Friction and Wear of Materials: Principles and Case Studies

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Lecture – 31
Overview: Cryogenic Wear Properties of Materials

So, welcome back to this NPTEL course on Tribology of materials in this lecture as well as the following lectures I will introduce you to 1 of the newer concepts and you are understanding of the tribological properties of materials as the slide says.

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Overview: cryogenic wear properties of materials

First I will give an overview of cryogenic wear of materials so cryogenic means here I will be discussing particularly how the friction and wear properties of several materials had influenced because of the change in taste environment from ambience to liquid nitrogen environment. So, for that 1 has to have a very special test streak with these test streaks one can evaluate the friction and wear properties of different materials.

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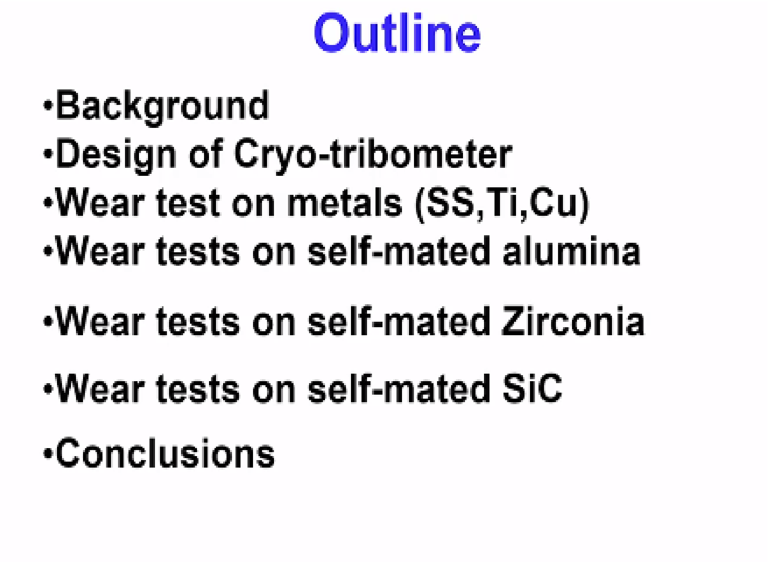

Outline

- •Background
- •Design of Cryo-tribometer
- •Wear test on metals (SS,Ti,Cu)
- •Wear tests on self-mated alumina
- •Wear tests on self-mated Zirconia
- •Wear tests on self-mated SiC
- •Conclusions

So, first I will give a brief background then that truly followed by design of what I say Cryo – tribometer like for high temperature tribology we need to have a separate tribometer similarly for cryogenic testing facility also we need to have a different test facility. Then I will give you some illustrative examples of the wear test and metals like stainless steel titanium copper followed by alumina and the Zirconia. Next to next a couple of lectures and silicon carbide and then I will conclude.

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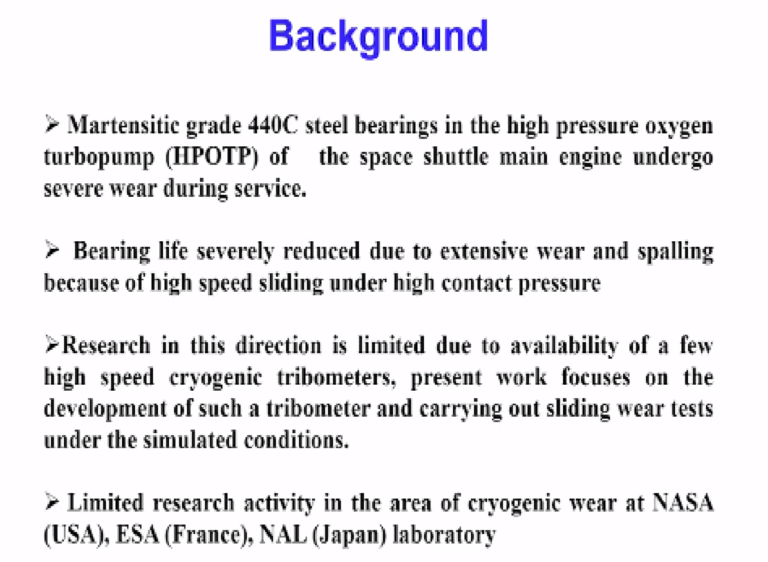

Background

➢ Martensitic grade 440C steel bearings in the high pressure oxygen turbopump (HPOTP) of the space shuttle main engine undergo severe wear during service.

➢ Bearing life severely reduced due to extensive wear and spalling because of high speed sliding under high contact pressure

➢Research in this direction is limited due to availability of a few high speed cryogenic tribometers, present work focuses on the development of such a tribometer and carrying out sliding wear tests under the simulated conditions.

➢ Limited research activity in the area of cryogenic wear at NASA (USA), ESA (France), NAL (Japan) laboratory

So, typically for cryo turbo pumps which is used in the space shuttle main engine those kind of very critical engineering very critical engines their require martensitic stainless steel bearings and this martensitic stainless steel is 440 secret at present. Bearing life is restricted because of the extensive wear and spalling because of this high speed sliding conditions under high contact pressure.

However, if we want to explore new materials to replace a 440C steel bearings 1 needs to have suitable experimental setup for high speed cryogenic tribometers and limited research activity in the cryogenic wear is currently being conducted at NASA, ESA European Space Agency and in NAL, Japan. These are the laboratories.

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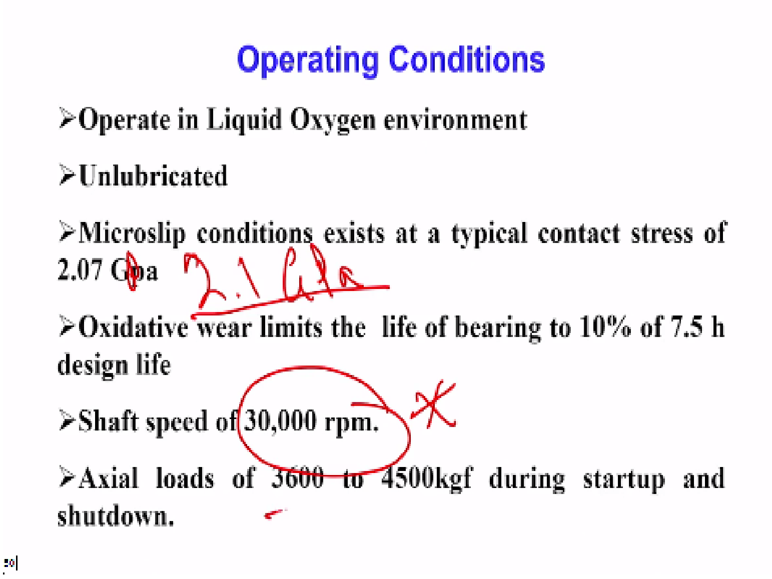

Operating Conditions

- Operate in Liquid Oxygen environment
- Unlubricated
- Microslip conditions exists at a typical contact stress of 2.07 Gpa 2.1 GPa
- Oxidative wear limits the life of bearing to 10% of 7.5 h design life
- Shaft speed of 30,000 rpm.
- Axial loads of 3600 to 4500kgf during startup and shutdown.

What are the operating conditions in this cryo- tribometer this operating conditions typical is liquid oxygen environment mostly it is unlubricated? Unlubricated contract conditions and there is a micro slip at a typical contact stress of around 2.01giga pascal. So, 2.1 gigapascal that is fairly high contact stress right and oxidative wears limits the life of these 440 C steel bearings and typical shafts speed at which the sliding takes place is 30000rpm.

30000 rpm is extremely high sliding speed and there is an axial load somewhere around 3600 to 4500 kilogram force during start up and start down.

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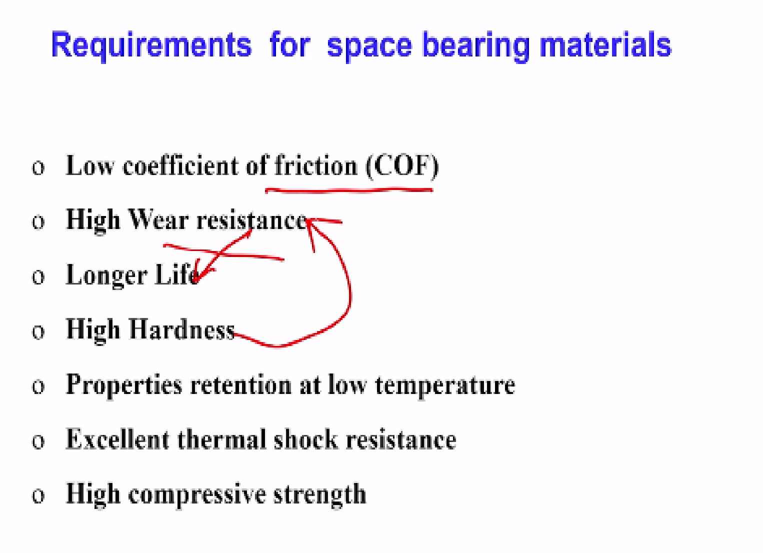

Requirements for space bearing materials

- Low coefficient of friction (COF)
- High Wear resistance
- Longer Life
- High Hardness
- Properties retention at low temperature
- Excellent thermal shock resistance
- High compressive strength

So, therefore against this backdrop of this requirement but against this backdrop the requirements for space bearing materials include it should have a low coefficient of friction and also high wear resistance and high wear resistance leads to longer life hardness is required to provide to ensure high wear resistance and properties should be retained at low temperature excellent thermal shock resistance and high compressive strength.

High compressive strength is required because as you have seen in the last slide I have mentioned 2 1 giga pascal is a typical contact stress.

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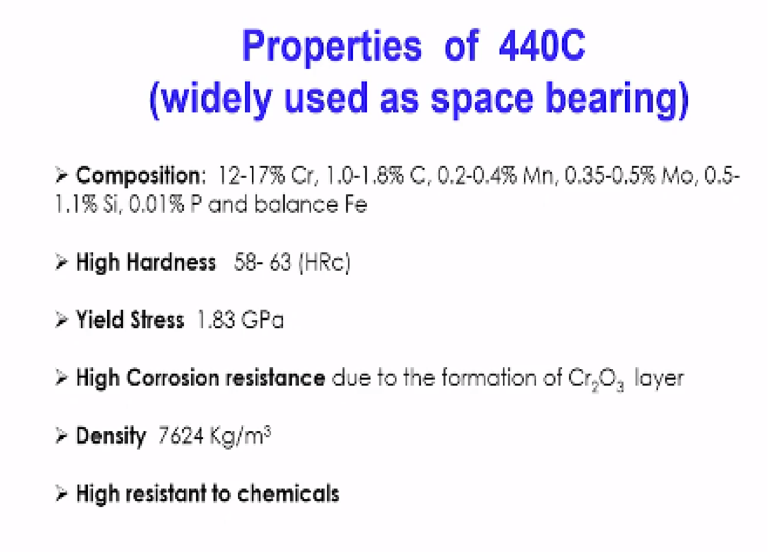

Properties of 440C (widely used as space bearing)

- **Composition:** 12-17% Cr, 1.0-1.8% C, 0.2-0.4% Mn, 0.35-0.5% Mo, 0.5-1.1% Si, 0.01% P and balance Fe
- **High Hardness** 58- 63 (HRc)
- **Yield Stress** 1.83 GPa
- **High Corrosion resistance** due to the formation of Cr_2O_3 layer
- **Density** 7624 Kg/m^3
- **High resistant to chemicals**

In order to sustain that kind of high contact stress materials must have high compressive strength now here goes that typical composition and other properties are 440C steel bearing is space bearing materials. The composition is around 12% to 17% chromium 1 to 1. 8% carbon fairly high carbon content and it has magnate each molly silicone and phosphorus. It has hardness very high around 58 to 63 in rock well see scale.

Yield stress is around 1.8 giga pascal high corrosion resistances and high resistance to chemicals.

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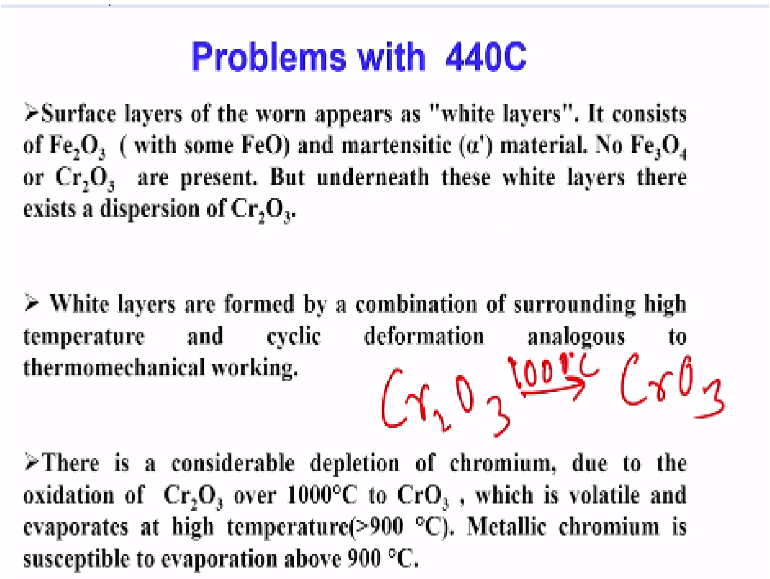

Problems with 440C

- Surface layers of the worn appears as "white layers". It consists of Fe_2O_3 (with some FeO) and martensitic (α') material. No Fe_3O_4 or Cr_2O_3 are present. But underneath these white layers there exists a dispersion of Cr_2O_3.

