

**Friction and Wear of Materials: Principles and Case Studies**  
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**Lecture – 32**

**Sliding Wear of Alumina Ceramics and Zirconia Ceramics in Cryogenic Environment**

So welcome back in this particular lecture, I will discuss that the sliding wear of alumina that is one of the brittle ceramic. In the last lecture I have shown you some of the very fascinating results that how the sliding wear of the metals. They vary with the sliding environment for example when you do the same sliding test identical sliding test in room temperature and when you do similar identical test at a liquidate the environment.

How does that sliding wear through coefficient of friction and sliding wear resistance of sliding wear behaviour changes? Now in next 30 minutes or so what I will go? I will walk through the specific research results that we have obtained a few years ago when we have conducted the sliding wear tests on a self-mated alumina

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**Fundamental Questions to be addressed**

✓ Whether LN<sub>2</sub> serves as a lubricant or coolant in reducing COF of the selected tribosystems?

✓ How does the material removal process occur in LN<sub>2</sub> environment?

32 ✓ How material property (both thermal and mechanical) does affects the frictional and wear behavior at cryogenic temperatures?

So fundamental questions that are to be addressed is that further so we are talking about sliding wear alumina so fundamental questions that have to be addressed is that well the liquid nitrogen can solve as a lubricant our coolant in reducing coefficient of friction. Second one how does it material removal process occur.

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## Self-mated Alumina

- Genesis of fracture of brittle materials in cryogenic environment?

- How does the fracture/deformation during cryo-sliding is different from pure mechanical fracture?

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So in case of self-mated alumina the particular the question of a particular interest is that what is the genesis of fracture because alumina is known as a model ceramic. So essentially the alumina is expected to undergo brittle fracture in these cryogenic sliding conditions. But the question is that how these cryogenic wear of alumina takes place in case in liquid nitrogen environment and how does the fracture deformation takes place during cryo-sliding and how is it different from pure mechanical fracture.

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## Experimental details

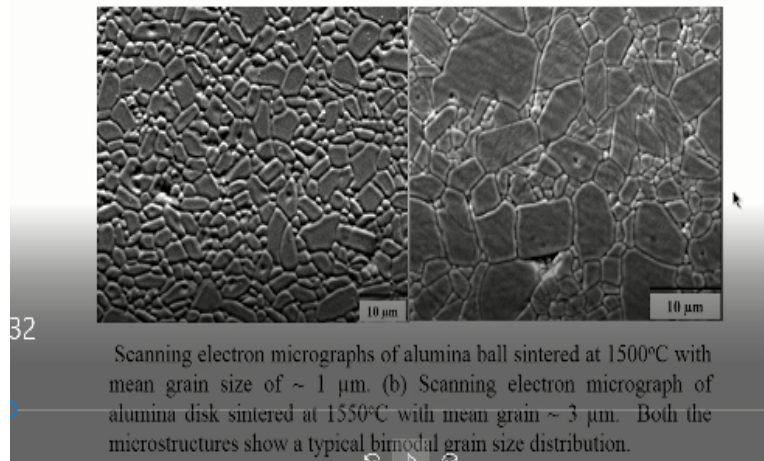
- Testing conditions:
  - Load: 2-15N,
  - Sliding speed: 2-7.46 m/s,
  - Time: upto 600s,
  - Max. Hertzian stress: 0.57-1.1 GPa

So what is a sliding contest sliding test conditions that we have used. Load is around 2-15 Newton sliding speed is somewhere around 2 to 15 Newton sliding speed is somewhere around 2

to 7.5 meter per second time is up to 600 second and maximum Hertzian stress is somewhere around 0.57-1.1 giga pascal.

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## Initial Microstructure (sintered alumina)

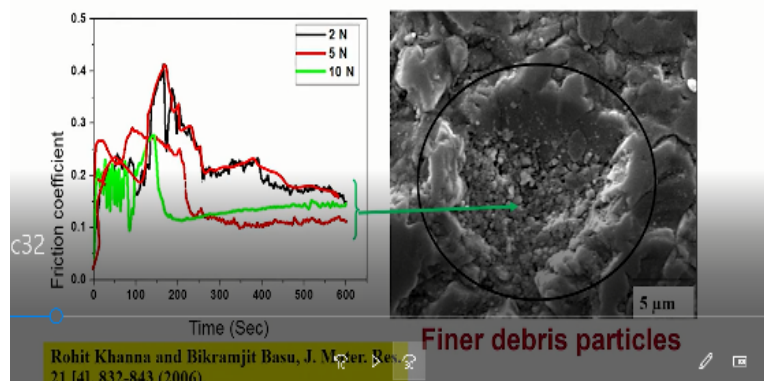


So this is the sintered molecular fracture of alumina alpha alumina and this is a scanning electron micrograph microscope a scanning micrograph so one is the flat and one is a ball so this is essentially is the ball and this is essentially is the flat micro structure. So you can see the this is the last tabular grains and these are fairly coarser grains right but when you see there are also bunch of smaller grains or finer grains are also there. So typical grain size is hammered on the 1 to 3 micron.

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## “Frictional Behavior of Alumina @ Cryogenic Environment”

Load: 2-10N, Sliding speed: 2-3.3 m/s, 600s,  
Max. Hertzian stress: 0.57-0.99 GPa. LN2



So we have done these experiments at liquid nitrogen temperature and this is the load that we are used 2-10 newton. Now as one of the fictional behaviour is concerned this particular self-mated alumina so its quite an interesting object was so it was quite an interesting characteristic for example at 2 newton load at low load this friction goes through a transition okay at 5 newton load you can see that the red one this red one.

It is also somewhere in the middle and the green one is 10 newton load so all it shows some it goes to very high value up to 0.3 0.4 in the first 3 minutes are up to 2 hours within first 3 minutes and in the next 7 minutes the friction drops and friction and co efficient goes to around 0.15 or somewhere in the window of 0.1 to 0.2. So this is the window of 0.1 to 2 point 0.1 to 0.2 that is how the COF changes.

