Friction and Wear of Materials: Principles and Case Studies

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Lecture – 25
Fretting wear of SiAlON Ceramics

Welcome back to this NPTEL lecture. Let me switch the gear from tribological studies of bioceramics and composites to fretting and wear properties of other non-oxide ceramics, SiAlON oxides, sialon ceramics.

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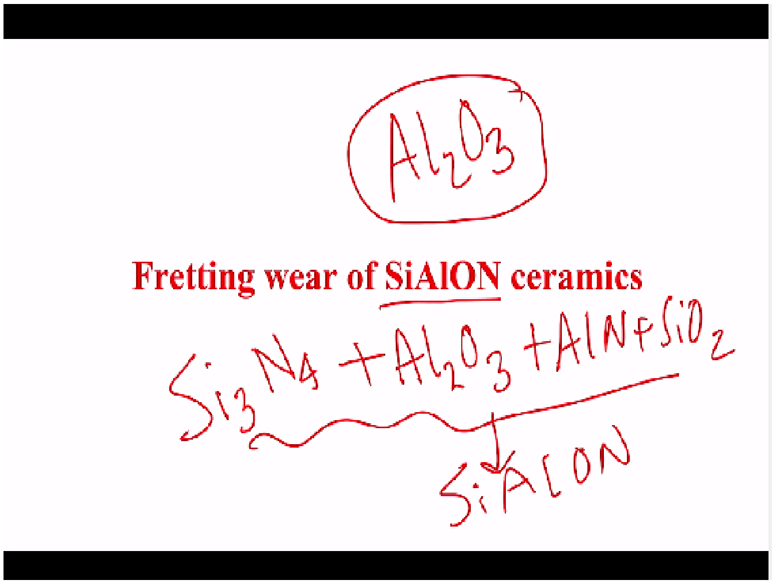

Al_2O_3

Fretting wear of SiAlON ceramics

$Si_3N_4 + Al_2O_3 + AlN + SiO_2$

$\downarrow$

SiAlON

Although sialon ceramics can be used for biomedical applications. There is a company based in US who is to develop this silicon nitride ceramics for biomedical applications. But however, it has not penetrated so far very deeply into the biomedical device industry. And I am not aware of too many companies who are developing silicon aluminium oxynitride based materials for biomedical applications.

Therefore, I would safely consider silicon oxynitride, silicon aluminium oxynitride based materials as in important class of ceramic materials which are currently being used for many of the structural applications and engineering applications. But certainly not for biomedical applications. Now what is silicon aluminium oxynitride? Those who are not from ceramics background, most of you know that silicon nitride Si3N4 is one of the popular ceramic materials.

But alumina is a kind of a model ceramic materials. Now silicon nitride can form solid solution when you use alumina, aluminium nitride and silica. And if you form this 4 component system, then we, the solid solution that form, so it is called silicon aluminium oxynitride. So silicon aluminium oxynitride is essentially a solid solution of silicon nitride with other oxide or nitride matted phases.

So those who are from metallurgy background, they know any material when exist in the solid solution phase, typically it has better mechanical properties, better hardness, better strength properties. A popular solid solution alloy is steel. Steel is a solid solution of iron and carbon. If there is no carbon, you cannot call it a steel. It is a pure iron. So similarly and why steel? Because pure iron is very soft but when you have carbon in the solid solution with iron, certainly that steel has a better hardness and strength properties.

Now in line with the similar argument in case of the ceramic alloys like silicon aluminium oxynitride also has better strength and better fracture toughness properties than silicon nitride.

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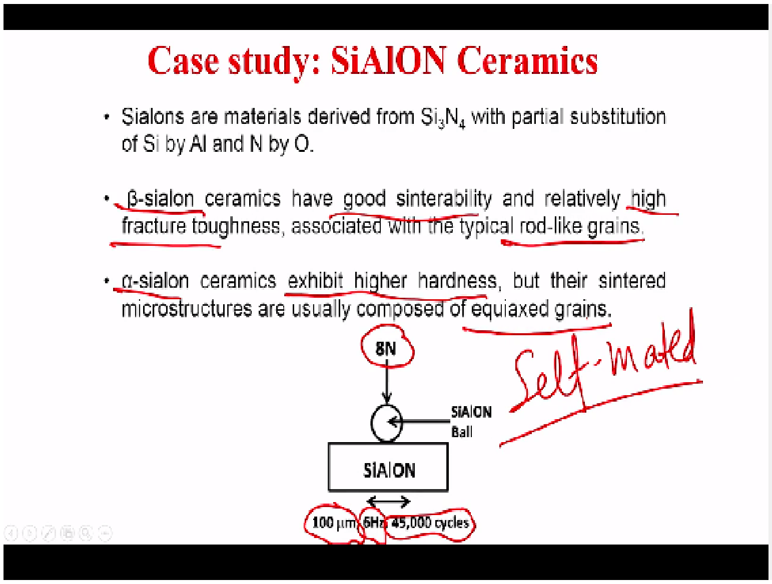

Case study: SiAlON Ceramics

- Sialons are materials derived from Si_3N_4 with partial substitution of Si by Al and N by O.
- β-sialon ceramics have good sinterability and relatively high fracture toughness, associated with the typical rod-like grains.
- α-sialon ceramics exhibit higher hardness, but their sintered microstructures are usually composed of equiaxed grains.

Therefore, in this particular case study, we will try to see that, so whether we can measure this wear resistance properties, how the wear resistance properties also depend on the microstructure of these material. So wear resistance properties mostly are measured using the sialon as a ball. So essentially we are discussing here self-mated couple like sialon against sialon0 ball. And then load that we have chosen, it is around 8 Newton.

This relative displacement at the contact is 100 micron, 6 Hz is the frequency and typically 45,000 is the cycles. Now silicon aluminium oxynitride materials also there are 2 polymorphs. One is that beta sialon, another one is alpha sialon. And beta sialon and alpha sialon is in line with the same argument because baseline material that is silicon nitride, they also have 2 polymorphs.