- White layers are formed by a combination of surrounding high temperature and cyclic deformation analogous to thermomechanical working.

$Cr_2O_3 \xrightarrow{1000°C} CrO_3$

- There is a considerable depletion of chromium, due to the oxidation of Cr_2O_3 over 1000°C to CrO_3 , which is volatile and evaporates at high temperature(>900 °C). Metallic chromium is susceptible to evaporation above 900 °C.

But how about the problems with 440 C is that typically if you do this wear test the surface layers of the worn surface appears white layers and white layers means it is essentially iron oxide with some SCO and martins it take alpha prime material and there is absolutely no Fe34 or CO3 and white layers are formed by a combination of surrounding high temperature and slightly deformation.

And there is a considerable depletion of chromium due to the oxidation of chromium oxide at around 1000degree Celsius to CrO3 and Cr2O3 that goes to CrO3 at 1000 degree Celsius. So, this kind of this kind of oxidation takes place at 1000 degrees Celsius and it evaporates that more than 900 degrees Celsius and metallic chromium is also susceptible to evaporation above 900 degree Celsius.

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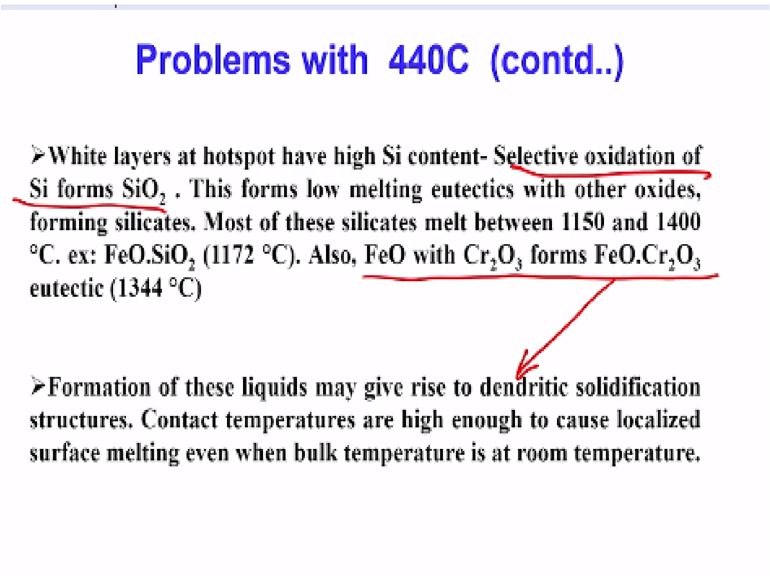

Problems with 440C (contd..)

- White layers at hotspot have high Si content- Selective oxidation of Si forms SiO_2 . This forms low melting eutectics with other oxides, forming silicates. Most of these silicates melt between 1150 and 1400 °C. ex: $FeO.SiO_2$ (1172 °C). Also, FeO with Cr_2O_3 forms $FeO.Cr_2O_3$ eutectic (1344 °C)
- Formation of these liquids may give rise to dendritic solidification structures. Contact temperatures are high enough to cause localized surface melting even when bulk temperature is at room temperature.

Now essentially this white layers that leads to selective oxidation of silicon to form silica iron oxide can form a double compound called FeO with CrO3 at 1344 degree Celsius. Now all these dendritic is low temperature liquid that may give rise to dendritic solidification which is very characteristic for many of the metals and alloys and contact temperatures are high enough to cause localized surface melting even when bulk temperature is at the room temperature.

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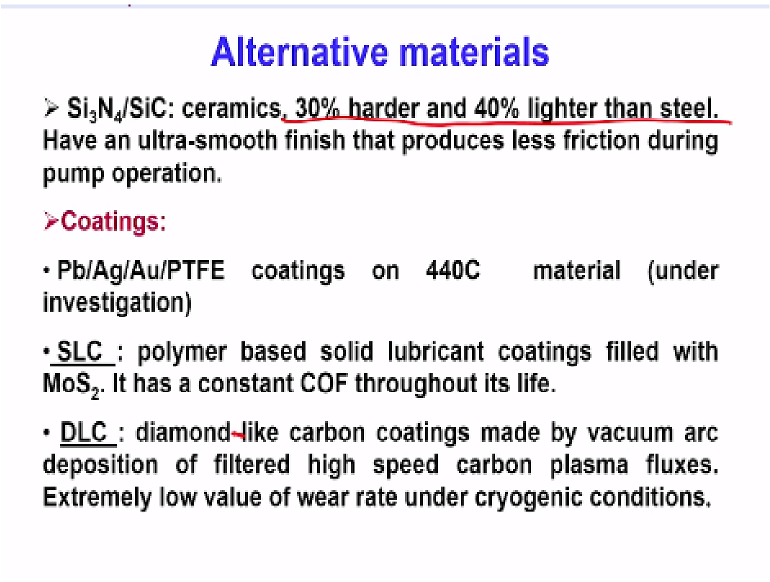

Alternative materials

- Si_3N_4/SiC: ceramics, 30% harder and 40% lighter than steel. Have an ultra-smooth finish that produces less friction during pump operation.
- Coatings:
  - Pb/Ag/Au/PTFE coatings on 440C material (under investigation)
  - SLC : polymer based solid lubricant coatings filled with MoS_2. It has a constant COF throughout its life.
  - DLC : diamond-like carbon coatings made by vacuum arc deposition of filtered high speed carbon plasma fluxes. Extremely low value of wear rate under cryogenic conditions.

Now what are the alternative materials? Alternative materials is certainly silicon ca nitride silicon carbide these headlights ceramic materials extremely registered to oxidate by be extremely resistant to oxidation particularly at such low temperature. It is 30% harder and 40% lighter than steel as you see these numbers. These numbers indicate that these materials while being lighter it is also harder.

Harder means it may have good abrasion resistance so what are the different coatings like late silver gold PTFE coatings on 440C material and SLC so polymer based solid lubricant coatings. SLC stands for solid lubricant coatings filled with MoS2 and it has a constant COF throughout its life and DLC is another material that is diamond like carbon coatings and these are made of they are made by vacuum or deposition of filtered high speed carbon plasma fluxes and this has the extremely low value of wear rate under cryogenic conditions.

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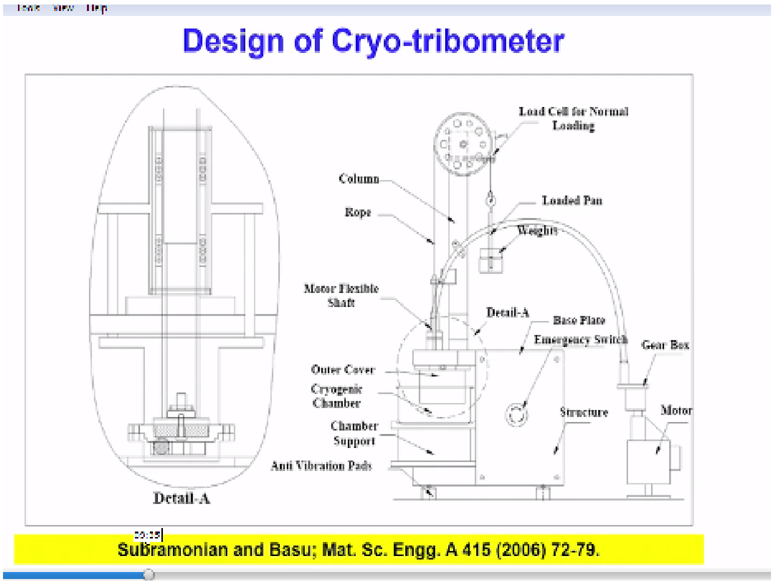

Design of Cryo-tribometer

Subramonian and Basu; Mat. Sc. Engg. A 415 (2006) 72-79.

Now this is the custom designed cryogenic wear tester that we have are developed and designed a way back more than a decade back as part of our space tribology program with the Indian space research organization is strong and what it typically does this particular staff and pulley regiment allows to experience that allows these are disc and pin assembly to experience extremely high relative motion and this speed rotational speed of the disc.

And go up to 36000 rpm and 36000 rpm is certainly sufficient to stimulate the space bearing applications as I mentioned a few minutes back and the load is somewhere around 2 to 20 newton. This load it can be varied by date wise and then one can record the friction coefficient extreme at very high sliding speed condition so this is kind of a very unique cryogenic tribo tester.

The other things I must mention you that this is this entire SMD is dipped into this particular cryo chamber and described to chamber that there is a constant flow of liquid nitrogen so that at any given point of time these particular assembly is completely emerged in liquid nitrogen and that is very important. Because liquid nitrogen if it is this if it is filled up in if it is submerged in liquid nitrogen then only we can simulate that cryogenic a sliding conditions.

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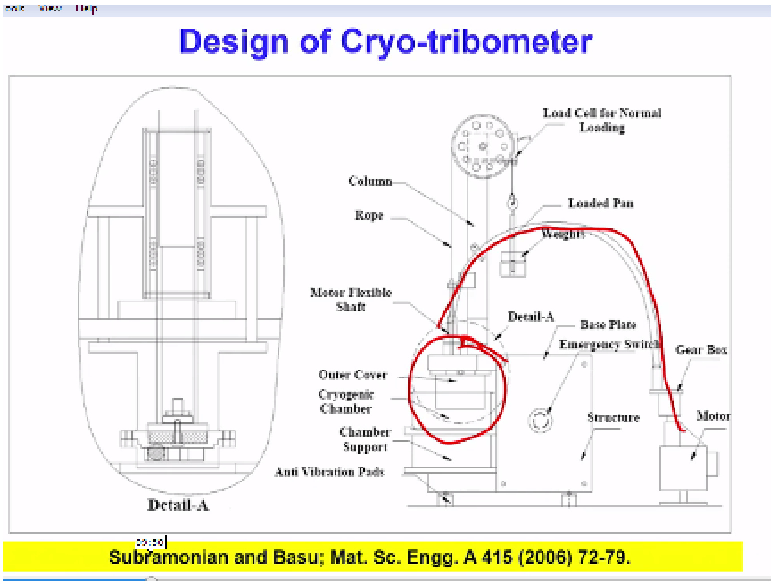

Design of Cryo-tribometer

Subramonian and Basu; Mat. Sc. Engg. A 415 (2006) 72-79.

This is much more detailed design of describing the Cryo – tribometer you can see this is the main region this is the cryogenic chamber and this is the motor and gearbox. This kind of a motor and flexible shaft and this kind of assembly external assembly allows that this to rotate at extremely high speed.

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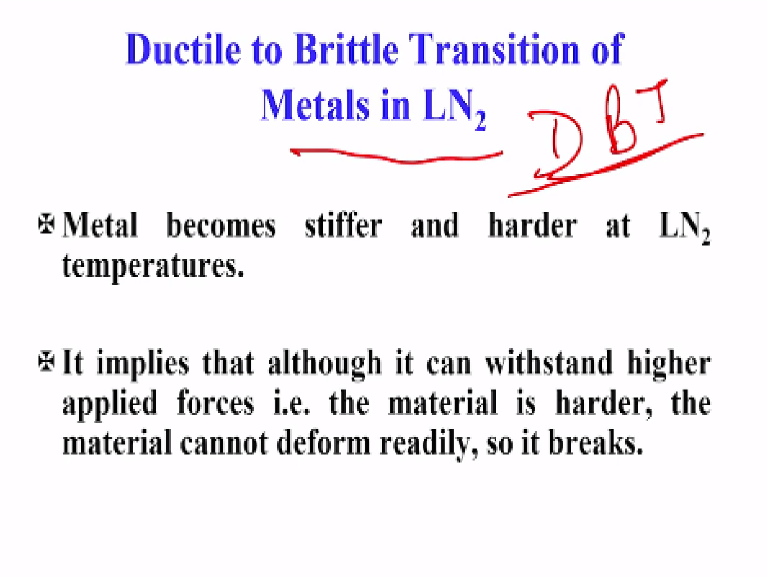

Ductile to Brittle Transition of Metals in LN_2

DBT

- Metal becomes stiffer and harder at LN_2 temperatures.
- It implies that although it can withstand higher applied forces i.e. the material is harder, the material cannot deform readily, so it breaks.

So, one of the problem with metal also that in new cryogenic conditions the metal can undergo Ductile to Brittle Transition. This is called DBT those who are from metal logy background they know that DBT leads to catastrophic failure at sub-zero temperature for many of the metals but that certainly would not be the case for ceramic based materials because ceramic based materials unlike metals they do not have our Ductile to Brittle Transition.