And if you look at this wear surfaces and if you then you can see that an extremely fine debris particles and these finer debris particles essentially add that the alumina where debris particles so what happens during sliding wear at high speed conditions this alumina grains they are crossed into finer debris particles and as a result this debris particles they are essentially lead to 3 body wear situation.

So this is one of the things that is fairly important for you to recognize that in case of self-mated ceramic it is the 3 body wear that can potentially take place and that can lower the coefficient of friction during as the sliding progresses.

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### What causes reduction in friction Coefficient?

❑ Under high sliding speed, contact temperature becomes too high, leading to evaporation of liquid nitrogen, giving rise to bubble formation as observed during experiments.

❑ Phonon-phonon interaction at cryogenic temperature:

Thermal conductivity,  $K \sim 1/T$ .  $K_{\text{alumina, 77 K}} = 10 K_{\text{alumina, 298 K}}$  [1,2].

❑ Role of wear debris (dual effect)

Therefore, high thermal conductivity of alumina at LN2 temperature and continuous flushing by liquid nitrogen leads to more intense heat dissipation from the contact zone, which leads to reduction in friction coefficient.

[1] W. D. Kingery, H. K. Bowen and D. R. Uhlmann, Introduction to ceramics ( 2nd ed.), A Wiley-Interscience publication.

[2] Wigley, D.A., Materials of construction and techniques of fabrication, Ch. 6, 322

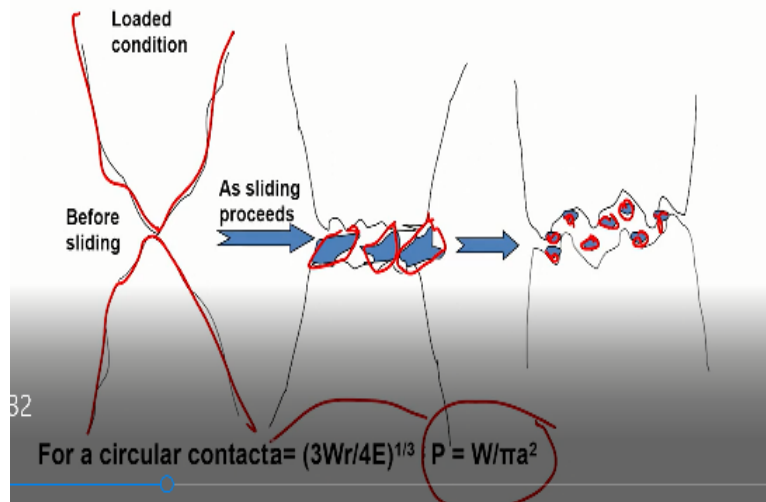
Now the question is what causes the reduction in the friction coefficient so under high sliding speed contact temperature goes shoots up and that leads to evaporation of liquid nitrogen. If the liquid nitrogen evaporates at the sliding surfaces that can expose the mating couples to room temperature conditions or not the liquid nitrogen but little bit higher temperature in case of alumina Phonon interaction can take place at the cryogenic temperature.

And that essentially leads to a thermal conductivity and this thermal conductivity can proportional to  $1/T$  and this is the in this particular case  $K$  thermal conductivity at 77 kelvin is somehow around 10 times the thermal conductivity what people can measure at room temperature and then third point I have already explained that wear debris has a dual effect. So what is retained the number what is therefore very apparent.

In the high thermal conductivity alumina at room temperature and continuous flushing by liquid nitrogen leads to more intense heat dissipation from the contact zone and this can explain that reduction in that friction coefficient.

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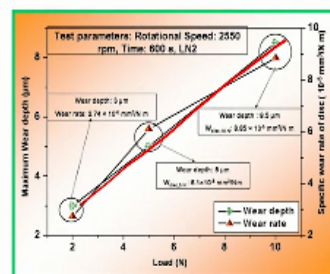
## Dual effect of wear debris particles



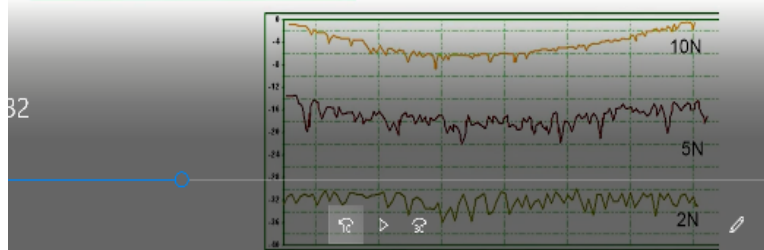
This is a schematic and cartoon image what it shows that this is the typical asperity from the flat and from the wall at high sliding speed you have seen that there is where debris formation and these where they breathe can be crossed further leading to smaller particles. Smaller particles here which are trapped in between the 2 fast bodies and in this is this takes place under pressure and this contact pressure this is the contact diameter what you know and these are contact pressure is controlled  $W/\pi a^2$ .

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## Wear Results



- High wear rate ( $\sim 10^{-5} \text{ mm}^3/\text{N.m}$ ) has been observed.
- Maximum wear depth varied from 3-8.5 µm with increasing load from 2-10N.



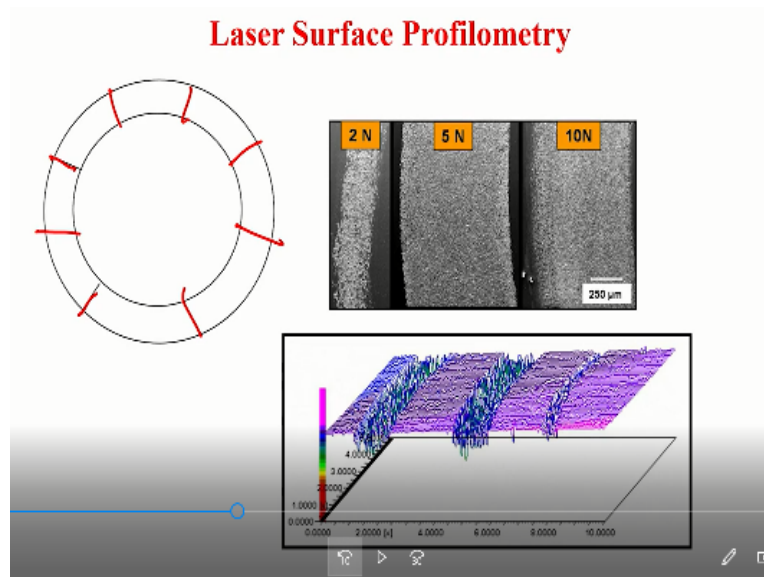
Now as far as the wear results are concerned if you see that where depth it typically where depth increases with increasing the load if you go from 2 to 10 newton. The similar trend like linear increase with load has been also observed in specific wear rate. Now these specific wear rate the

order of magnitude is more important for us and these order of magnitude it may be  $-10$  to the power  $-5$  milli meter cube per newton meter.