Alpha silicon nitride and beta silicon nitride. So similarly alpha sialon and beta sialon ceramics, both of them have similar properties. For example, beta sialon has good sinterability and relatively high fracture toughness. And these are associated with the rod-like grains. Alpha sialon on the other hand has higher hardness but their sintered microstructures typically composed of the equiaxed grains.

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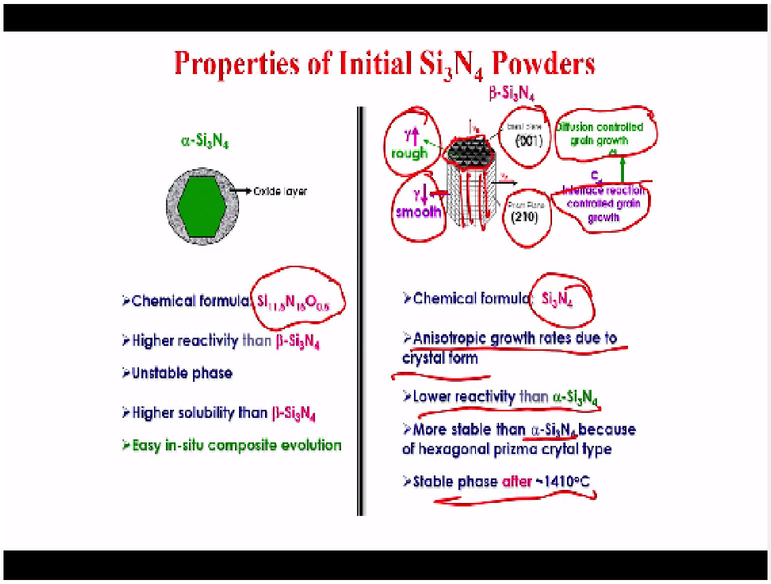

Properties of Initial Si_3N_4 Powders

- Chemical formula $Si_{11.5}N_{15}O_{0.5}$
- Higher reactivity than β-Si_3N_4
- Unstable phase
- Higher solubility than β-Si_3N_4
- Easy in-situ composite evolution

- Chemical formula Si_3N_4
- Anisotropic growth rates due to crystal form
- Lower reactivity than α-Si_3N_4
- More stable than α-Si_3N_4 because of hexagonal prizma crytal type
- Stable phase after ~1410°C

Now this is little bit more details of the alpha sialon and beta sialon based materials. So you have that initial silicon nitride powders, alpha silicon nitride which is the typical composition of Si 11.5 N 15 oxygen 0.5 and it has also higher reactivity than beta sialon with silicon nitride. It is clearly an unstable phase. It is higher solubility than beta silicon nitride. Now whereas in case of beta silicon nitride, it has a chemical formula Si3N4 that is stoichiometric formula.

It is anisotropic growth rates due to crystal form. These anisotropic growth you can see very clearly. This is your basal plane of the hexagonal crystal, right. And this is the anisotropic growth rate here. This has prism plane of 210 and basal plane of 0001, 0001 plane. So along C axis, you have interface reaction controlled grain growth. And on a axis, you have a diffusion controlled grain growth.

And you have a difference in the surface energy like gamma is decreased along this direction which is smooth but on the top basal plane, because it is a rough surface, it has more interfacial energy. And compared to alpha silicon nitride that is the counter path, it has a lower reactivity and more stable and it is a stable phase after 1410 degree Celsius.

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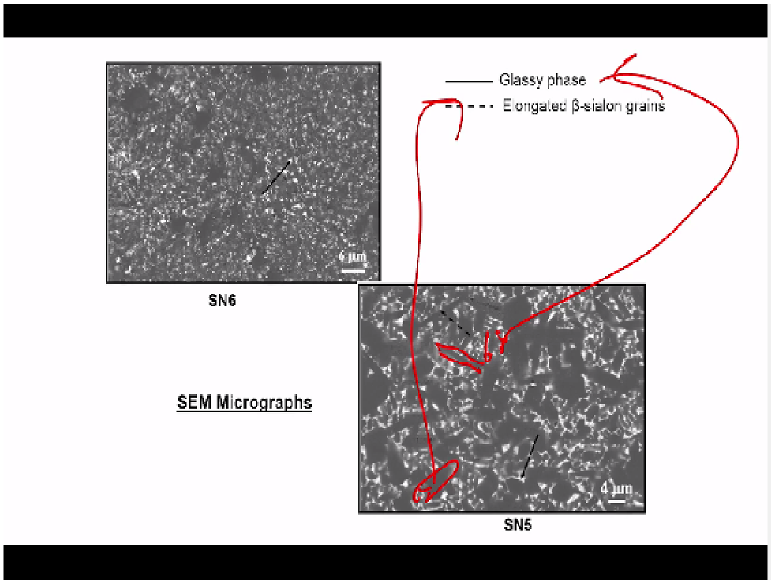

Now in this beta silicon nitride or elongated beta sialon grains, there are different by changing the composition of the starting powders. Essentially you can get different phase fractions in these particular silicon aluminium oxynitride phases or sialon phases. What you see in the bright contrast, these are like residual glass phases. And some of the cases you will see that there is elongated crystals. So these are the elongated beta silicon nitride, beta sialon grains.

So essentially the microstructure is composed of residual glass phases and also elongated beta sialon grains. And these elongated beta sialon grains are very important because this beta sialon grains, they have the potential to reduce the crack growth resistance on the fracture surfaces. And if the crack growth resistance is reduced then what would happen, that this particular microstructure may have better fracture resistance properties as well, okay.