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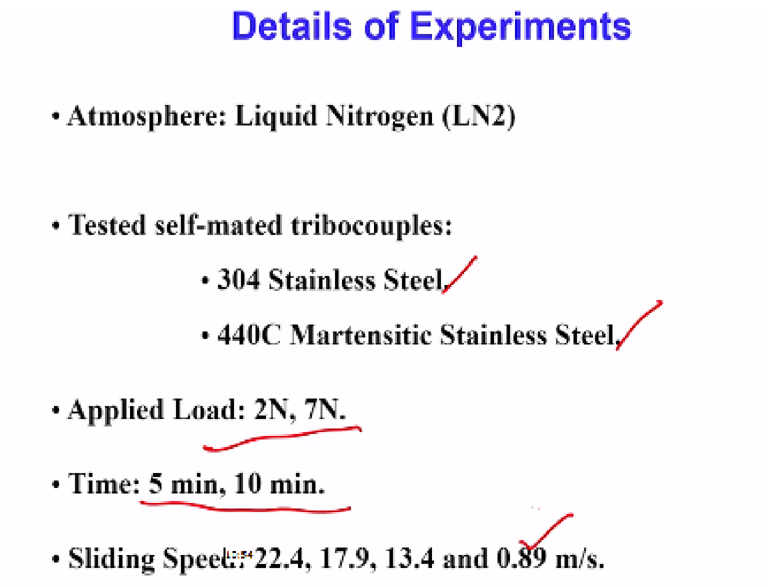

Details of Experiments

- **Atmosphere: Liquid Nitrogen (LN2)**
- **Tested self-mated tribocouples:**
  - **304 Stainless Steel**
  - **440C Martensitic Stainless Steel**
- **Applied Load: 2N, 7N.**
- **Time: 5 min, 10 min.**
- **Sliding Speed: 22.4, 17.9, 13.4 and 0.89 m/s.**

So what are the materials that we have done the experiments when you started the space tribology program a first set of materials we did is 304 stainless steel and 440C martensitic stainless steel the load is 2Newton and 7 Newton we cannot do these taste for extremely longer time period longer sliding time but certainly at 5minutes to 10 minutes time sliding speed was varied 0 89 meter per second to 22.4 meter per second so that is extremely high sliding speed.

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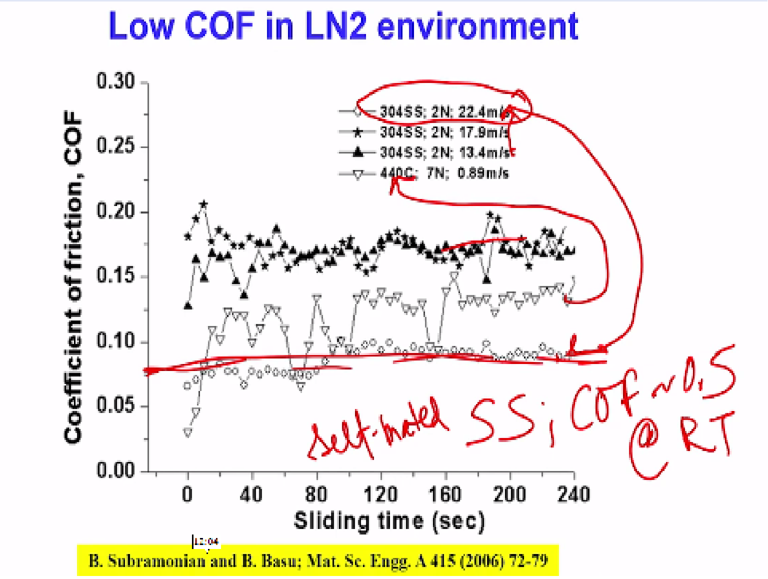

Low COF in LN2 environment

Self-mated SS; COF ~0.5 @ RT

B. Subramonian and B. Basu; Mat. Sc. Engg. A 415 (2006) 72-79

So you see that this is the circle ones is the 304 stainless steel this is for the circle 1 this 1 is for 440C martensitic steel at 0.89 meter per second and you have these 2 newton and when you increase the speed you see that typically at higher and at higher speed. The coefficient of friction actually slower so one can get extremely low coefficient of friction at around 0.08 at speed of 22.4 meter per second.

Typically, stainless steel there for self-made of stainless steel if I am talking about the self-metal stainless steel at room temperature. The COF is around 0.5 so this is at 0.5 at room temperature but at decode it at a temperature at the highest sliding speed conditions as you can notice here. That coefficient of friction almost reduced 5 times less. So, .52 < 0.1 so these kinds of things and this is one of the important observations here.

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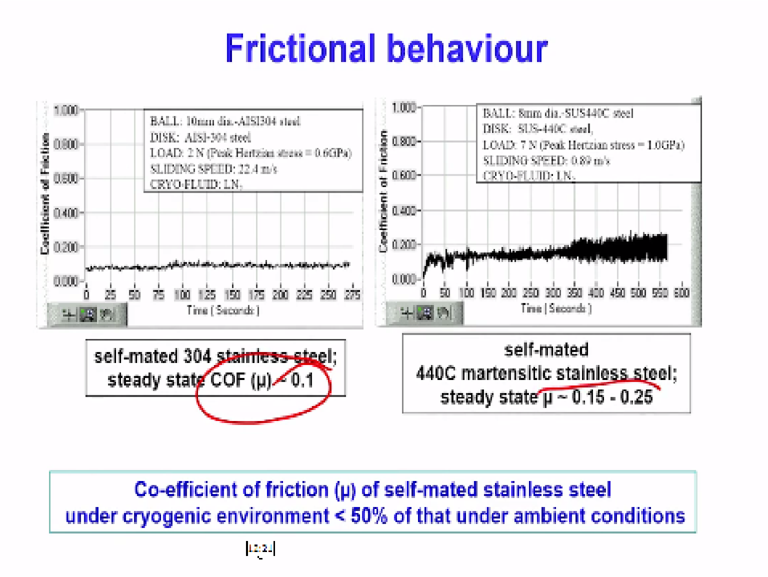

Frictional behaviour

self-mated 304 stainless steel;
steady state COF (μ) ~ 0.1

self-mated
440C martensitic stainless steel;
steady state μ ~ 0.15 - 0.25

Co-efficient of friction (μ) of self-mated stainless steel
under cryogenic environment < 50% of that under ambient conditions

Now when you do these other when we have done the other experiments like self-mated 304 stainless steel and 304 stainless steel here COF is 0 .1 and in 440 stainless steels uf is 0. 15 to 0.25. So, it is very clear that at liquid nitrogen cryogenic temperature that so frictional behaviour is significantly influenced.

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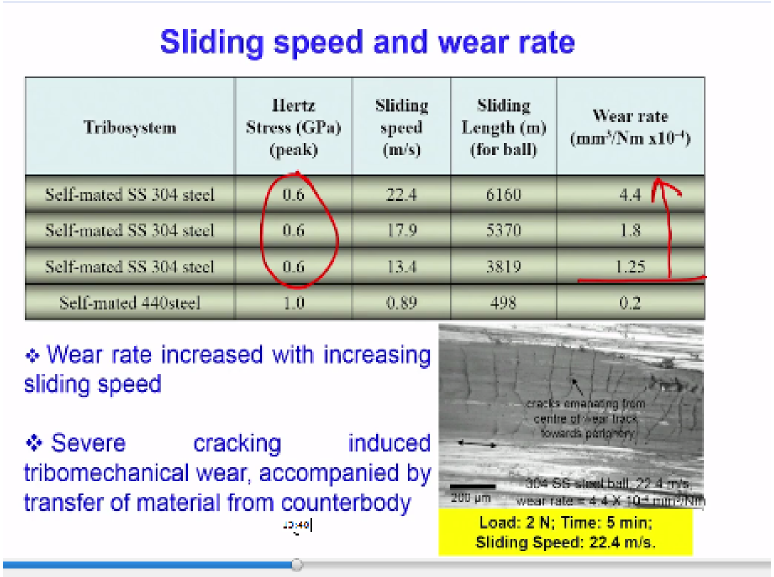

Sliding speed and wear rate

Tribosystem	Hertz Stress (GPa) (peak)	Sliding speed (m/s)	Sliding Length (m) (for ball)	Wear rate ($mm^3/Nm \times 10^{-4}$)
Self-mated SS 304 steel	0.6	22.4	6160	4.4
Self-mated SS 304 steel	0.6	17.9	5370	1.8
Self-mated SS 304 steel	0.6	13.4	3819	1.25
Self-mated 440steel	1.0	0.89	498	0.2

- ❖ Wear rate increased with increasing sliding speed
- ❖ Severe cracking induced tribomechanical wear, accompanied by transfer of material from counterbody

Load: 2 N; Time: 5 min;
Sliding Speed: 22.4 m/s.

Now if you look at the wear it of these materials if you look at this as you increase the sliding speed the wear it is essentially increases for particularly for these 3 materials wear self-mated 304 steel is hertz stress 0.6 giga pascal which is not very low value. It is fairly high value and so essentially your coefficient with sliding speed your coefficient of friction is reduced but wear it increases.

At the fairly initial lectures of this NPTEL course, I have mentioned that coefficient of friction and wear resistant they cannot. They are not directly correlated what it means what I made it very categorically at that point of time that in a tribo couple makes experience local coefficient of friction. But the same tribal couple can experience high wear it similarly a tribe couple may experience extremely high coefficient of friction.

But the same tribe a couple under the identical conditions can experience low wearied so these are like you know it is not directly correlated.

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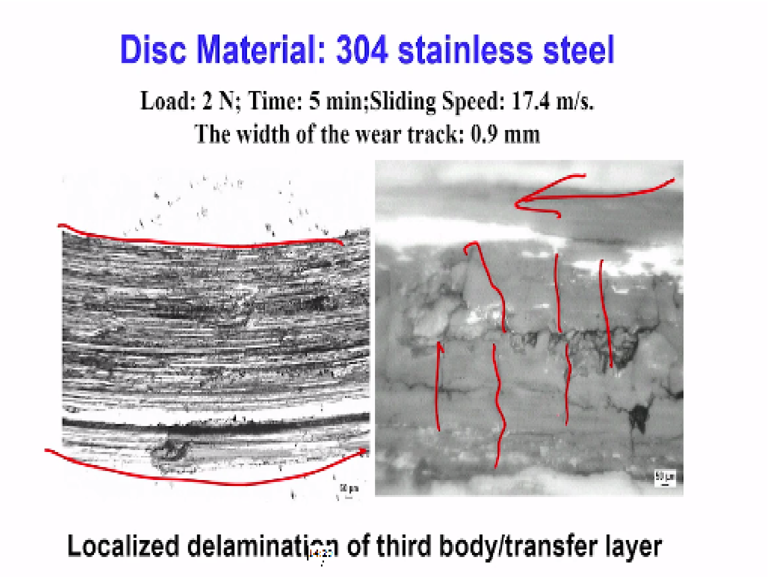

Disc Material: 304 stainless steel

Load: 2 N; Time: 5 min;Sliding Speed: 17.4 m/s.
The width of the wear track: 0.9 mm

Localized delamination of third body/transfer layer

Now of you see 304 stainless steel it is 2N load and sliding speed is 17.4 metre per second and this is a typical how this track wear track sliding wear track they look like and if you look at this a little bit if you look at the higher magnification optical microscopy images. You can clearly see this because they are sliding is taking place in this direction and there are cracking who have most of the cracks they are oriented perpendicular to the sliding directions.

This is quite expected because the tensile stress direction the cracks wall is provocateur on the tensile stress directions.

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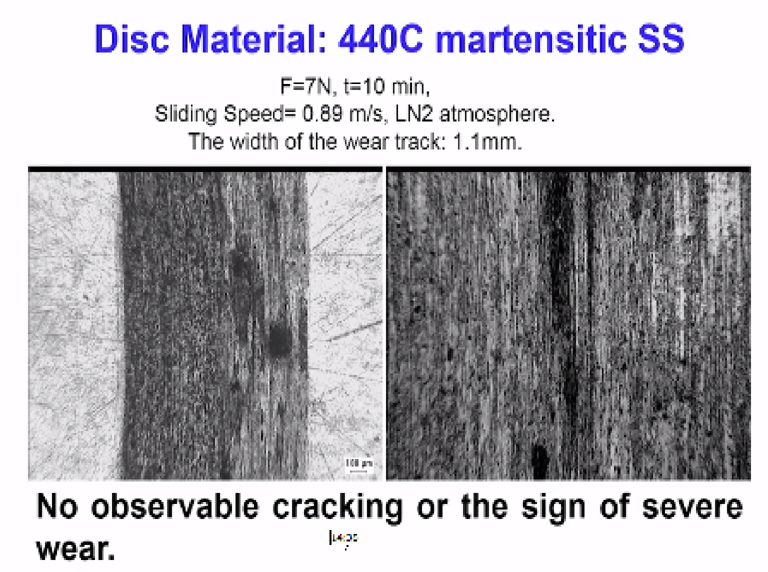

Disc Material: 440C martensitic SS

F=7N, t=10 min,
Sliding Speed= 0.89 m/s, LN2 atmosphere.
The width of the wear track: 1.1mm.