So this is typically for ceramics it is around  $10$  to the power  $-6$  millimetre cube newton per meter at unlubricated room temperature sliding conditions for most of the ceramics there can be an exception to that but what you have noticed here in case of alumina at liquid nitrogen temperature cryogenic temperature this wear this little high it is like one order of magnitude higher. Compared to what is typically reported for ceramics at room temperature sliding conditions.

Now if you look at this where depth or 2D surface profile we do not see very large wear depth you know this wear depth is typically limited to  $4$   $5$   $6$  micron depth and  $5$  to  $6$  micron depth also this wear depth is also absorbed in case of  $2$  to  $10$  Newton and  $5$  newton load.

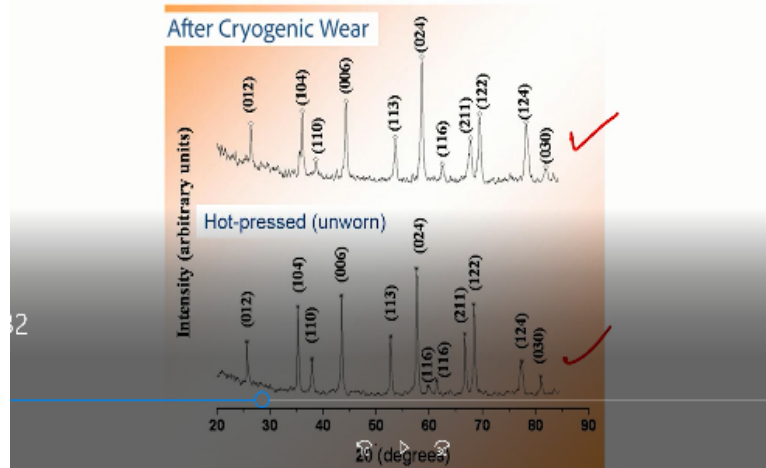
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Now laser surface profilometry that is it standard technique that we used for several other materials and also have used here at these materials so you have a track we can measure the track wheel and also the track tape at different locations and from there you can precisely determine what is the wear volume of the wear track. So all these measurement takes lot of time and but these gives you much more reliable results.

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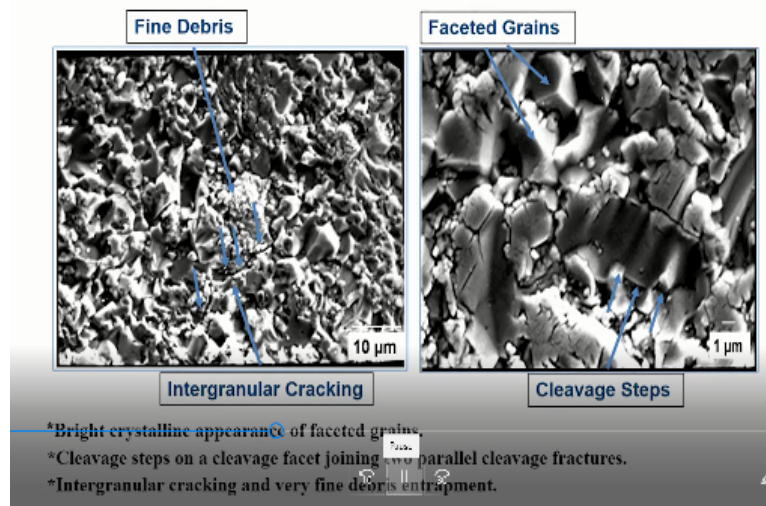
## Phase stability @ Cryogenic Environment



Now as far as the phase stability is concerned so this is the arm on this is the lower one is the unworn surfaces and this is the cryogenic wear surfaces so what do you notice that there is not much difference in terms of the exerted patterns of different phases these are all alpha alumina. Alpha alumina all the characteristic peaks are present with similar intensity on both own and unknown surface.

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### Cryogenic Wear of Alumina Ball after sliding at 10N

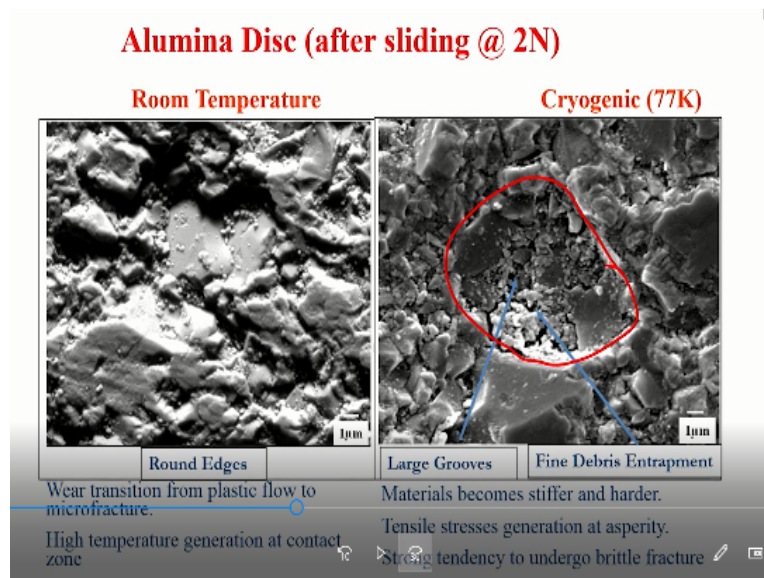


Now these are some of the fascinating microstructures more you will see when I will discuss about these Zirconia. Sliding wear of Zirconia so what do you see there are typically there re general observations of the fine debris particles but more importantly you will see here there are

signs of the cleavage steps okay and this cleavage steps and there are faceted grains these are some of the features.