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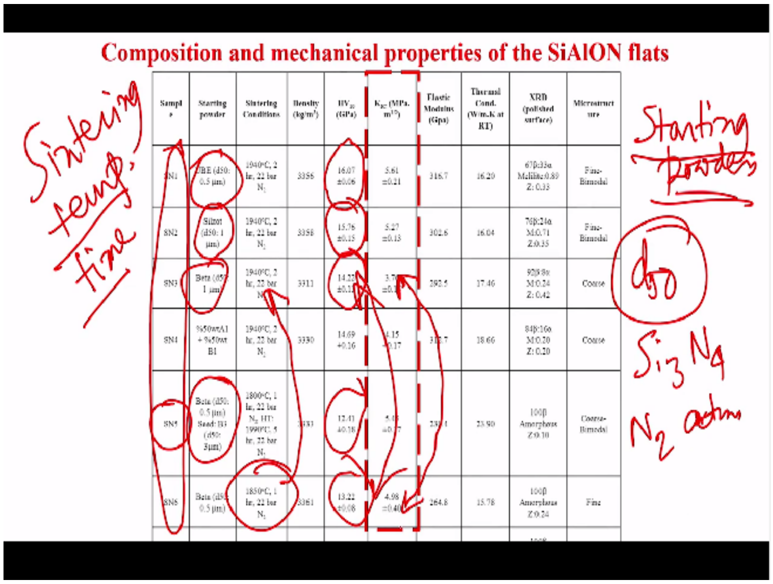

Composition and mechanical properties of the SiAlON flats

Sample	Starting powder	Sintering Conditions	Density (kg/m³)	HV_{10} (GPa)	K_{IC} (MPa. m$^{1/2}$)	Elastic Modulus (Gpa)	Thermal Cond. (W/m.K at RT)	XRD (polished surface)	Microstructure
SN1	UBE (d50: 0.5 µm)	1940°C, 2 hr, 22 bar N_2	3356	16.07 ±0.06	5.61 ±0.21	316.7	16.20	67β:33α Melilite:0.89 Z: 0.33	Fine-Bimodal
SN2	Silzot (d50: 1 µm)	1940°C, 2 hr, 22 bar N_2	3358	15.76 ±0.15	5.27 ±0.13	302.6	16.04	76β:24α M:0.71 Z:0.35	Fine-Bimodal
SN3	Beta (d50: 1 µm)	1940°C, 2 hr, 22 bar N_2	3311	14.22 ±0.1[illegible]	3.70 ±0.1[illegible]	292.5	17.46	92β:8α M:0.24 Z: 0.42	Coarse
SN4	%50wtA1 + %50wt B1	1940°C, 2 hr, 22 bar N_2	3330	14.69 ±0.16	4.15 ±0.17	31[illegible].7	18.66	84β:16α M:0.20 Z: 0.20	Coarse
SN5	Beta (d50: 0.5 µm) Seed: B3 (d50: 3µm)	1800°C, 1 hr, 22 bar N_2 HT: 1990°C, 5 hr, 22 bar N_2	[illegible]	12.41 ±0.18	5.[illegible] ±0.[illegible]	23[illegible]	23.90	100β Amorphous Z:0.10	Coarse-Bimodal
SN6	Beta (d50: 0.5 µm)	1850°C, 1 hr, 22 bar N_2	3361	13.22 ±0.08	4.98 ±0.40	264.8	15.78	100β Amorphous Z:0.24	Fine

Sintering temp, time

Starting Powders

d50

Si_3N_4

N_2 atm

Now if you look at this particular table, let me spend little more time. Now what I am trying to emphasize here again those for the non-ceramic background people, there are 2 things. One is that starting powder's composition and one is the sintering conditions, sintering temperature, time and so on.

By changing these 2 parameters like starting powder, sintering temperature and time, it is possible to obtain a range of different microstructures, thereby one can also obtain a range of properties in terms of harness properties, in terms of fracture toughness properties and so on. So here is the example. For ease of comparison, I have specified it as SN1 to SN6 or some different grades and then we have used that Ube powder that is Japanese powder of silicon nitride which is a d50 that is that average particle sizes.

So d50 essentially is average particle sizes of around 0.5 micron and if you sinter them at 1940 degree Celsius for 2 hours at 22 bar nitrogen, remember in the silicon nitride case, one has to use the nitrogen as an atmosphere. Why nitrogen atmosphere? If you use the normal ambient conditions here, then all the silicon nitride will be oxidized to silica, okay. Now if you use the nitrogen atmosphere, then what would happen?

Silicon nitride decomposition also can be prevented and oxidation of silicon nitride to silica, that can be also well prevented. And this sintering conditions as you can see, this is quite an extreme because 1940 degree Celsius is quite a high sintering temperature. If you compare that of alumina, alumina can be sintered typically at 1600 degree Celsius for 2 hours or 4 hours. If you remember that of the zirconia, I have mentioned in the case study on zirconia that zirconia sintering temperature is somewhere around 1400 to 1450 degree Celsius.

So compared to oxide ceramics like alumina or zirconia, typically non-oxide ceramics like silicon nitride or silicon carbide, they require higher sintering temperature for their full consolidation or full densification. So simply because of the fact that these materials, they have very poor self-diffusion coefficient and it is the diffusional mass transport which leads to sintering or consolidation of the powder compact.

So if the self-diffusion coefficient is very low, these materials, these powders you cannot be densify or consolidated to full density. Now what is the hardness of these materials? This hardness of these materials, sialon phases, that is these materials, this first material SN1, it has hardness of around 16.1 gigapascal. If you go to SN2, that is the different source of the powder where d50 is 1 micron, it has around 15.8 gigapascal.

If you go to third one, is the beta, it is 14 gigapascal. If you go to SN5 which is a mixture of beta with certain seed of the beta (()) (11:52) nitride. It is 12.4 gigapascal. And if you go to simple beta but at different sintering conditions, 1850 versus 1940, 1850 one hour, then also you get the hardness of 13.2 gigapascal. So same material, there is a differences around 1 gigapascal.

