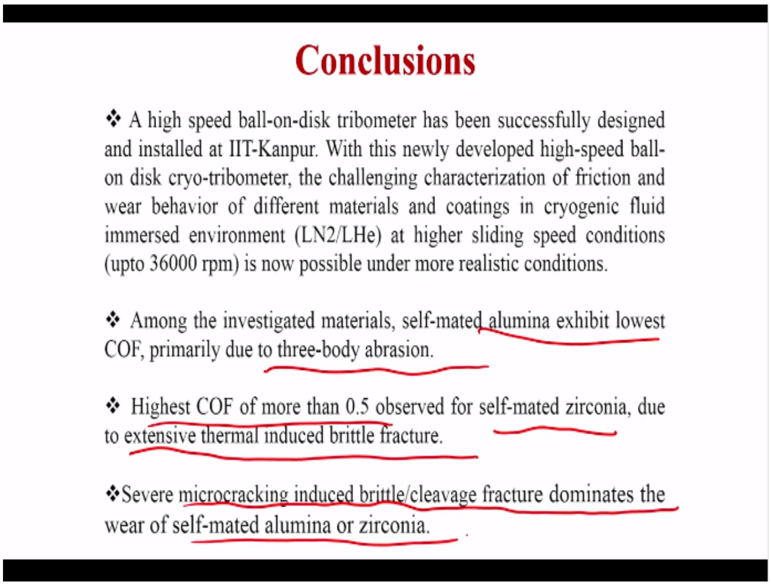

Conclusions

- ❖ A high speed ball-on-disk tribometer has been successfully designed and installed at IIT-Kanpur. With this newly developed high-speed ball-on disk cryo-tribometer, the challenging characterization of friction and wear behavior of different materials and coatings in cryogenic fluid immersed environment (LN2/LHe) at higher sliding speed conditions (upto 36000 rpm) is now possible under more realistic conditions.
- ❖ Among the investigated materials, self-mated alumina exhibit lowest COF, primarily due to three-body abrasion.
- ❖ Highest COF of more than 0.5 observed for self-mated zirconia, due to extensive thermal induced brittle fracture.
- ❖ Severe microcracking induced brittle/cleavage fracture dominates the wear of self-mated alumina or zirconia.

So these are the conclusions, this is that high speed ball-on-disk tribometer has been used and this has been designed, fabricated and this performance has been tested to study the sliding wear of self-mated alumina, self-mated zirconia and self-mated silicon carbide and self-mated alumina exhibits the lowest COF primarily due to three-body abrasion, highest COF of 0.5 is observed for self-mated zirconia.

Again it is extensive thermal induced fracture, severe microcracking and cleavage fracture dominates the wear of self-mated alumina or zirconia.

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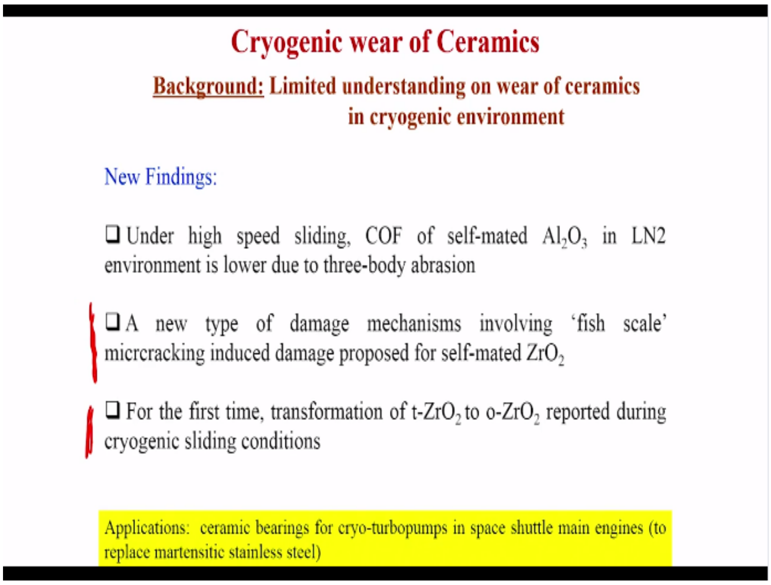

Cryogenic wear of Ceramics

Background: Limited understanding on wear of ceramics in cryogenic environment

New Findings:

❑ Under high speed sliding, COF of self-mated Al_2O_3 in LN2 environment is lower due to three-body abrasion

❑ A new type of damage mechanisms involving 'fish scale' micrcracking induced damage proposed for self-mated ZrO_2

❑ For the first time, transformation of t-ZrO_2 to o-ZrO_2 reported during cryogenic sliding conditions

Applications: ceramic bearings for cryo-turbopumps in space shuttle main engines (to replace martensitic stainless steel)

And this is the kind of key summary on this cryogenic wear of ceramics and then what we call is that new findings like under high speed sliding conditions, coefficient of friction of self-mated alumina in liquid nitrogen is lower due to three-body abrasion and for the first time we have reported I think it was in American Ceramic Society Journal, the tetragonal zirconia to orthorhombic zirconia phase transformation can take place under cryogenic sliding conditions.

And if you remember correctly that one of the intriguing observations was the physical microcracking pattern which is observed for self-mated zirconia.

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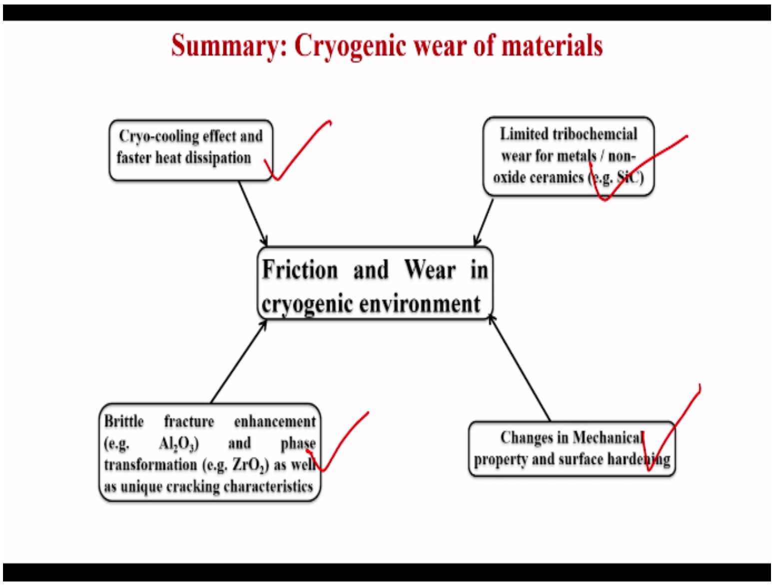

Summary: Cryogenic wear of materials

Friction and Wear in cryogenic environment

Cryo-cooling effect and faster heat dissipation

Limited tribochemcial wear for metals / non-oxide ceramics (e.g. SiC)

Brittle fracture enhancement (e.g. Al_2O_3) and phase transformation (e.g. ZrO_2) as well as unique cracking characteristics

Changes in Mechanical property and surface hardening

So this is the summary of the cryogenic wear of materials in so what we have observed that the differences in the wear mechanism can be attributed to a host of factors and those include the cryo-cooling effect and faster heat dissipation, second one is the brittle fracture enhancement and phase transformation and unique cracking characteristics, third one is changes in the mechanical property and surface hardening.

And fourth one in case of silicon carbide, it is a limited tribochemical wear for metals or non-oxide ceramics that is silicon carbide okay. So with this I close this all these lectures on the cryogenic sliding wear of materials and then I will now start with this wear of the high temperature ceramics. Thank you.