No observable cracking or the sign of severe wear.

In 440C martensitic stainless steel we do not see any observation of cracking on the worn surface like what you have noticed here.

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Cryogenic Wear of SUS 440C ball Vs coated SUS 440C disk

Now I will just show you some of the other research results where we have used our stainless steel for 440C ball versus coated stainless steel 440C disc.

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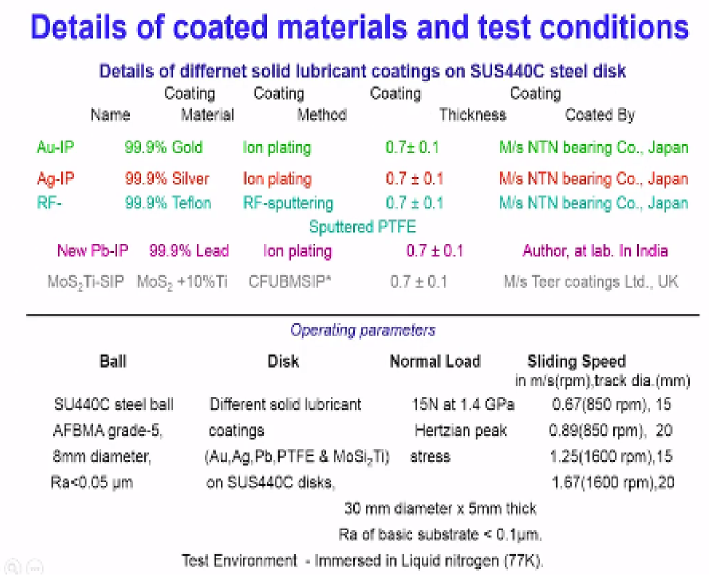

Details of coated materials and test conditions

Details of differnet solid lubricant coatings on SUS440C steel disk

Name	Coating Material	Coating Method	Coating Thickness	Coated By
Au-IP	99.9% Gold	Ion plating	0.7± 0.1	M/s NTN bearing Co., Japan
Ag-IP	99.9% Silver	Ion plating	0.7 ± 0.1	M/s NTN bearing Co., Japan
RF- Sputtered PTFE	99.9% Teflon	RF-sputtering	0.7 ± 0.1	M/s NTN bearing Co., Japan
New Pb-IP	99.9% Lead	Ion plating	0.7 ± 0.1	Author, at lab. In India
MoS_2Ti-SIP	MoS_2 +10%Ti	CFUBMSIP*	0.7 ± 0.1	M/s Teer coatings Ltd., UK

Operating parameters

Ball	Disk	Normal Load	Sliding Speed in m/s(rpm),track dia.(mm)
SU440C steel ball AFBMA grade-5, 8mm diameter, Ra<0.05 µm	Different solid lubricant coatings (Au,Ag,Pb,PTFE & $MoSi_2Ti$) on SUS440C disks,	15N at 1.4 GPa Hertzian peak stress	0.67(850 rpm), 15 0.89(850 rpm), 20 1.25(1600 rpm),15 1.67(1600 rpm),20

30 mm diameter x 5mm thick

Ra of basic substrate < 0.1µm.

Test Environment - Immersed in Liquid nitrogen (77K).

So what it means is that so we have put these coatings and this particular part of that research was done in collaboration with Satoshi Takei coaters lab in japan and so you can use these gold indium phosphate coatings and silver indium phosphate and these are the coatings these are these are obtained from NTN bearing company japan and this coating thickness is typically .7 micron so 0.7 micron coating is fairly thin coating.

And they have a different grade of coatings like Ti , UK also we have got Mos2 titanium Si coatings so what are the different operating parameters? different operating parameters is that you have a ball as SUS440 C ball you have a disc different solid lubricant your normal load is around 1.4 giga pascal and your sliding speed is 0.67 to it can go to up to 1.67 meter per second.

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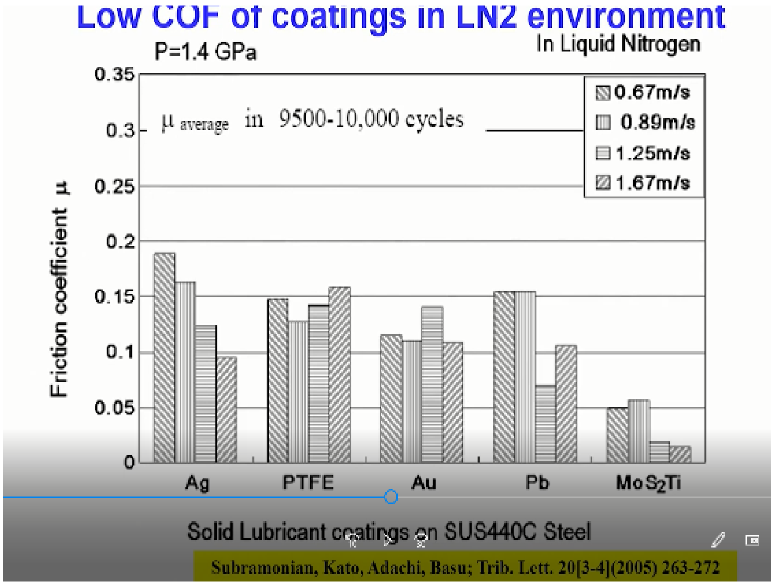

Low COF of coatings in LN2 environment

Subramonian, Kato, Adachi, Basu; Trib. Lett. 20[3-4](2005) 263-272

Now let us look at this a summary of the free coefficient of friction results what do you notice here? Clearly Mos2 with titanium when it is quoted on 440C it has a coefficient of friction of 0.05. So 0.05 co efficient friction is fairly small and fairly good values now if you have simple gold coating that means friction is around 0.1 higher if you have silver coatings this is 0.1 and above up to 0.17. So this is that new average in 9500 to 10 000 cycles and different bars essentially indicate what is a sliding speed that you use you or a sliding experiments.

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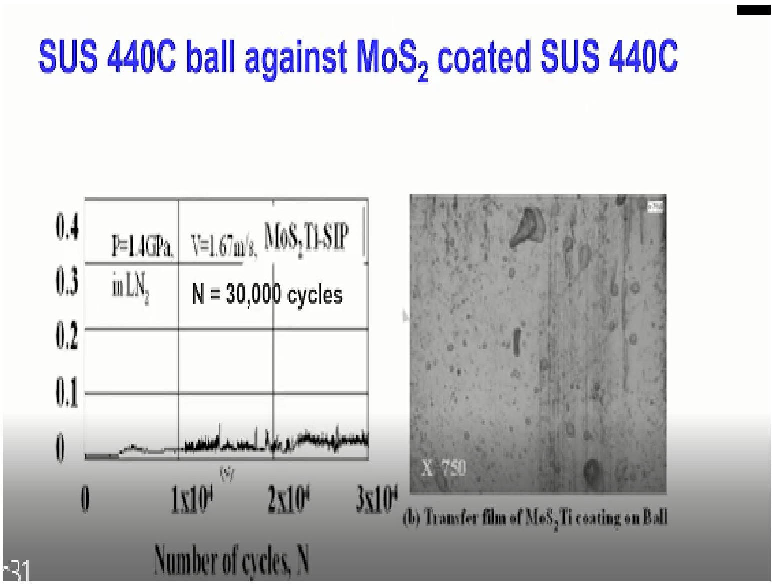

SUS 440C ball against MoS_2 coated SUS 440C

(b) Transfer film of MoS_2Ti coating on Ball

Now individually if you look at the different material combinations what you see that the most to titanium coatings which experience extremely low friction of 0.5 here we are clearly see the transfer flame on this material this is that this is the were trapped here.

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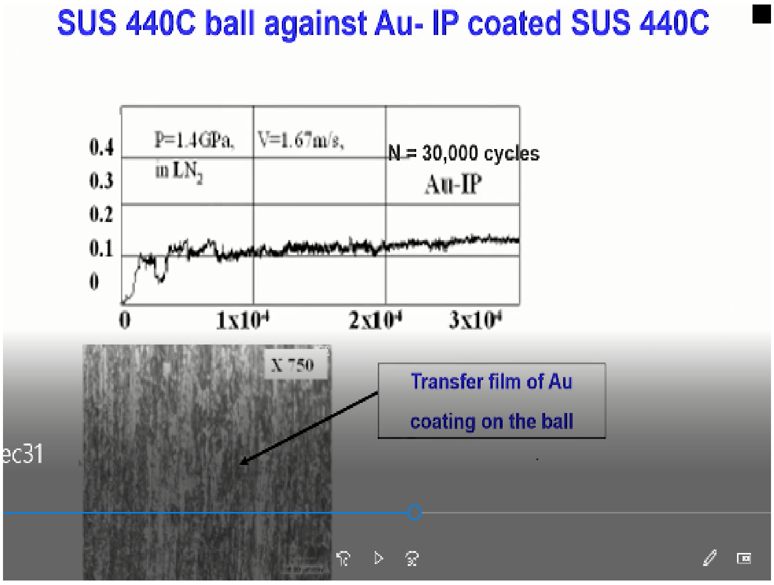

SUS 440C ball against Au- IP coated SUS 440C

Transfer film of Au coating on the ball

Similarly, formation of transfer film on gold coating on the ball is all sorts of on this particular material against a stainless steel for 440 C material.

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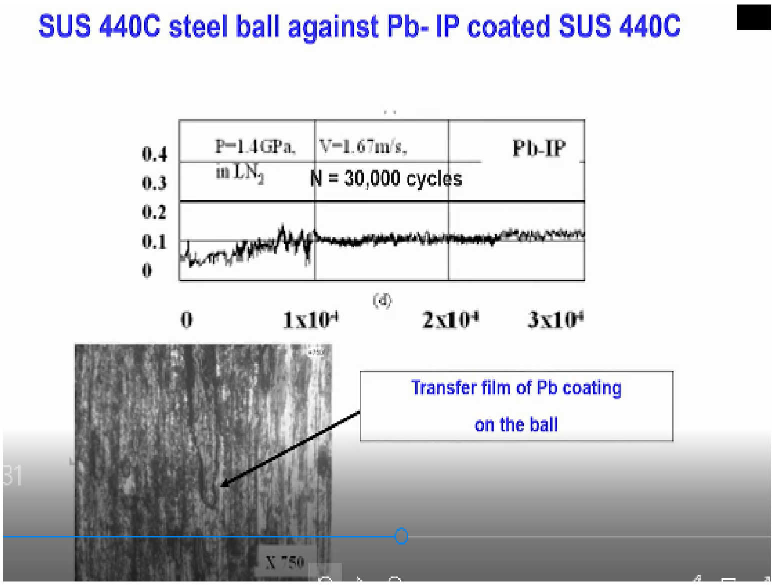

SUS 440C steel ball against Pb- IP coated SUS 440C

Transfer film of Pb coating on the ball

The same is true for the late coatings on the late coatings and it is transferred you it is transferred on the ball. So all these images are taken at certain magnifications of the ball on surfaces.

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Materials Science and Engineering A 415 (2006) 72–79

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Development of a high-speed cryogenic tribometer:
Design concept and experimental results

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Received in revised form 31 August 2005; accepted 20 September 2005

Abstract

In order to evaluate the tribological properties of ball bearings used in high-speed cryo-turbopumps of liquid rocket engines, a tribometer to operate at high sliding speed in cryogenic environment is required. In order to meet the above requirements, a new high-speed cryo-tribometer has been designed. With this new tribometer, the evaluation of friction and wear mechanisms of various self-mated materials and solid lubricant coatings under high sliding speeds (>0.5–45 m/s) with high Hertzian contact stresses (up to 2.5 GPa) in cryogenic fluid is now possible. Two additional features available with this new cryo-tribometer are: (a) a mono-ball rolling-on-disk tester under the above conditions for the analysis of rolling friction and (b) the testing of unlubricated and lubricated ball bearing (10 mm diameter bore) submerged in liquid nitrogen or liquid helium under very high-speed (36,000 rpm). An additional provision has been made for the continuous measurement of bearing frictional torque at a maximum axial load of 50N. The design details of the new tribometer are the major focus of discussion in this paper. This paper also reports the results of a very first set of experiments conducted on self-mated 440C steel and AISI 304 steel under liquid nitrogen (LN_2) immersed condition with selected testing parameters. Testing of ball bearings with Pb-IP coating at 36,000 rpm under liquid nitrogen also has been carried out. Additionally, the wear mechanisms for self-mated steel in cryogenic liquid are also discussed.

Now these are some of the results are published in Materials science and engineering channel.