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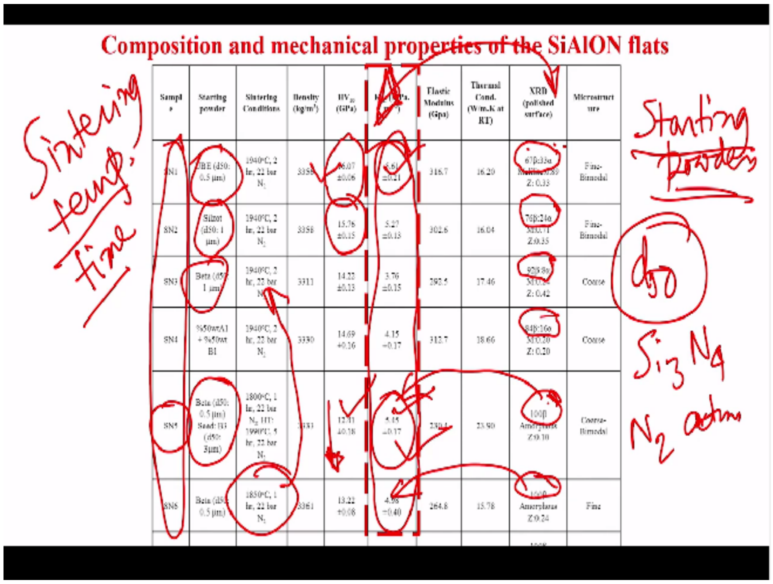

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SN6	Beta (d50: 0.5 µm)	1850°C, 1 hr, 22 bar N_2	3361	13.22 ±0.08	4.98 ±0.40	264.8	15.78	100β Amorphous Z:0.24	Fine

If you see their fracture toughness, which is typically measured using the indentation cracking method in case of the ceramics, you can vary not only the hardness properties, but also you can vary the fracture toughness of this material. Now what you have seen in one of the earlier lectures by me, I have categorically emphasized that fracture toughness of zirconia needs to be tailored in order to optimize the wear resistance property.

In other words, the major message of that particular lecture was that you can always strife towards obtaining better and better crack growth resistance property or fracture toughness properties. But at the expanse of reduction in the wear resistance property. So therefore, one has to have a compromise between fracture toughness and wear resistance property. This is fairly important for you to remember.

Now in this particular case study, I would once again highlight that how fracture toughness properties play an important role in determining wear resistance properties for a different class of materials, that is called oxynitride ceramics like silicon oxynitride ceramics, like sialon silicon aluminium oxynitride ceramics. So this is again another example of the high toughness ceramics. Typically fracture toughness of ceramics is varying somewhere between 3 to 4 like alumina and many other silicon carbide.

But here silicon oxynitride one can go to as high as 5.5 or 5.6 MPa square root meter which is close to that of the double of that alumina based ceramics. So therefore, in ceramic community, 6 MPa square root meter or 10 MPa square root meter is a respectable number as far as the fracture toughness of this materials are concerned. So I re-iterate my earlier statement by tuning or by tailoring the sintering temperature and time as well as starting powder composition.

One can get a range of fracture toughness values in this particular set of silicon aluminium oxynitride materials. And these fracture toughness is largely due to, the differences in the fracture toughness is largely due to differences in the phase composition, phase assemblage. Now if you compare these 2 columns, my statement can be clarified. For example, here you can see that beta/alpha ratio is 67 to 33, that is 2:1.

Here you can see beta/alpha ratio is 76/24 that means more or less 3:1. Here you can get beta/alpha ratio is 92/8, so that means again it is more than close to 12:1. Here it is 16:84 like 5:1. This is 100% beta. So any of 100% beta, here again is a 100% beta. You can see that these materials have little bit higher fracture toughness. But also here beta fraction is higher, it is 2:1. Here again one has obtained 5.6 gigapascal.

But if it is 100% beta, then you can get better fracture toughness properties but hardness is lower. But if you have to get a good combination of fracture toughness and hardness, then you should go for 2:1, like you need to have some alpha phase as well as beta phase. Because if you remember, in one of the earlier slides in this lecture, I have emphasized that alpha and beta, there are distinct differences like beta phase or beta silicon nitride or beta sialon phase.

They typically have higher fracture toughness. Whereas alpha sialon or alpha silicon nitride, they typically have higher hardness. So that is being reflected here. If you have predominantly beta phase with little bit of alpha phase, then once can clearly have the best combination of fracture toughness and hardness. But if you have pure beta phase or 100% beta phase, then it is possible that your fracture toughness still remained higher; however, because of the absence of the alpha phase in the microstructure, you can expect lower hardness properties.

And this statement is very well proven in this particular example for the ceramic SN5 and SN6.

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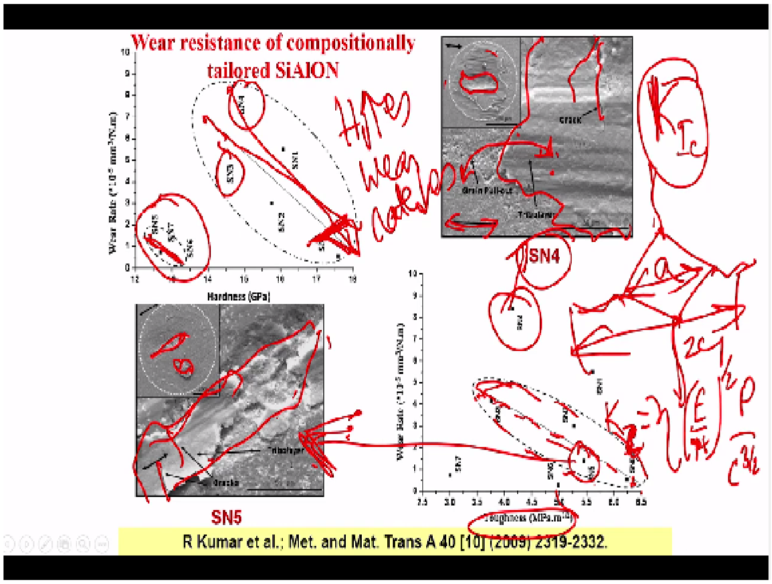

Wear resistance of compositionally tailored SiAlON

SN4

SN5

R Kumar et al.; Met. and Mat. Trans A 40 [10] (2009) 2319-2332.