Which are mostly representative of the brittle fracture in materials like alumina. So internal cracking and fine debris particles cleavage facet joining two parallel cleavage fractures plays.

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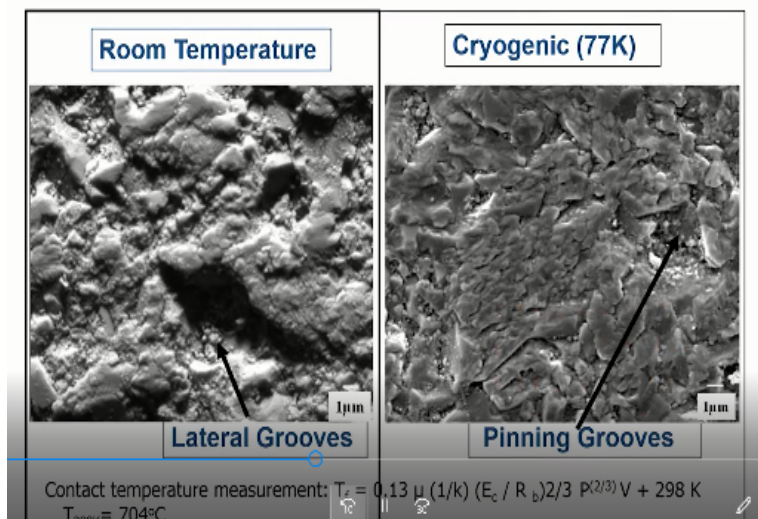


These are kind of some of the characteristic feature of characteristic features now if you compare now room temperature and cryogenic sliding conditions as you have seen these particular image before the cryogenic sliding where there is a deeper groove and also finer debris particles. Those observations we could not make in that after sliding at room temperature after sliding at room temperature all those surfaces are very it rough which is quite expected.

But we did not see much of this cleavage steps but they accept that fact that there is this roughness of the surface is promoted by some fracture on this own surfaces with round edges.

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## Alumina Disc (after sliding @ 10N)



So what we can summarize at room temperature you have plastic flow and fractured whereas severe brittle fracture is taking place at cryogenic sliding conditions.

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## Calculation of Heat dissipation

$$q = \mu Pa V (A_a/A_r)$$

$q$  = Heat generated per unit area ( $J/m^2$ )

$\mu$  = Coefficient of friction

$Pa$  = Load per unit apparent area ( $P/A_a$ ) ( $N/m^2$ )

$P$  = Load (N)

$A_a$  = Apparent area of contact ( $m^2$ )

$A_r$  = Real area of contact ( $m^2$ )

$V$  = Sliding speed (m/s)

$$\text{Frictional heat dissipated (J/m}^2\text{)} = 96 \times 10^{11} \text{ J/m}^2$$

**Fracture Surface Energy of alumina ( $J/m^2$ )**

(1 0 1 1), 298 K  $\rightarrow$  6  $J/m^2$

(1 0 1 0), 298 K  $\rightarrow$  7.3  $J/m^2$

(1 1 2 3), 77 K  $\rightarrow$  32  $J/m^2$

(1 0 1 1), 77 K  $\rightarrow$  24  $J/m^2$

(2 2 4 3), 77 K  $\rightarrow$  16  $J/m^2$

(1 1 2 3), 77 K  $\rightarrow$  24  $J/m^2$

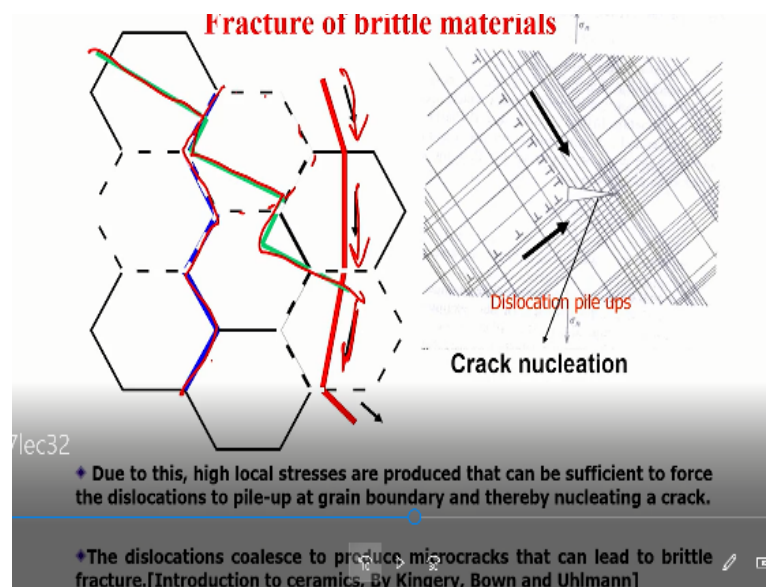
So heat dissipation the contact heat generation as well as heat dissipation these are one of the physical events that take place are during the sliding condition during the sliding tests. So we have calculated what is approximately that the dissipated heat generated per unit area that is  $q$  and that is certainly dependent on coefficient of friction there is a load per unit apparent area and sliding velocity and so on.

And here feature heat dissipated is around  $96 \times 10^{11}$  that is joules per meter square. So these are some of the things that these numbers are quite important. But if you look at the fact of surface image of alumina for different planes for example 101 bar 1 or 101 bar 0 and 112 bar 3 or 101 bar 1 so at 298 kelvin they are around 6 joules per meter square. At higher at lower temperature as you go in the temperature the fractures of the symmetry increases.

So as you can see that 101 bar 1 at 77 kelvin it is going to be 24 joules per meter square at room temperature whether it is 6 joules per meter square it is 4 times larger. So therefore at higher fracture surface energy of alumina is theoretically predicted but here we are seeing extremely high featural dissipated energy. So that is taking place in case of the sliding set in case of cryogenic sliding conditions.