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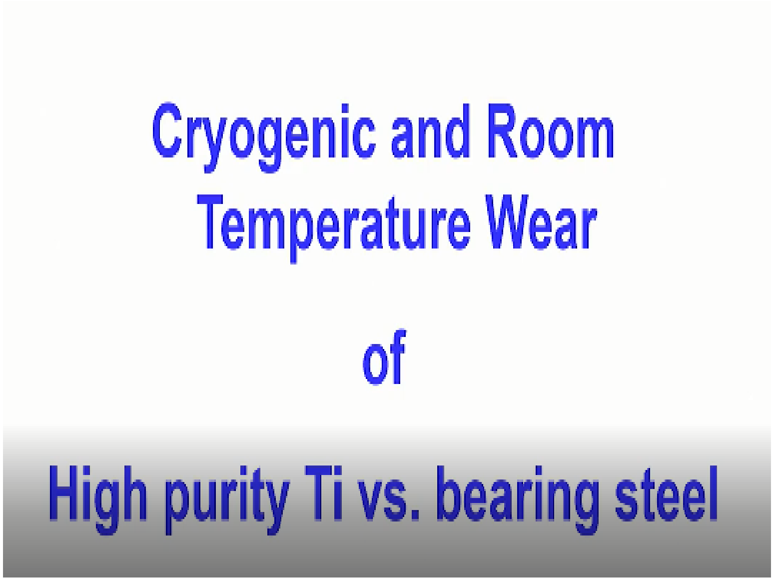

Cryogenic and Room Temperature Wear

of

High purity Ti vs. bearing steel

And now I will show you that we both have some of the reasons that we have done some of the results we have obtained with high purity titanium now this is that extremely high purified titanium and this titanium is own bearing steel.

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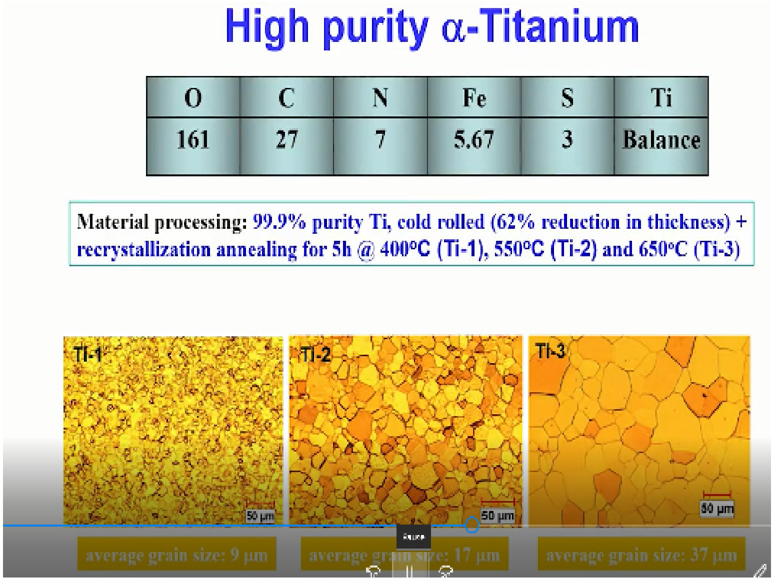

High purity α-Titanium

O	C	N	Fe	S	Ti
161	27	7	5.67	3	Balance

Material processing: 99.9% purity Ti, cold rolled (62% reduction in thickness) + recrystallization annealing for 5h @ 400°C (Ti-1), 550°C (Ti-2) and 650°C (Ti-3)

average grain size: 9 µm

average grain size: 17 µm

average grain size: 37 µm

Now by changing the heat treatment conditions in titanium what we have done. We have so these are that pure 99% or 99 9% purity titanium which is cold rolled to 62% reduction in thickness and then the same titanium is a has undergone reconstruction annulling at 5 hours at 440 degrees Celsius titanium 1500 degrees Celsius for 5 hours titanium tool so 400 degrees Celsius is this microstructure.

50 degrees Celsius is this micro structured and 650 degrees Celsius is this micro structure? So this is that this is that optical images optical microscopic images of the edged titanium surfaces now those what from metallurgy background they would clearly distinguish that this microstructure that differ in terms of the grain sizes and that is quite expected because if you increase the heat treatment temperature.

One would expect each to a corresponding change in the micro structure in terms of the length scale in terms of the grain size right. And the same thing you will notice here he had the average grain size is 9 micron and here the average size is 17 micron and here the average grain size is 37 microns so 9 17 37 micron now when we use these three different kind of three different titanium surfaces.

And we use others another mapping couple is steel and if you use these three different micro structures. This would allow us to understand what is the influence of the grain size on the wear resistance properties. Grain size has direct influence on the hardness and eel’s strength of metals so but using 3 different grain sizes 9 micron 17 micron and 37 micron one would immediately expect that these titanium 1 titanium 2 titanium 3 would have different hardness.

And would have different eel strain. So if you have different hardness particularly for metals if the wear is dominated by the abrasive wear conditions than one would expect a difference in terms of wear resistance. Let us see how our results look like in the next few slides.

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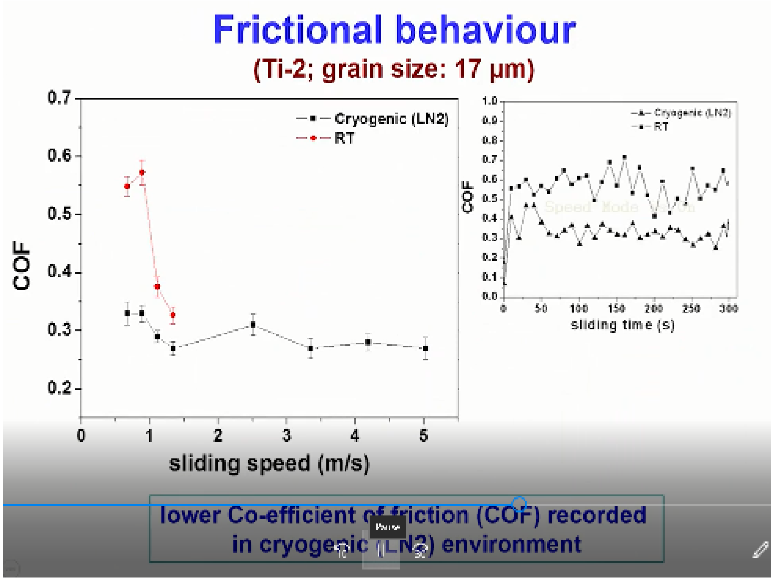

Frictional behaviour

(Ti-2; grain size: 17 μm)

lower Co-efficient of friction (COF) recorded in cryogenic (LN2) environment

Now in terms of frictional behaviour this is for the titanium tool we are comparing here room temperature versus liquid nitrogen results are different sliding speed at room temperature there is certainly higher coefficient of friction if you see that this deeper COF it is 0.5 versus 0.35. So these differences around 0.2 and these differences certainly reduced when you go up to the high sliding conditions particularly that are more than 1 meter per second.

And for cryogenic conditions we have done extended experiments up to 5 meter per second and where do you can see the coefficient of friction is somewhere around 0.28 okay. So it is certainly not very low compared to what you have seen earlier in case of stainless steel.

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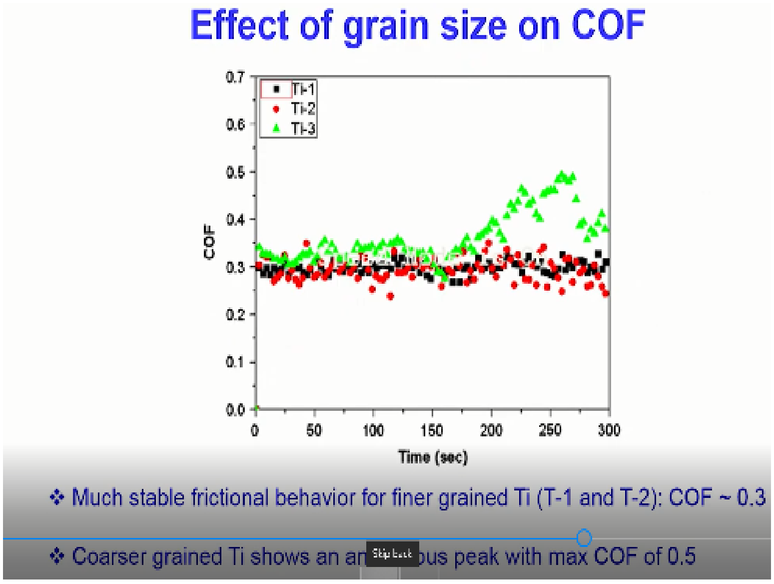

Effect of grain size on COF

- ❖ Much stable frictional behavior for finer grained Ti (T-1 and T-2): COF ~ 0.3
- ❖ Coarser grained Ti shows an an ous peak with max COF of 0.5

Now if you look at them if you look at the other materials absolutely grain size does not have any influence on coefficient of friction and that is what is not that is what is also expected.

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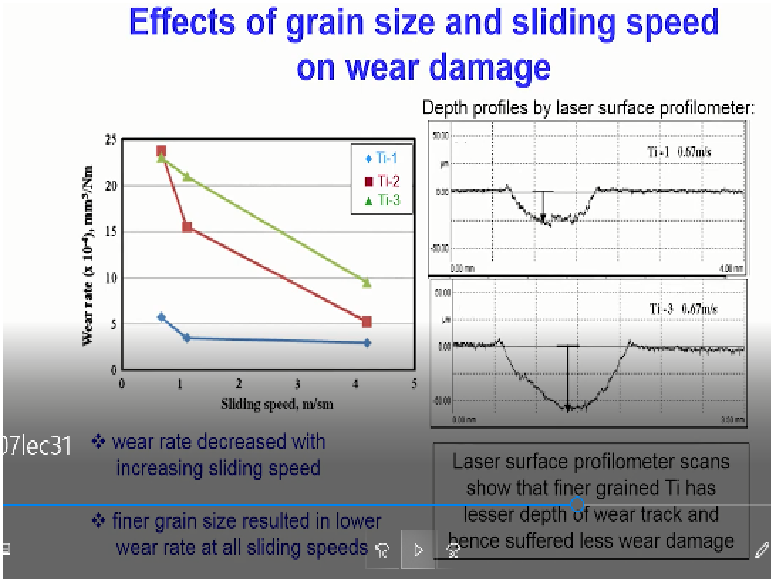

Effects of grain size and sliding speed on wear damage

Depth profiles by laser surface profilometer:

- ❖ wear rate decreased with increasing sliding speed
- ❖ finer grain size resulted in lower wear rate at all sliding speeds

Laser surface profilometer scans show that finer grained Ti has lesser depth of wear track and hence suffered less wear damage

However, if you now plot wear it as a function of sliding speed for three materials titanium 1 titanium 2 titanium 2 the sliding speed influence you see that is not much in case of the titanium 1 but titanium 2 and titanium 3 particularly this is for the titanium 3 there is a clear linear decrease in the wear rate with high with sliding speed what it means that at higher sliding speed where the decreases.

That is not only true for titanium 3 but also true for titanium 2 as well and this is the two deep stresses that what we get from the laser surface profilometer and then what is this typical depth like you know it is close to 50 micron depth which is very significant wear.

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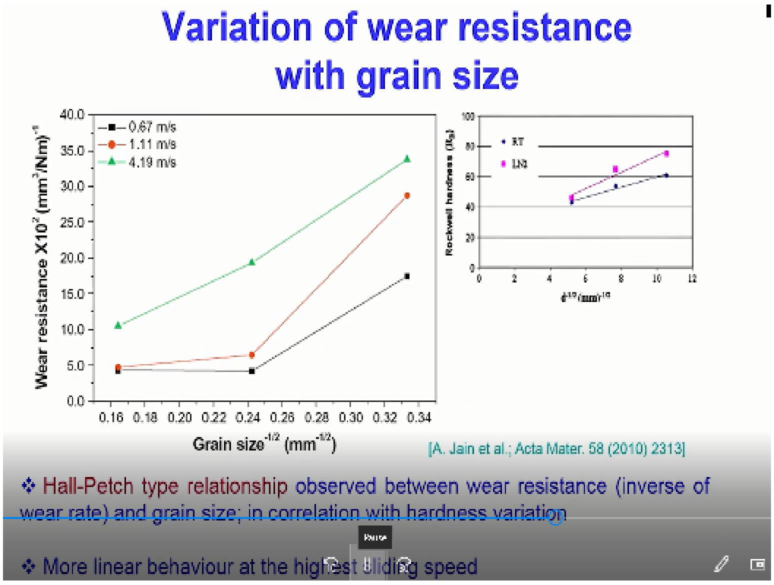

Variation of wear resistance with grain size

[A. Jain et al.; Acta Mater. 58 (2010) 2313]

- ❖ Hall-Petch type relationship observed between wear resistance (inverse of wear rate) and grain size; in correlation with hardness variation
- ❖ More linear behaviour at the highest sliding speed

Now this is quite an interesting observations now those who are from metallurgy background they know sigma = eel strength for molecules and materials sigma = sigma 0+K d to the power -1/2 where sigma Y is the yield strength of the polycrystalline material sigma 0 is the yield strength of single crystals because from this you can clearly see that when d= infinity sigma y= sigma 0.

So when d=infinity that means when grains is extremely large and if it grains are extremely large you can consider a single crystal materials and in case of single crystals your poly crystal be consumed with strength so and T is your grain size and K is the Hall-Petch coefficient. So this is the famous Hall-Petch equation which most mythologists they are they are fully aware of.