Now with this background on the materials, let me now explain that how wear rate is vary with respect to hardness and the fracture toughness of this materials. And what I am going to show you now that first in case of hardness properties, wear rate typically decreases with increase in hardness properties, even it is a hardness of the sialon. So these are the grades of the different sialon that I have mentioned.

So there are 2 type of circles. One circle you can see that within the narrow range of 12.5 to 13.5 gigapascal hardness variation, there is a decrease in the wear rate. But this wear rate decreases not that significant. But if you look at this another circle, larger circle of variation here. When the hardness decreases somewhere around 13 or 14 to 18 gigapascal, then there is also a decrease in the wear rate from 8*10 to the power -5 to somewhere around 1 or less than 1*10 to the power -5.

So that means there is couple of times reduction in the wear rate of this compositionally tailored sialon materials. Now so the major message from this plot is that if hardness increases, wear rate decreases or in other words, wear resistance increases. Now let us now look at very closely how does this wear rate decreases, how this wear rate varies with fracture toughness which is again measured by indentation cracking which I have explained to you earlier but let me explain again.

This is the Vickers hardness. In case of brittle materials like ceramics, from the indent corners, these cracks will generate and then they will propagate. And if you measure the crack length and this crack length, based on the crack length and based on these diagonal, you can measure the fracture toughness or KC values is some eta parameter E/H to the power 1/2, then P/C to the power 3/2.

So C is your half of the crack length, H is your hardness. So this hardness is to be measured at indent load where there is absolutely no cracking. So indent load where you are measuring the fracture toughness, you cannot use the same hardness value and plugin in this particular equation to get the fracture toughness values. So this is KC. KC stands for critical crack tip stress intensity factor.

Many times people use it is as a K1C which is wrong because 1 means this is the critical crack tip stress intensity factor under mode 1 loading. But this indentation cracking methods to measure the fracture toughness does not involve purely mode 1 type of loading. What people can achieve by (()) (20:48) kind of techniques where there will be at the crack tip, purely tensile loading. but that is not the case in this particular scenario.

Therefore, it is appropriate to mention is the KC, it is the eta E/H to the power 1/2 and P/C to the power 3/2. So what you notice here that with increase in the fracture toughness from around 3.5 to around 6.5 MPa square root meter, your wear rate is reduced from 5 to less than 1 MPa square meter. So with increase in the fracture toughness for a specific group of silicon oxynitride ceramics, one can clearly see there is a reduction in the wear rate with increase in the fracture toughness.

So what it means that that seems this particular case and we will see now in the SN images. In scenario where wear or material removal is dominated by cracking and cracking induced spalling, then if you use materials with higher fracture toughness or higher crack growth resistance properties; therefore, there it is possible to achieve better wear resistance simply because in higher fracture toughness materials, one will have difficulty or the cracks will find it more difficult to propagate in the microstructure leading to spalling.

So therefore, crack growth induced spalling can be minimized and therefore materials with better fracture resistance or better fracture resistance will be useful for better wear resistance property as well. Now let me substantiate my statement further. Now if you look at this worn surface image, the way I am tracing, so these are the typical cracking, right. And this cracking is typically at perpendicular to that of the fretting directions.

And you can clearly see there is a thick tribolayer and then thickness can be confirmed from the contrast difference between the worn surface as well as this tribolayer. And these you are seeing from the middle of this worn surface and this is how this tribolayer is formed. Now at different SN4 for example which is very high wear resistance, very high wear rate but low wear resistance.

This is the scenario. In the SN5, which is somewhere here where the material has, this is this one and this is this one. SN5 is the material where you can see that there is tribolayer. There is a cracking but tribolayer seems to rotate the materials quite nicely and that you can see from the center of the worn surface. So what you notice here that crack growth resistance is increased as a result the wear resistance also increased.

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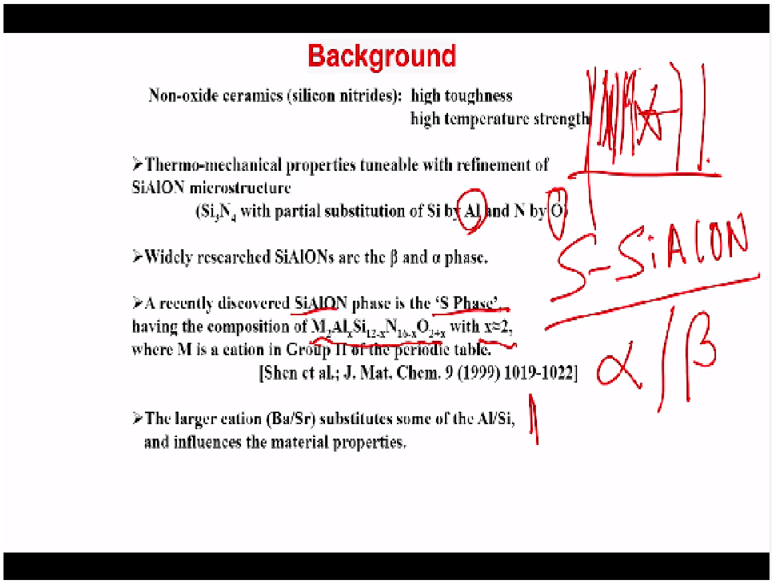

Background

Non-oxide ceramics (silicon nitrides): high toughness
high temperature strength

- Thermo-mechanical properties tuneable with refinement of SiAlON microstructure
  (Si_3N_4 with partial substitution of Si by Al and N by O)
- Widely researched SiAlONs are the β and α phase.
- A recently discovered SiAlON phase is the 'S Phase', having the composition of $M_2Al_xSi_{12-x}N_{16-x}O_{2+x}$ with x≈2, where M is a cation in Group II of the periodic table.
  [Shen et al.; J. Mat. Chem. 9 (1999) 1019-1022]
- The larger cation (Ba/Sr) substitutes some of the Al/Si, and influences the material properties.