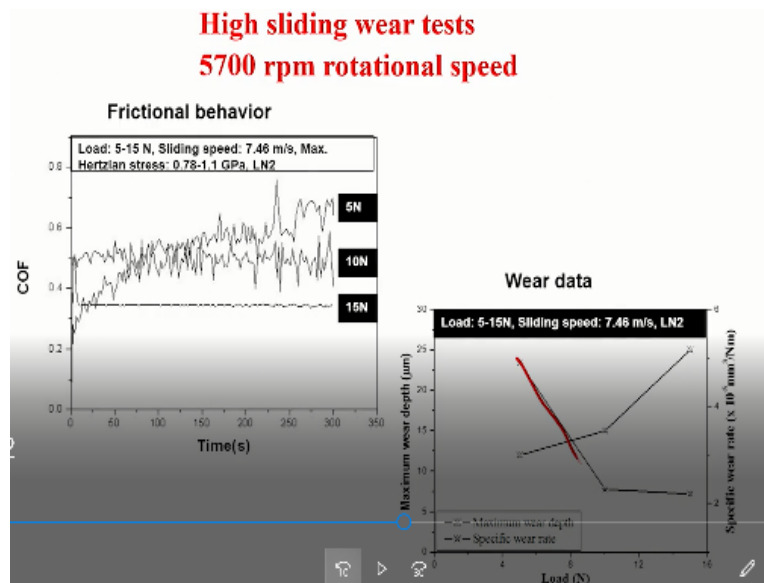
So frictional heat dissipation is certainly energy is much greater than the energies of the factors energies.

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So what we believe that these particular for example if in this way a sliding takes place so there are 2 ways these cracks can propagate one is the inter granular fracture and one is a trans granular fracture like when fracture crack path crosses the grains here. So if it crosses if you see in this cartoon that if it goes through this inter granular crack, so this dotted lines essentially indicate this is the potential grain that can be lifted from the own surface.

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Now if you go through specific wear rate specific wear rate if you see that specific wear rate is used and then maximum wear depth is increased in this particular case of alumina.

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### **Experimental sliding conditions:** **Self-mated Y-TZP**

Testing conditions:

- ☐ Load: 5-15N,
- ☐ Sliding speed: 1.1 m/s,
- ☐ Time: 300s,
- ☐ Max. Hertzian stress: 0.49-0.71 GPa

Now we come to that another sliding system again in the cryogenics gliding conditions and that is the Yttria-Stabilized Tetragonal Zirconia poly crystals and this is the Yttria -Stabilized Tetragonal Zirconia poly crystals we have got the load at 5 newton 10 newton 15 newton sliding speed is 1.1 meter per second time is 5 minutes and maximum Hertzian stress is 0.49 to 0.7 giga pascal.

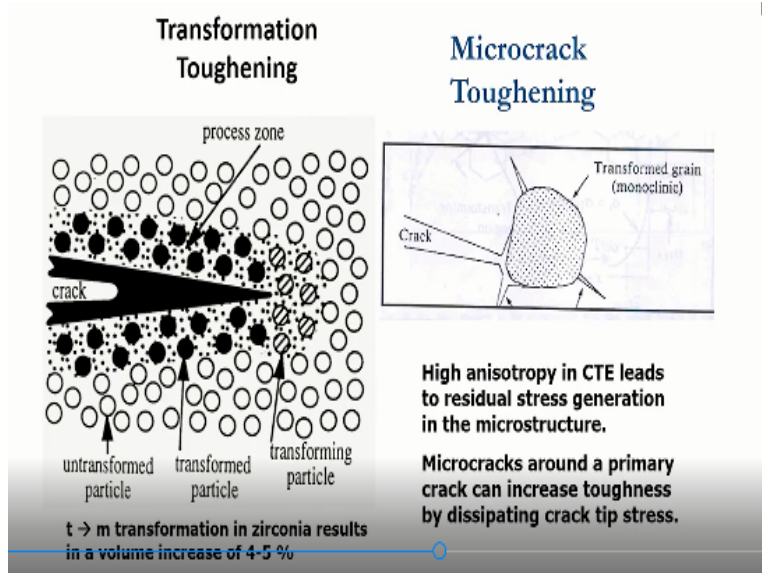
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## Self-mated Zirconia

- Does transformation toughening or microcrack toughening occur in cryogenic environment and if it occurs, how it affects the fracture behavior?
- How does the mechanism of wear change from RT to LN2 temperature?

So the question that we are going to address is this transformation toughening that I have discussed at length in one of our earlier lectures that Tetragonal to monoclinic Zirconia crystals are going whether those and the weather that characteristic first transformation takes place in cryogenic wear or not and how does it influence the wear resistance in this particular case.

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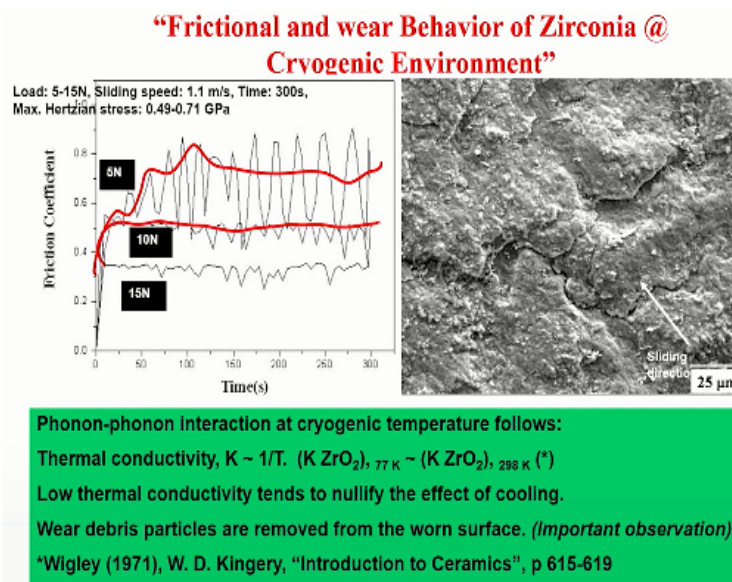


So this is the cartoon the left one is that indicates that when a crack propagates and you have a tetragonal Zirconia retained in the micro structure. Now tetragonal zirconia if it is transformed to monoclinic zirconia and this is a transforming particles these are the transfer mono clinic zirconia that leads to compressive stresses on the crack deep and therefore it leads to the crack deep closer leading to higher crack growth resistance.