Now in line with this argument that since yield strength also has a proportional relationship to d to the power -1/2 we tried to explore that further wear resistance which is nothing but 1/wear rate has similar propionate relationship with grain size to the power -1/2 and what do you notice here? that they have very linear relationship at 4.19 meter per second that is very higher sliding speed this relationship also is valid or at 1.11 meter per second and 0.67 meter per second.

But this particular linear relationship between wear resistance grain size d power -1/2 for most significant in only at the highest sliding speed because in this particular two data points you do not see much increase. But as you go to the higher value of grain size dance d to the power -1/2 we do notice that wear resistance increases. So this is one of the things that i have mentioned at the beginning of this was experimental of when i started showing the experimental results with titanium.

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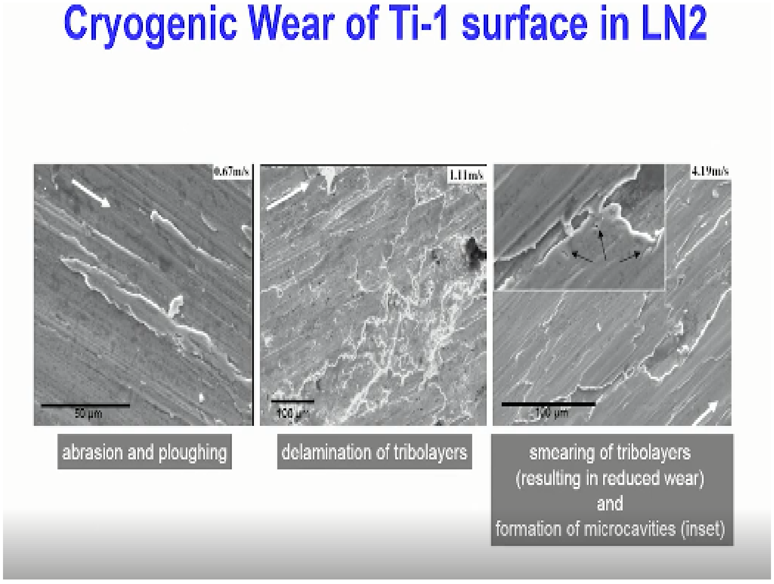

Cryogenic Wear of Ti-1 surface in LN2

abrasion and ploughing

delamination of tribolayers

smearing of tribolayers
(resulting in reduced wear)
and
formation of microcavities (inset)

So titanium 1 surface if you see this abrasion and ploughing that you can see and there is also de lamination of the tribolayers that is also absorbed on this particular titanium 1 surface after sliding with liquid nitrogen.

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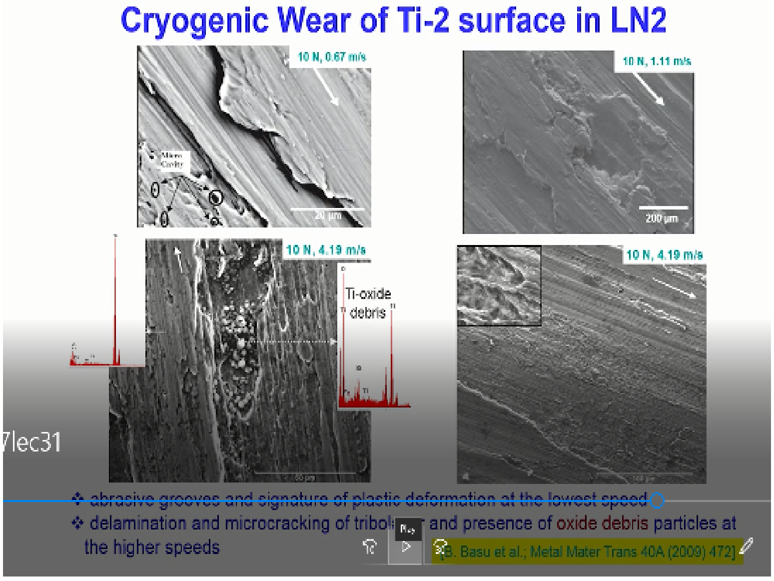

Cryogenic Wear of Ti-2 surface in LN2

- abrasive grooves and signature of plastic deformation at the lowest speed
- delamination and microcracking of tribol and presence of oxide debris particles at the higher speeds

[B. Basu et al.; Metal Mater Trans 40A (2009) 472]

Other interesting observations that we have observed that in case of titanium 2 at a they particular grain size we see that varies brittle kind of like of titanium dioxide titanium oxide wear particles. So these titanium oxide wear particles are trapped in these particular groups and once they are trapped in these particular groups it would lead to the hard body wear. And other interesting abrasion.

That we have observed that there is an extensive ductile deformation which leads to at some part shear leap kind of behaviour and this shear leap essentially leads to plastic deformation is the signature of the extensive plastic deformation at the lowest speed.

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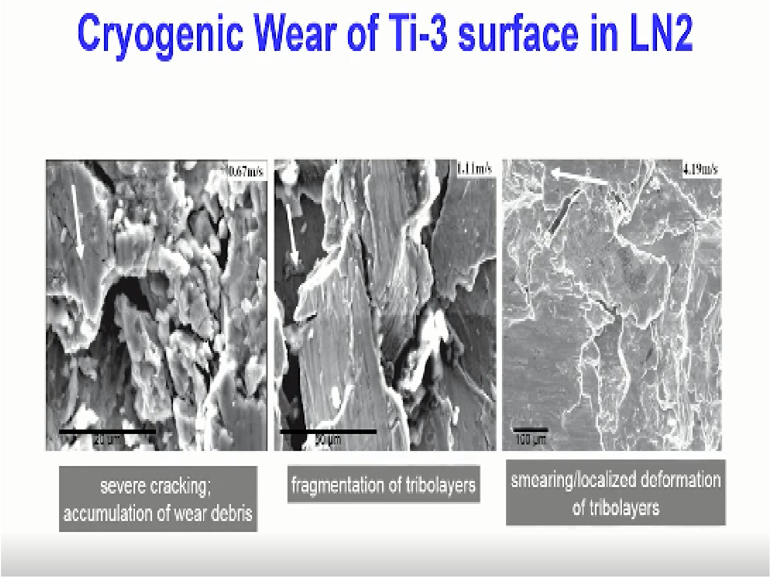

Cryogenic Wear of Ti-3 surface in LN2

severe cracking; accumulation of wear debris

fragmentation of tribolayers

smearing/localized deformation of tribolayers

So we have done a systematic observation of the micro structure just to observe spalling and cracking severe cracking in this case of titanium.

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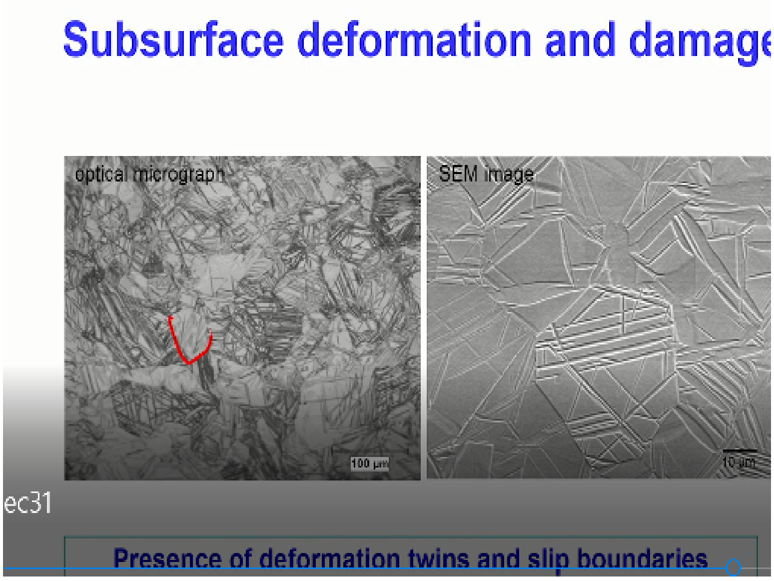

Subsurface deformation and damag

Presence of deformation twins and slip boundaries

Now from mythological point of view what is more important for us to show was to see the subsurface deformation and damage and what you notice here that this is the peer particularly case for one of the titanium materials and if you carefully edge the surfaces we do see in individual grains lot of twin. So these twins are one of the deformation mode for metals there are two type of deformation mode one is sleep.

And one is twinning so these twinning if you see this is the large cranes has grain size like you know 40 micron or so and you see these twins they normally extend from one grain to anna that unless they impinge on another growing twins. So for example these twin cannot extend to the other end of the cranium edge. Because it is actually interacting with one of the longer twins in the micro on this specific lens.

So this twinning is one of the more of the deformation in case of the what we observed in the sub surface deformation in this particular case.

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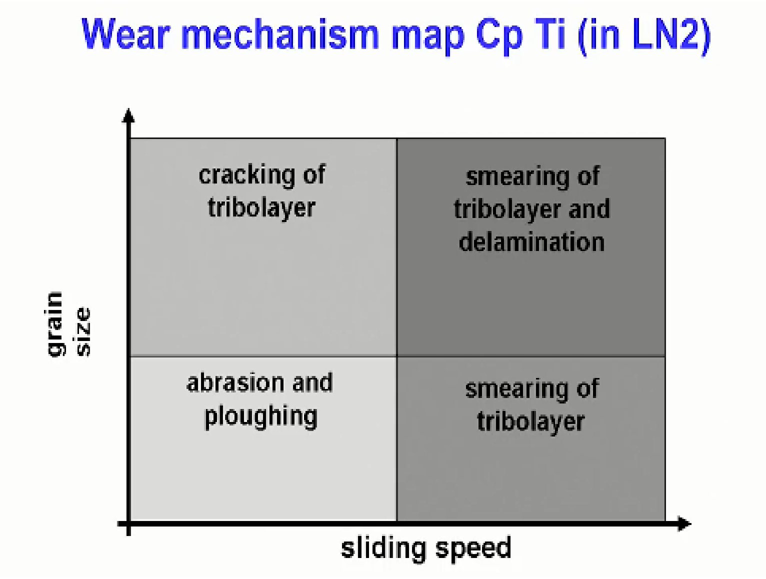

Wear mechanism map Cp Ti (in LN2)

Now if you look at this this particular wear mechanism map where we have shown the grain size versus sliding speed. So what we are seeing that at very low very large grain size it is the cracking of tribal layer whereas at various high in the sliding speed it is smearing of tribolayer which is very important.

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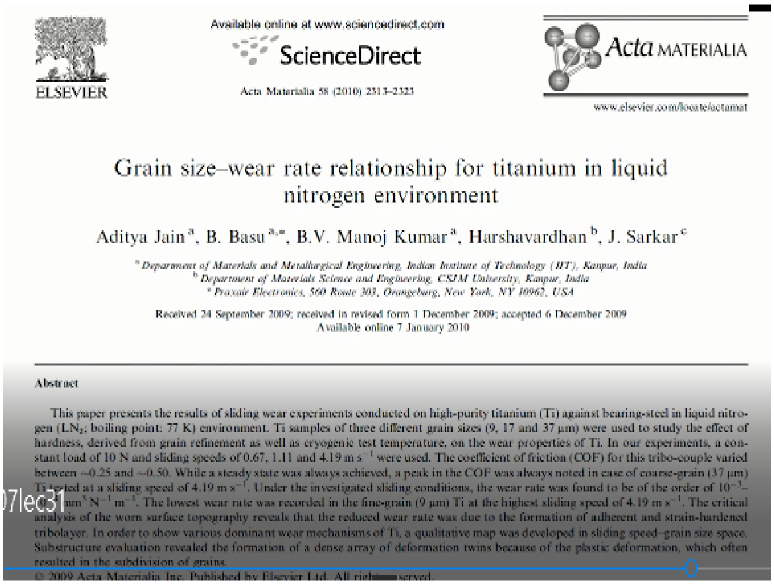

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Acta Materialia 58 (2010) 2313–2323

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Grain size–wear rate relationship for titanium in liquid nitrogen environment

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Received 24 September 2009; received in revised form 1 December 2009; accepted 6 December 2009
Available online 7 January 2010

Abstract

This paper presents the results of sliding wear experiments conducted on high-purity titanium (Ti) against bearing-steel in liquid nitrogen (LN_2; boiling point: 77 K) environment. Ti samples of three different grain sizes (9, 17 and 37 μm) were used to study the effect of hardness, derived from grain refinement as well as cryogenic test temperature, on the wear properties of Ti. In our experiments, a constant load of 10 N and sliding speeds of 0.67, 1.11 and 4.19 m s^{-1} were used. The coefficient of friction (COF) for this tribo-couple varied between ~0.25 and ~0.50. While a steady state was always achieved, a peak in the COF was always noted in case of coarse-grain (37 μm) Ti tested at a sliding speed of 4.19 m s^{-1}. Under the investigated sliding conditions, the wear rate was found to be of the order of 10^{-3}–[illegible] mm^3 N^{-1} m^{-1}. The lowest wear rate was recorded in the fine-grain (9 μm) Ti at the highest sliding speed of 4.19 m s^{-1}. The critical analysis of the worn surface topography reveals that the reduced wear rate was due to the formation of adherent and strain-hardened tribolayer. In order to show various dominant wear mechanisms of Ti, a qualitative map was developed in sliding speed–grain size space. Substructure evaluation revealed the formation of a dense array of deformation twins because of the plastic deformation, which often resulted in the subdivision of grains.