S-SiAlON

α / β

Now in next few minutes, I am going to show you another class of materials and this class of materials is called S-sialon materials. S-silicon aluminium oxynitride materials. It is S phase sialon. You already know there are 2 phases, alpha phase and beta phase. And then we have already mentioned that non-oxide ceramics like silicon nitride, they have high fracture toughness and high temperature strength which is not the case for oxide ceramics.

So therefore, non-oxide ceramics are very important for various engineering applications, okay. So non-oxide ceramics are very important and then you can do this silicon, part of the silicon in the silicon nitride lattice is replaced by aluminium and part of the nitrogen replaced by oxygen, then you form the sialons. So in 1999, almost like 20 years ago, James Shen's group at Sweden, I think, Stockholm, they have discovered sialon is a new phase that is S phase sialon which is a composition of M2AlxSi12-x nitrogen 16-x and O2+x and with x=2.

And M is a cation of group III or group II of the periodic table. And larger cation barium to strontium substitutes some part of the aluminium to silicon.

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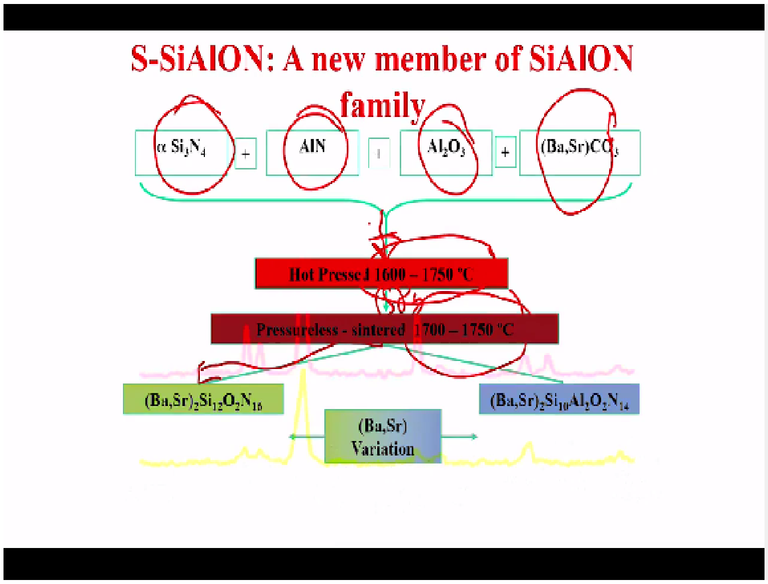

S-SiAlON: A new member of SiAlON family

α Si_3N_4 + AlN + Al_2O_3 + (Ba,Sr)CO_3

Hot Pressed 1600 – 1750 °C

Pressureless - sintered 1700 – 1750 °C

$(Ba,Sr)_2Si_{12}O_2N_{16}$

(Ba,Sr) Variation

$(Ba,Sr)_2Si_{10}Al_2O_2N_{14}$

And that influence the material properties. How this materials are done, how these materials are processed? As I have mentioned earlier that sialon phase, sialon materials you use the silicon nitride, aluminium nitride and alumina. So all these phases are same but to dope with barium, it is we have to use barium and strontium. We have to use the precursor powder that is barium or strontium carbonate.

You mix it, you dry it, you hot press this dried powder mix at 1600 to 1750 degree Celsius. You can do alternatively pressureless sinter. So it is or pressureless sintered at 1700 to 1750 degree Celsius. Then you get barium strontium silicon oxygen nitrogen. So that is the barium or strontium variation of the sialon family.

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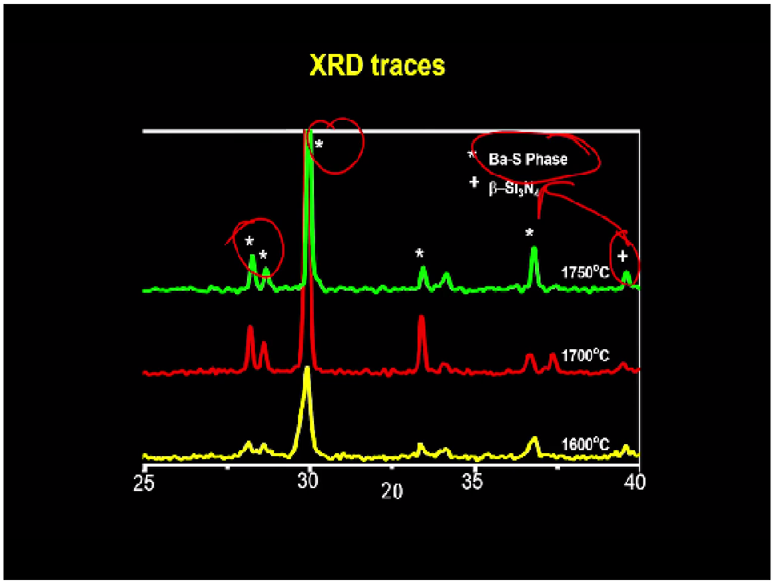

XRD traces

This is the typical XRD traces. And what you notice here that you have a barium dope S phase, silicon S sialon phase. This is that phase (()) (26:35) and also there is one more phase called beta silicon nitride but there amount is certainly much less because barium based S-sialon phase is very significant amount.

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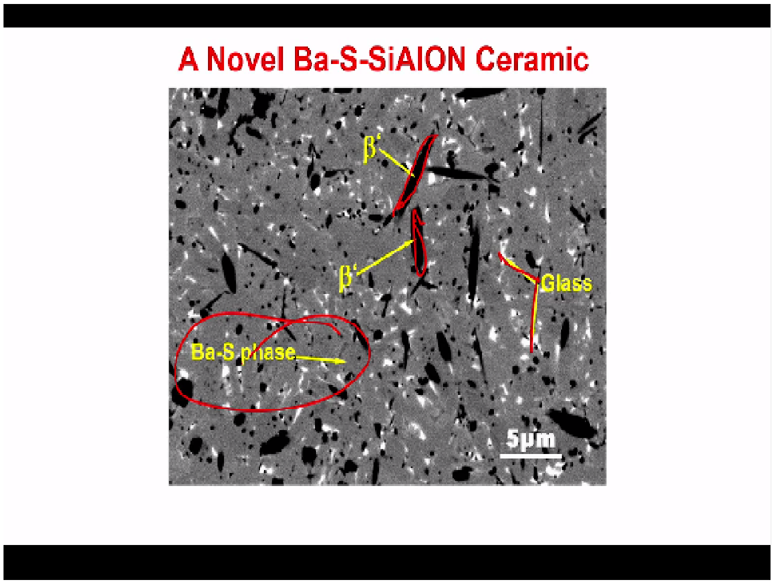

A Novel Ba-S-SiAlON Ceramic

So typically beta phase silicon nitride, it is a typical needle that which is dispersed in the microstructure. You have the residual glass phase and you have the barium dope S-sialon phase which you can clearly see.