So the end point is that as a result of the Tetragonal Monoclinic transformation around the crack deep one can realize higher fracture toughness in these materials other thing is that you have a primary crack deep right and these primary crack deep add that at the tip of the primary crack if there is a tetragonal zirconia phase tetragonal zirconia transforms to monoclinic zirconia it can potentially lead to the micro cracks from the age of the transformed one of monoclinic zirconia.

By causing the micro cracks and by allowing the micro cracks to grow in the microstructure the driving force for the propagation of the primary crack is certainly reduced for what it means that by allowing the micro cracks to grow essentially you are not you are essentially you are restricting the primary crack to grow in the micro structure leading to more fractured toughness.

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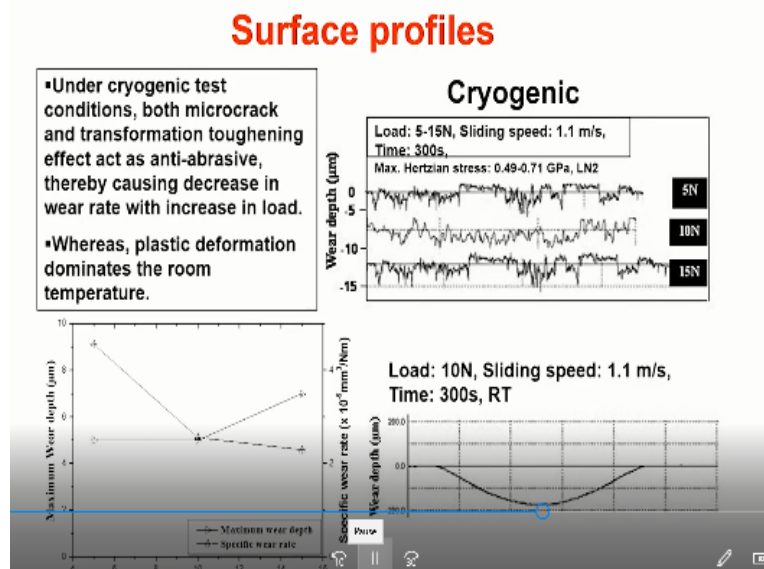
So these are the 2 things that can happen so now let us first look at that that is a frictional behaviour in this particular case of self-mated zirconia of zirconia plate or zirconia disc is being slid against zirconia ball. Now at 15 newton at lower load like 5 newton load that is a lot of undulation in terms of the friction coefficient at 10 Newton load the coefficient of friction is reduced to around 0.5 at 15 Newton load coefficient of friction is reduced further to  $< 0.4$ .

So this is the typical frictional properties but like alumina Zirconia the fractured Zirconia also the thermal conductivity is proportional to  $1/T$ . What it means that but in this particular case that K

of Zirconia like thermal conductivity Zirconia at 77 degrees kelvin is almost similar to of that of the thermal conductivity Zirconia 298 kelvin and low thermal conductivity typically thermal conductivity zirconia is around 2 watts per meter per kelvin.

That tends to nullify the effect of cooling and wear debris particles that are also removed from the own surface.

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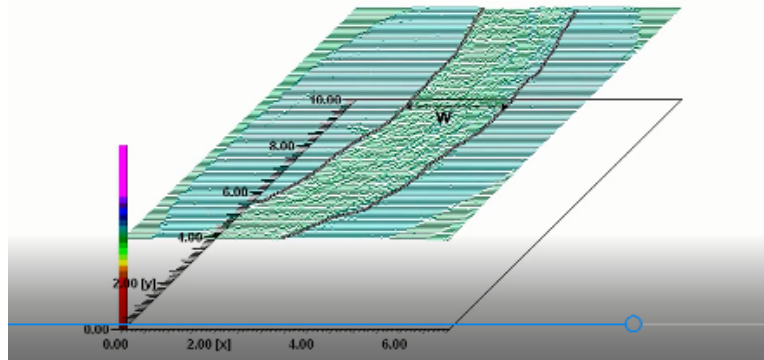
In terms of the wear rate what we have noticed that if you see that maximum wear depth it is going it does not show it maximum where depth shows little increased at higher load but wear rate if you see that shows a systemic decrease with the increase in the load. And this is the typical 2D profiles at room temperature the trace of the 2D profiles clearly show that this is very smooth own surface and this smooth own surfaces are very characteristic features at sliding test of 10 Newton load and sliding speed of 1.1 meter per second.

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## 3D topography of disk track

Load: 15N, Sliding speed: 1.1 m/s, Time: 300s

Width, w: 2.2mm



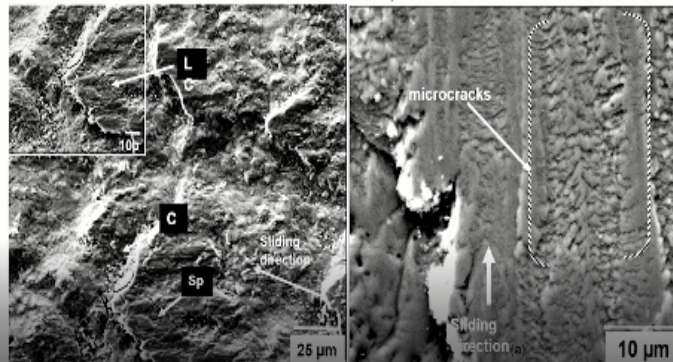
So this is the typical laser surface profilometer of the traces of this how these were tracked there appeared on the own Zirconia.