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So this was published in Acta Materialia which is one of the flagship journals in the field.

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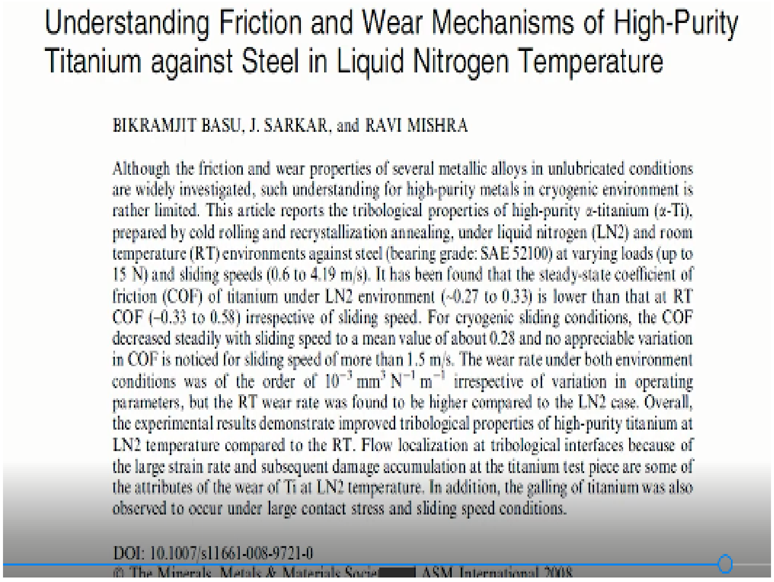

Understanding Friction and Wear Mechanisms of High-Purity Titanium against Steel in Liquid Nitrogen Temperature

BIKRAMJIT BASU, J. SARKAR, and RAVI MISHRA

Although the friction and wear properties of several metallic alloys in unlubricated conditions are widely investigated, such understanding for high-purity metals in cryogenic environment is rather limited. This article reports the tribological properties of high-purity α-titanium (α-Ti), prepared by cold rolling and recrystallization annealing, under liquid nitrogen (LN2) and room temperature (RT) environments against steel (bearing grade: SAE 52100) at varying loads (up to 15 N) and sliding speeds (0.6 to 4.19 m/s). It has been found that the steady-state coefficient of friction (COF) of titanium under LN2 environment (~0.27 to 0.33) is lower than that at RT COF (~0.33 to 0.58) irrespective of sliding speed. For cryogenic sliding conditions, the COF decreased steadily with sliding speed to a mean value of about 0.28 and no appreciable variation in COF is noticed for sliding speed of more than 1.5 m/s. The wear rate under both environment conditions was of the order of 10^{-3} mm^3 N^{-1} m^{-1} irrespective of variation in operating parameters, but the RT wear rate was found to be higher compared to the LN2 case. Overall, the experimental results demonstrate improved tribological properties of high-purity titanium at LN2 temperature compared to the RT. Flow localization at tribological interfaces because of the large strain rate and subsequent damage accumulation at the titanium test piece are some of the attributes of the wear of Ti at LN2 temperature. In addition, the galling of titanium was also observed to occur under large contact stress and sliding speed conditions.

DOI: 10.1007/s11661-008-9721-0
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The other one we are published in metalogical transaction say.

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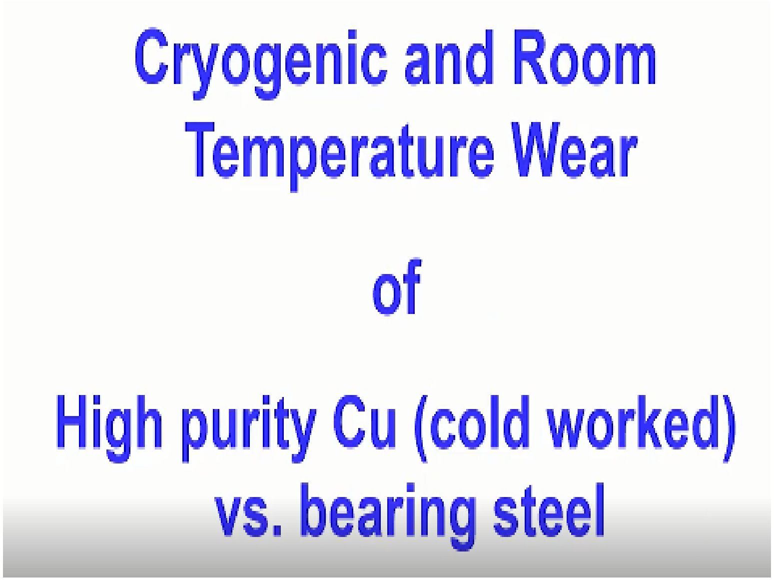

Cryogenic and Room Temperature Wear

of

High purity Cu (cold worked) vs. bearing steel

So quickly few more slides on copper so titanium is one of the harder material copper is relatively software materials so we have done also a series of experiments cryogenic sliding where experiments on copper and what you see here this with these particular copper is you can high purity copper which is cold work

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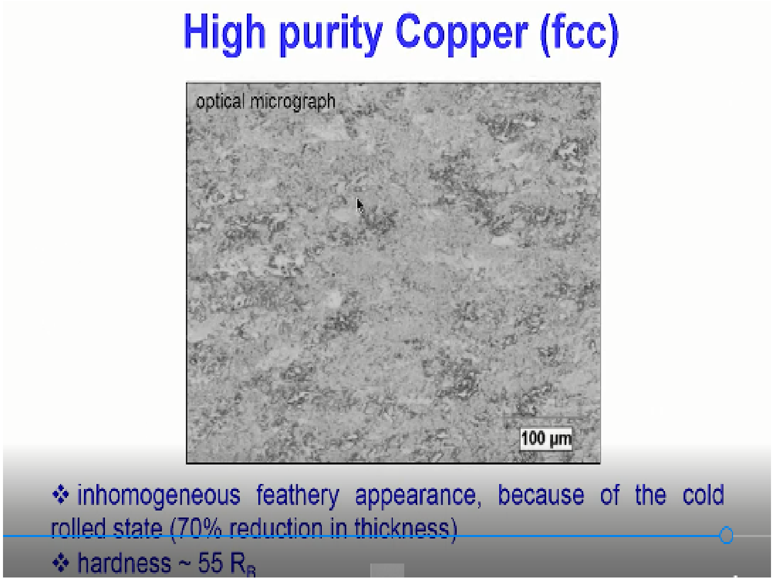

High purity Copper (fcc)

- inhomogeneous feathery appearance, because of the cold rolled state (70% reduction in thickness)
- hardness ~ 55 R_B

And in these cold work materials it is fcc copper you can see that in homogeneous feathery appearance because of the cold rolled state hardness is rock well B it is not Rockwell C.

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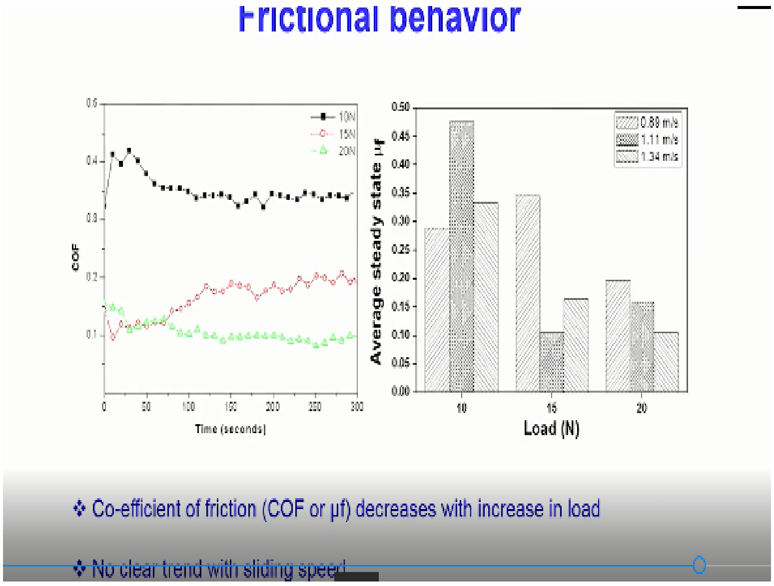

Frictional behavior

- Co-efficient of friction (COF or µf) decreases with increase in load
- No clear trend with sliding speed

It is 55 in the Rockwell B hardness scale now we have valued that load 10 newton to 20 newton and you can see are the higher load that means higher contact stress cultural friction drops and it is going to 0.1 and d it s what are the high lower load the friction is fairly high it is around 0.35.

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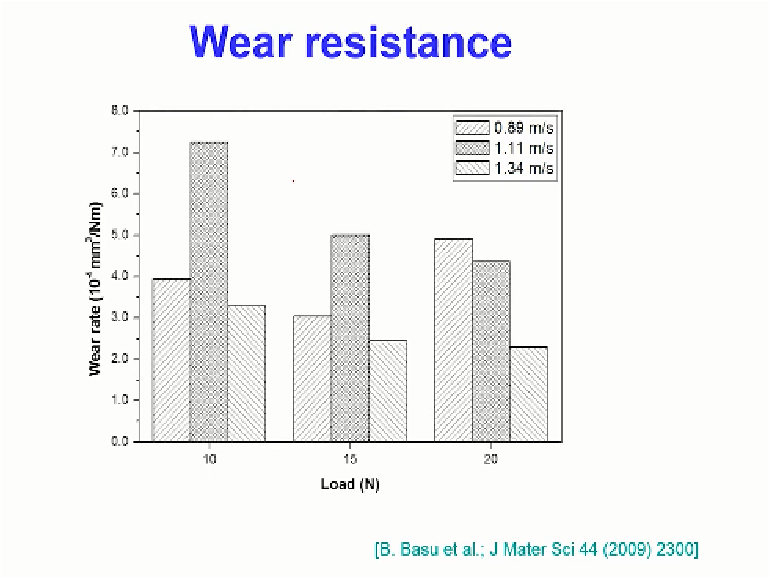

Wear resistance

[B. Basu et al.; J Mater Sci 44 (2009) 2300]

So in terms of the wear it if you go to the lower lord and then higher sliding speed again that that compared to the lower compared to the lower load that wear rate decreases but decreased may not be very significant. But certainly there is a trend of decreasing particularly if you look at this one there is trend in decrease with the increase in the lord as far as the wire resistance is concerned.

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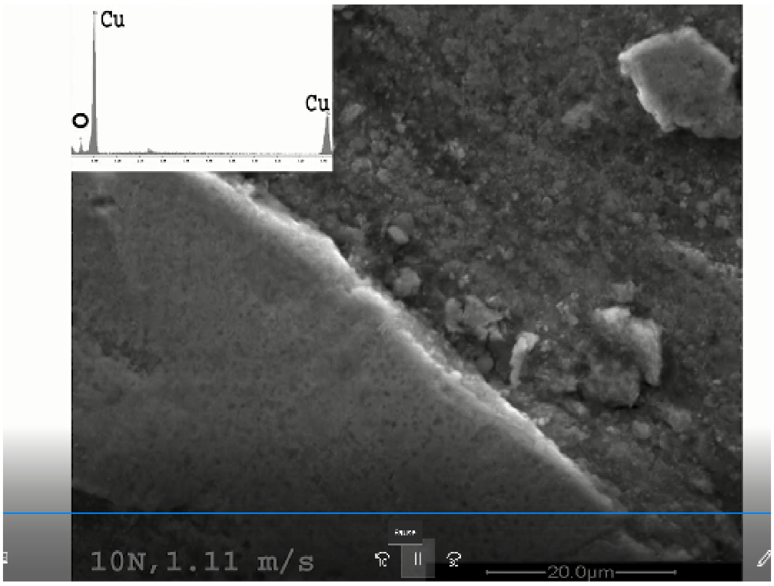

So the copper also again it undergoes a oxidation or the wear surface and you can see here there are copper oxide particles which are also formed on the materials. So this is this copper and these copper this is a this is the sliding wear track and the sliding wear track you can see that the in the end in the sliding wear track you can very clearly notice that there are signs of deformation ploughing in this region you can see like farmers will plough the fields.

Similarly, during the sliding wear the harder materials the plough the software materials and this is one of the mechanism of the wear what we have observed in case of copper.

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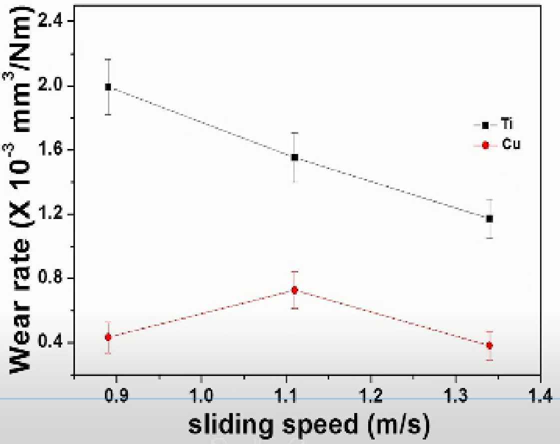
Wear of Ti (Ti-2) vis-a-vis Cu (identical hardness)



Now if you compare these two materials titanium and copper what do you notice the titanium case? the sliding speed where it decreases in copper in the case of titanium copper we do not see any very clear trend.