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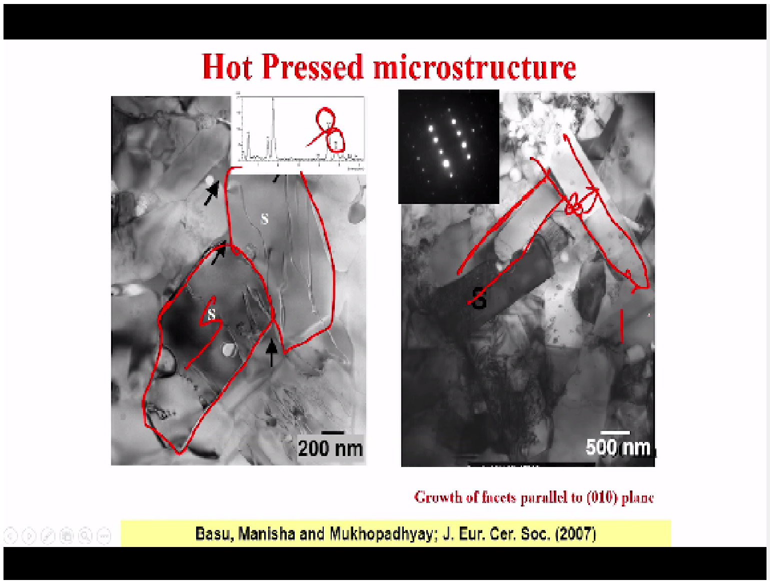

Hot Pressed microstructure

Growth of facets parallel to (010) plane

Basu, Manisha and Mukhopadhyay; J. Eur. Cer. Soc. (2007)

This is a very fascinating microstructure. This is a tabular shaped barium silicon aluminium oxynitride phase and you can see that barium silicon oxynitride phase. If you take a EDAX spectra, in TEM you can clearly see the composition that barium peak. So you have that typical tube shaped or large separate materials and then you can see also, these are different magnification. So typically the width of this tabular phases parallel to the 1-0 plane, it is around some 400 to 500 nanometer.

And also there is a definite aspect ratio of this phase and this aspect ratio is somewhere 8 to 9 aspect ratio.

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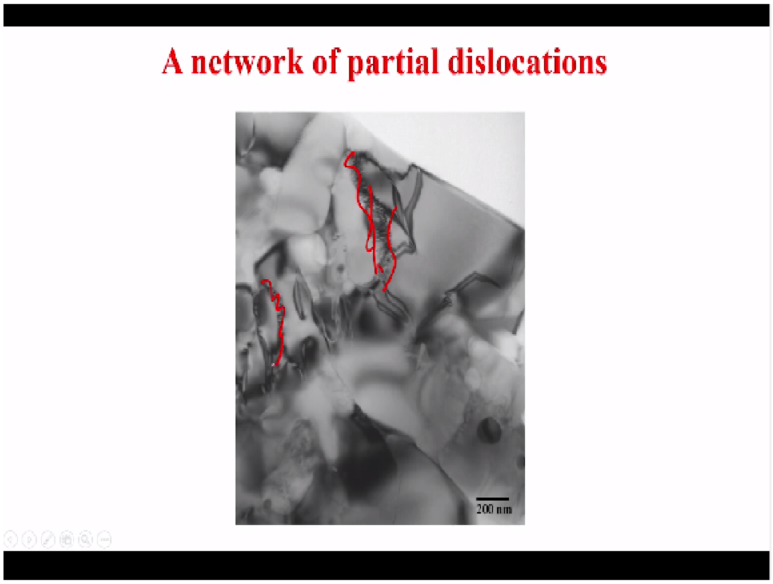

A network of partial dislocations

We can see very clearly that there are threads of dislocations. So these are defect structure and somewhere also we have seen APB, antiphase boundary. These are there dislocations threads in this particular ceramic materials.

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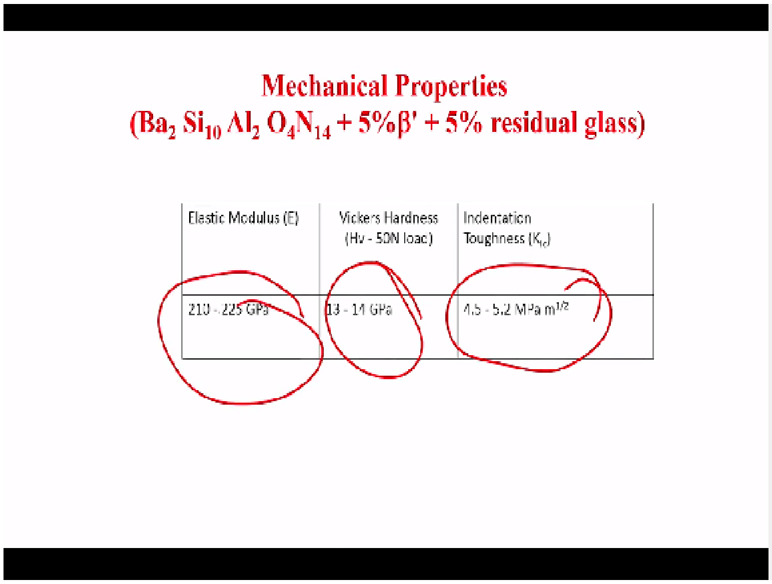

Mechanical Properties

($Ba_2\ Si_{10}\ Al_2\ O_4N_{14} + 5\%\beta' + 5\%$ residual glass)

Elastic Modulus (E)	Vickers Hardness (Hv - 50N load)	Indentation Toughness (K_{Ic})
210 - 225 GPa	13 - 14 GPa	4.5 - 5.2 MPa $m^{1/2}$

So as per the properties are concerned, this is not a great property in terms of the hardness and fracture toughness because in the last few slides I have shown that by using the different starting powders and hot pressing conditions or sintering conditions, one can obtain the fracture toughness and hardness over a large range. And this is somewhere in the middle. So here it is around 4.5 MPa square root meter which is not too bad. But at the same time, this material has a steel like elastic modulus like 210 gigapascal.