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## Worn surfaces of Ball after cryogenic sliding

Load: 10N, Sliding speed: 1.1 m/s, Time: 300s, Max. Hertzian stress: 0.62 GPa

Load: 5N, Sliding speed: 1.1 m/s, Time: 300s, Max. Hertzian stress: 0.49 GPa



Possible cause of spalling: Thermal shock resistance  $\propto k/E$

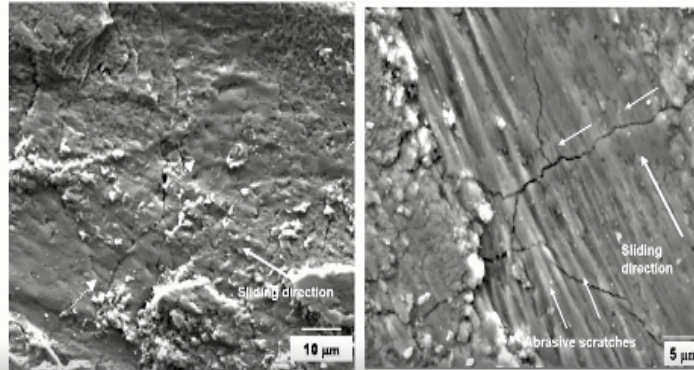
Where,  $k$ = thermal conductivity;  $E$ : elastic modulus

Okay now coming to the discussion on the wear mechanisms of the self-mated zirconia what you see here in low load of 5 newton and sliding speed of 1.1 meter per second this is your sliding directions so there are cracks which are there are numerous cracks and this is that in sample of cracks they are located at 90 degree to the sliding directions and these at load = 10 Newton you also see there are cracks and this is the sliding directions and you can see there are signs of abrasive wear and severe cracking in these materials.

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## Worn surfaces of disc after cryogenic sliding

Load: 5N, Sliding speed: 1.1 m/s, Time: 300s, Max. Hertzian stress: 0.49 GPa  
Load: 15N, Sliding speed: 1.1 m/s, Time: 300s, Max. Hertzian stress: 0.62 GPa



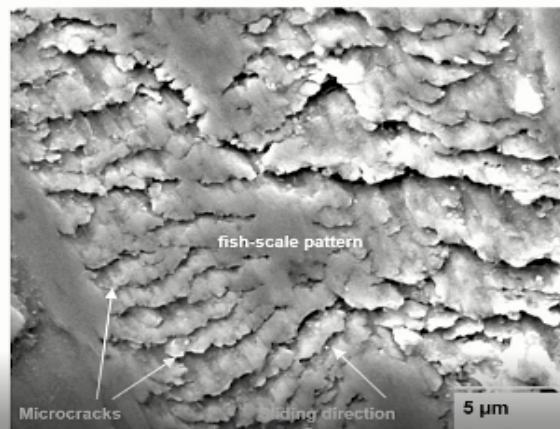
Cracks are emanated perpendicular to sliding direction.

Abrasion and fatigue-cracking: major wear mechanism.

Okay now on that on the disc at 15 newton load this is the sliding directions although these cracks started growing parallel to the sliding directions but they started deviating from the crack path but this is the longer cracks which grows straight across the sliding track and perpendicular to the sliding directions as well.

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Load: 15N, Sliding speed: 1.1 m/s, Time: 300s,  
Max. Hertzian stress: 0.71 GPa



Signature of Microcrack Toughening

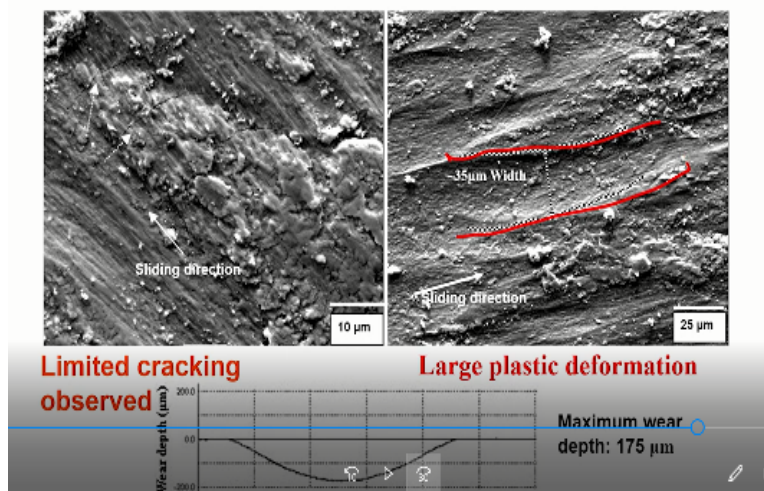
This is one of the most fascinating micro structure that I have ever seen on the own ceramic surface and this is the condition is the load is =15 newton sliding speed is =1.1 meter per second what do you call it a fish scale pattern now if you take any of the freshwater fish and if you look at the scale on the surface of the fish you normally low notice these kind of features. Do you see that these are the scale which is very regularly placed and physically apart?

And these particular fish scale is kind of placed here these kind of micro structural features we call it as a fish tail pattern. So essentially what it means that in this particular case this micro cracks they form they grow preferably at the direction which is perpendicular to the sliding directions here and then they do not grow to a very large extent simply because of the limited sliding time that we have it is like 300 seconds like 5 minutes sliding.

So if you continuously do this sliding it is expected that this particular fish scale can grow significantly leading to the more leading to the formation of a macro crack which can extend to a much larger length.

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### Room temperature sliding wear @10N



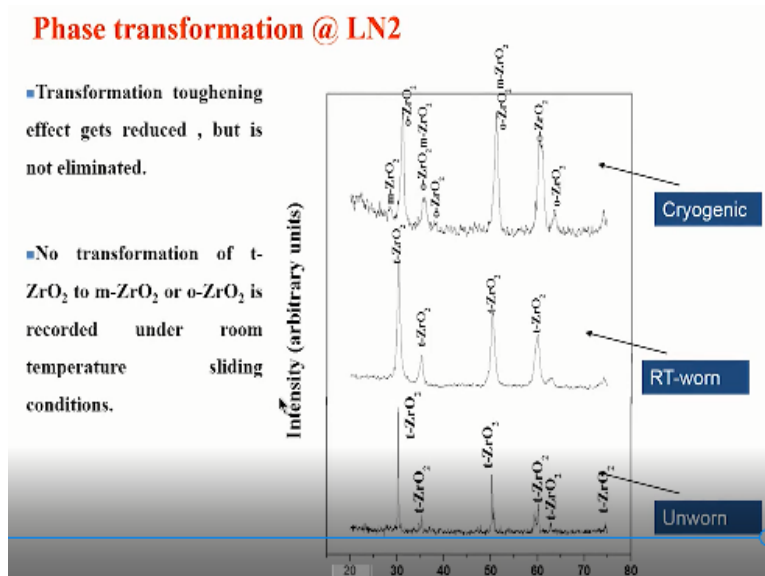
In contrast to this fish scale pattern and micro cracking patterns on the own surface what you notice a completely different scenario when the same tribo system or the same tribo couple are slid at room temperature or ambient temperature okay. What you see here very clear observations of the plastic deformation and grew formation and these group formation is very clear here if you see this is the sliding direction.