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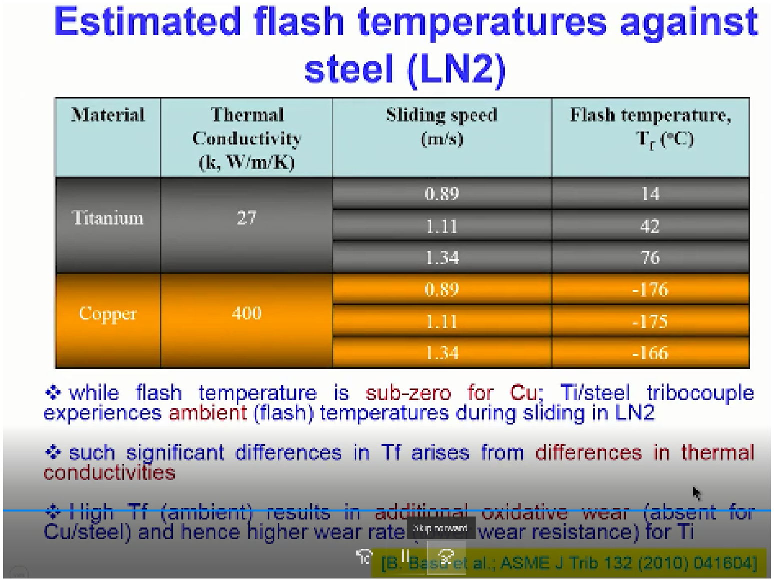

Estimated flash temperatures against steel (LN2)

Material	Thermal Conductivity (k, W/m/K)	Sliding speed (m/s)	Flash temperature, T_f (°C)
Titanium	27	0.89	14
		1.11	42
		1.34	76
Copper	400	0.89	-176
		1.11	-175
		1.34	-166

- while flash temperature is sub-zero for Cu; Ti/steel tribocouple experiences ambient (flash) temperatures during sliding in LN2
- such significant differences in Tf arises from differences in thermal conductivities
- High Tf (ambient) results in additional oxidative wear (absent for Cu/steel) and hence higher wear rate (lower wear resistance) for Ti

[B. Basu et al.; ASME J Trib 132 (2010) 041604]

In terms of the flash temperature calculations what we have observed that in case of the copper because of the high thermal conductivity flash temperature is fairly low. But titanium flash temperatures above 0 degrees Celsius or even above room temperature for example here and here in titanium. So because of the difference in the flash temperature the wear rate what you see in case of cooper is fairly low.

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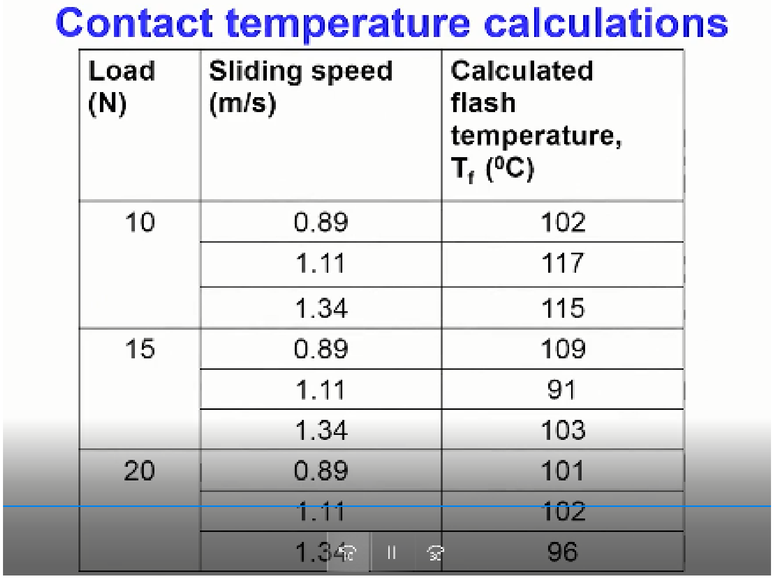

Contact temperature calculations

Load (N)	Sliding speed (m/s)	Calculated flash temperature, T_f (°C)
10	0.89	102
	1.11	117
	1.34	115
15	0.89	109
	1.11	91
	1.34	103
20	0.89	101
	1.11	102
	1.34	96

So these are the actual calculated flash temperature for this for this specific tribocouple.

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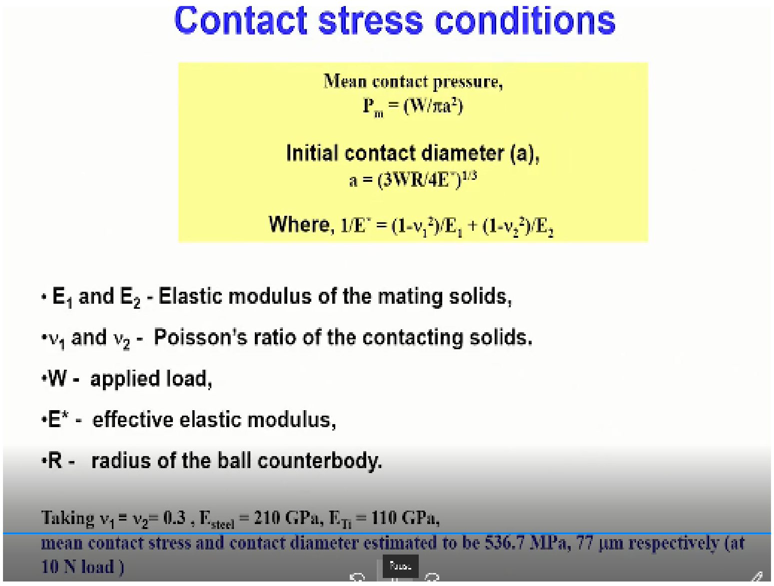

Contact stress conditions

Mean contact pressure,
$P_m = (W/\pi a^2)$

Initial contact diameter (a),
$a = (3WR/4E^*)^{1/3}$

Where, $1/E^* = (1-\nu_1^2)/E_1 + (1-\nu_2^2)/E_2$

- E_1 and E_2 - Elastic modulus of the mating solids,
- ν_1 and ν_2 - Poisson's ratio of the contacting solids.
- W - applied load,
- E* - effective elastic modulus,
- R - radius of the ball counterbody.

Taking $\nu_1 = \nu_2 = 0.3$, $E_{steel} = 210$ GPa, $E_{Ti} = 110$ GPa,
mean contact stress and contact diameter estimated to be 536.7 MPa, 77 μm respectively (at 10 N load)

And we have also calculated the contact stress in this particular case by taking the titanium as hundred 110 giga pascal and we are getting context as 537 megapascal.

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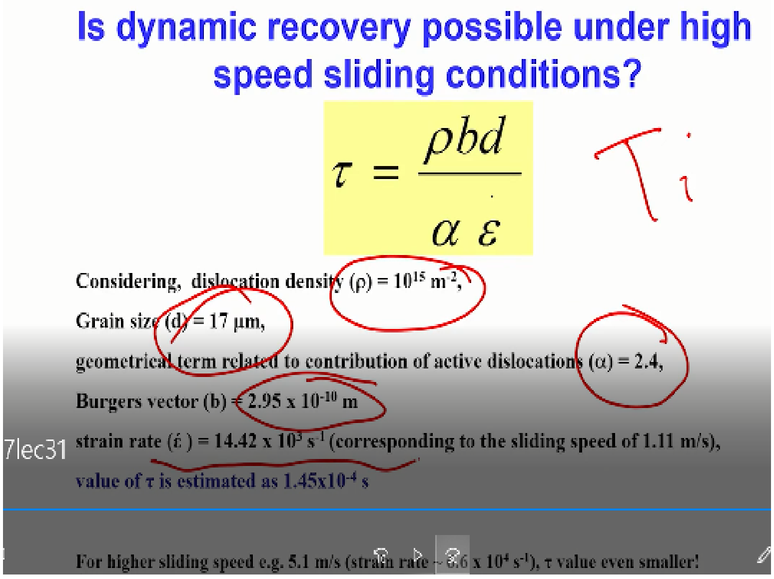

Is dynamic recovery possible under high speed sliding conditions?

$$\tau = \frac{\rho b d}{\alpha \dot{\varepsilon}}$$

Ti

Considering, dislocation density (ρ) = 10^{15} m^{-2},
Grain size (d) = 17 μm,
geometrical term related to contribution of active dislocations (α) = 2.4,
Burgers vector (b) = 2.95 x 10^{-10} m
strain rate ($\dot{\varepsilon}$) = 14.42 x 10^3 s^{-1} (corresponding to the sliding speed of 1.11 m/s),
value of τ is estimated as 1.45x10^{-4} s

For higher sliding speed e.g. 5.1 m/s (strain rate ~ 6.6 x 10^4 s^{-1}), τ value even smaller!

Now we wanted to see that with a dynamic recovery is one of the possibility under high speed sliding condition and for that we have calculated that watch would be the dislocation. What is the dislocation density we considered what is the grain size increase of Titanium and what is the burges vector of the dislocations and these are the fraction of the active based dislocations?

And what is the strain rate corresponding to the sliding speed of 1.1 meter per second. So what we have found that value of tau shear stress is around 1.45 10 to the power you know tau this is this value of tau in this particular equation is 1.45 10 to the power -4 seconds where it is at the rate of it is even less than a millisecond. So for higher tau value will be smaller.

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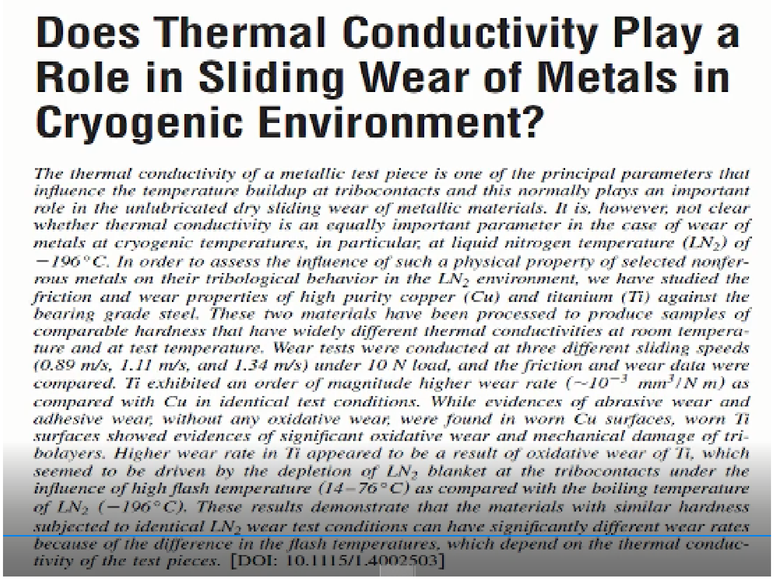

Does Thermal Conductivity Play a Role in Sliding Wear of Metals in Cryogenic Environment?

The thermal conductivity of a metallic test piece is one of the principal parameters that influence the temperature buildup at tribocontacts and this normally plays an important role in the unlubricated dry sliding wear of metallic materials. It is, however, not clear whether thermal conductivity is an equally important parameter in the case of wear of metals at cryogenic temperatures, in particular, at liquid nitrogen temperature (LN_2) of −196°C. In order to assess the influence of such a physical property of selected nonferrous metals on their tribological behavior in the LN_2 environment, we have studied the friction and wear properties of high purity copper (Cu) and titanium (Ti) against the bearing grade steel. These two materials have been processed to produce samples of comparable hardness that have widely different thermal conductivities at room temperature and at test temperature. Wear tests were conducted at three different sliding speeds (0.89 m/s, 1.11 m/s, and 1.34 m/s) under 10 N load, and the friction and wear data were compared. Ti exhibited an order of magnitude higher wear rate ($\sim 10^{-3}$ $mm^3/N\ m$) as compared with Cu in identical test conditions. While evidences of abrasive wear and adhesive wear, without any oxidative wear, were found in worn Cu surfaces, worn Ti surfaces showed evidences of significant oxidative wear and mechanical damage of tribolayers. Higher wear rate in Ti appeared to be a result of oxidative wear of Ti, which seemed to be driven by the depletion of LN_2 blanket at the tribocontacts under the influence of high flash temperature (14–76°C) as compared with the boiling temperature of LN_2 (−196°C). These results demonstrate that the materials with similar hardness subjected to identical LN_2 wear test conditions can have significantly different wear rates because of the difference in the flash temperatures, which depend on the thermal conductivity of the test pieces. [DOI: 10.1115/1.4002503]

So this work is published in wear of metals in cryogenic. Thank you.