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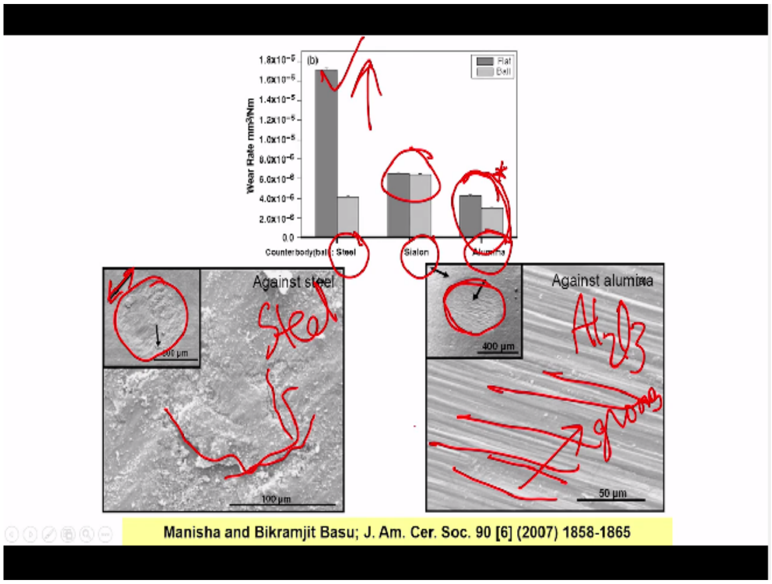

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Now whenever a new material is being tested for wear resistance property, it is always recommended to use 3 different kind of matting solids. Like here we have used steel, sialon and alumina. So this is the case of this against steel based materials. This is the case for against alumina based materials. So first let me explain that how does this wear rate varies. For sialon versus alumina, this total wear rate, so total wear rate of the system, it is significantly lowest here.

In case of alumina, it is very high. Here in case of steel, and for self-matted couple like sialon versus sialon, there is wear rate is moderate or intermediate between that of steel and alumina. As far as the wear resistance is concerned, what you see here that in case of steel materials, there is a very thick tribolayer and there is also delamination wear. And this particular tribolayer is spalled off and this is your fretted wear surface.

So this is your fretting direction. In case of alumina, there is abrasive scratches and also there is a deeper abrasive groups. So these are like called groups, abrasive groups which are present on this worn surface. And then we have a very clear fretted scar on this particular materials.

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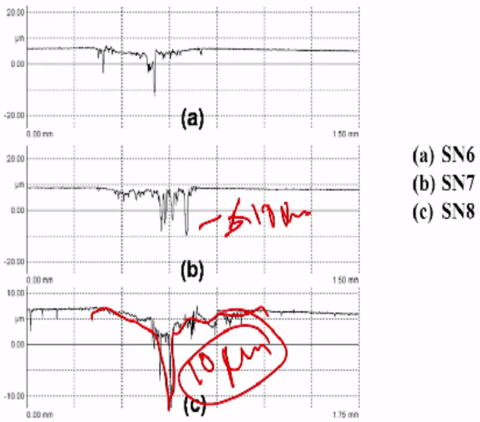
2D surface profiles



(a) SN6
(b) SN7
(c) SN8

Now these are some of the 2D surface profiles just to show that how the surfaces, the depth of this 2D surfaces which is fairly good like around 10 micron and in some cases, depth is around less than 10 micron also we have observed.

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journal

J. Am. Ceram. Soc., 90 [6] 1858–1865 (2007)
DOI: 10.1111/j.1551-2916.2007.01700.x
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Tribological Properties of a Hot-Pressed Ba-Doped S-Phase Sialon Ceramic

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In this study, we report the tribological properties of hot-pressed S-sialon ceramics. In our experiments, the tribological properties have been studied against bearing grade steel, alumina, and sialon using a ball-on-flat fretting tribometer under dry unlubricated conditions. While the steady-state coefficient of friction (COF) remains the same (0.63) irrespective of the counterbody, the wear rate varies in the range of 10^{-5}–10^{-6} mm^3/N·m, depending on the counterbody. Tribochemical wear as well as adhesion and abrasion are found to be the underlying mechanisms for material removal. The possible influence of Hertzian contact stress and the contact temperature increase on the possible damage mechanisms/tribological interaction has been analyzed.

ious structural components requiring a good combination of mechanical and tribological properties over a wide range of load conditions (5–90 N load). In another attempt to improve the properties of α-sialon by β-sialon fiber reinforcement, it was interestingly observed that the wear rate increases as the fiber content increases.[5] The wear mechanisms of β-sialon fiber-reinforced α-sialon composites were dominated by adhesion and micro-abrasion between the rubbing surfaces. Nakamura *et al.*[7] studied the wear properties of α-Si_3N_4 reinforced with in-plane aligned rod-like β-Si_3N_4 grains using a block-on-ring tribometer against a Si_3N_4 counterbody. The grain pullout, reported to be caused by propagation of microcracks parallel to the sliding surface, was the major wear mechanism.

So all these properties we have published in journal of American ceramics society papers in 2007, more than 10 years ago. And these particular ceramic is being investigated in other research groups as well for their various potential engineering applications. Thank you.