And there is a kind of 35 micron width these typical plastic groups and also these and this is that kind of depth is quite high as you can see. But in the here I have given these 2D laser profilometer traces for you to realize from the scale it is 200 micron so that depth is around 175

micron. So 35 micron width and typical I am talking about typical length scale and 175 micron is the tape.

So essentially what you notice what do you see that width have these kind of 5 times. So it is much deeper this group formation that in taking place we did not see any signature of micro cracking except there are 1 or 2 occasions of the cracking but while in cryogenic sliding conditions we did notice significant cracking, micro cracking on this own surfaces.

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Now from material science point of view it is also important for us to analyse that what is the whether the phase transformation of Zirconia is taking place during the sliding wear conditions for that what we have done we have taken their sliding track we have recorded that extra dye fraction pattern from the unworn that is before the sliding test is over then after that we have focused on extra beam to warm the sliding wear track.

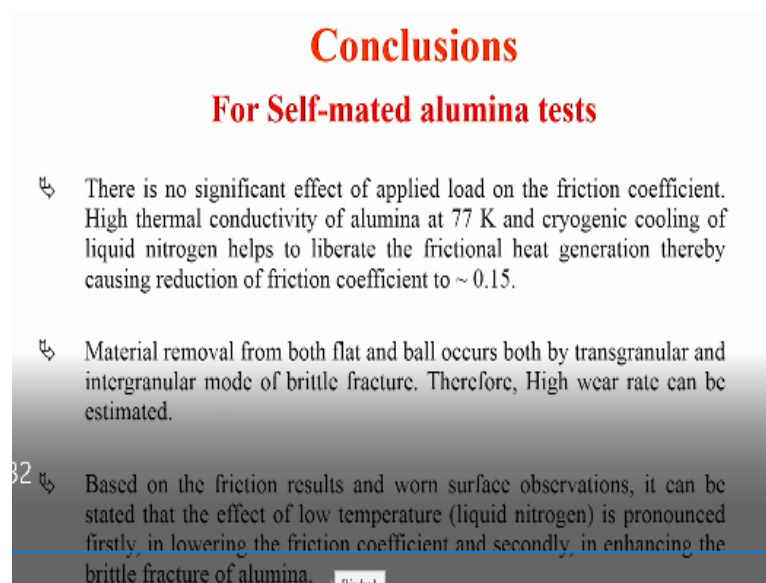
After the room temperature sliding conditions and also liquid nitrogen sliding conditions what do you notice that is a very sharp crystalline Tetragonal Zirconia no sign of mono clinic zirconia or any other phase. After room temperature sliding conditions we do see Tetragonal Zirconia is present here however at different observations we have made when we have closely looked at the exerted traces what we have recorded at the cryogenic sliding conditions.

We see some signs of orthorhombic Zirconia and monoclinic zirconia. But some of the peaks are very critical we were very clear science of this orthorhombic zirconia only. So what happens in the tetragonal zirconia can undergo phase transformation to orthorhombic zirconia as an intermediate transformation product before it goes to full transformation to monoclinic zirconia. So what happens in cryogenic sliding conditions you are doing these test for 5 minutes.

So you are not allowing the system to undergo full transformation from tetragonal to monoclinic zirconia and that is quite an interesting observations that we have clearly see at the same time scale when tetragonal zirconia is retained it does not undergo any first hand transformation at room temperature sliding but under cryogenic sliding condition and the same samples and the identical sliding conditions.

Tetragonal zirconia undergoes phase transformation to orthorhombic zirconia and there are also signature of thermionic Zirconia formation.

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### Conclusions

#### For Self-mated alumina tests

- ↳ There is no significant effect of applied load on the friction coefficient. High thermal conductivity of alumina at 77 K and cryogenic cooling of liquid nitrogen helps to liberate the frictional heat generation thereby causing reduction of friction coefficient to  $\sim 0.15$ .
- ↳ Material removal from both flat and ball occurs both by transgranular and intergranular mode of brittle fracture. Therefore, High wear rate can be estimated.
- 32 ↳ Based on the friction results and worn surface observations, it can be stated that the effect of low temperature (liquid nitrogen) is pronounced firstly, in lowering the friction coefficient and secondly, in enhancing the brittle fracture of alumina.

Slide back

So this is the kind of a closure notes on the self-mated alumina we have seen that is significant effect of load and also material removal from both flat and ball by trans granular and intergranular mode of brittle fracture.

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## For Self-mated Zirconia tests

- High steady state COF of 0.35-0.75 can be experienced for tribological testing at 5-15N load and high rotational speed (850 rpm) in LN<sub>2</sub>. A linear decrease in wear rate ( $3.8 \times 10^{-4}$ - $7 \times 10^{-6}$  mm<sup>3</sup>/Nm) with increasing load (5-15N) is measured in our experiments.
- Under the selected operating conditions, t-ZrO<sub>2</sub> transforms to o-ZrO<sub>2</sub> and m-ZrO<sub>2</sub> phase during sliding in LN<sub>2</sub> environment, whereas no transformation was observed at RT test conditions.
- Spalling and microcrack-induced damage are the major wear mechanisms under the cryogenic test conditions; whereas large plastic deformation is the predominant wear mechanism under RT sliding.

In case of zirconia high steady state COF of 0.35-0.75 has been noticed and it is experienced for tribological testing at 5 to 15 Newton and then spalling and micro crack induced damage are the major wear mechanism under the cryogenics sliding conditions. Thank you.