Sialon Hot-Section Components: Phase I Wrap-up

Work performed under Contract 4000017596: Hot Section Materials Development of Advanced Microturbines.

Russ Yeckley, Kennametal

Prof. John Hellmann, Prof, James Adair and Kevin Fox; The Pennsylvania State University

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Phase I

- Sialon Mechanical/Corrosion Behavior
  - Demonstrate sialon's potential for microturbine hot-section component.
  - Processing capability
- Microstructure Characterization
- Impression Creep
- Colloidal Processing

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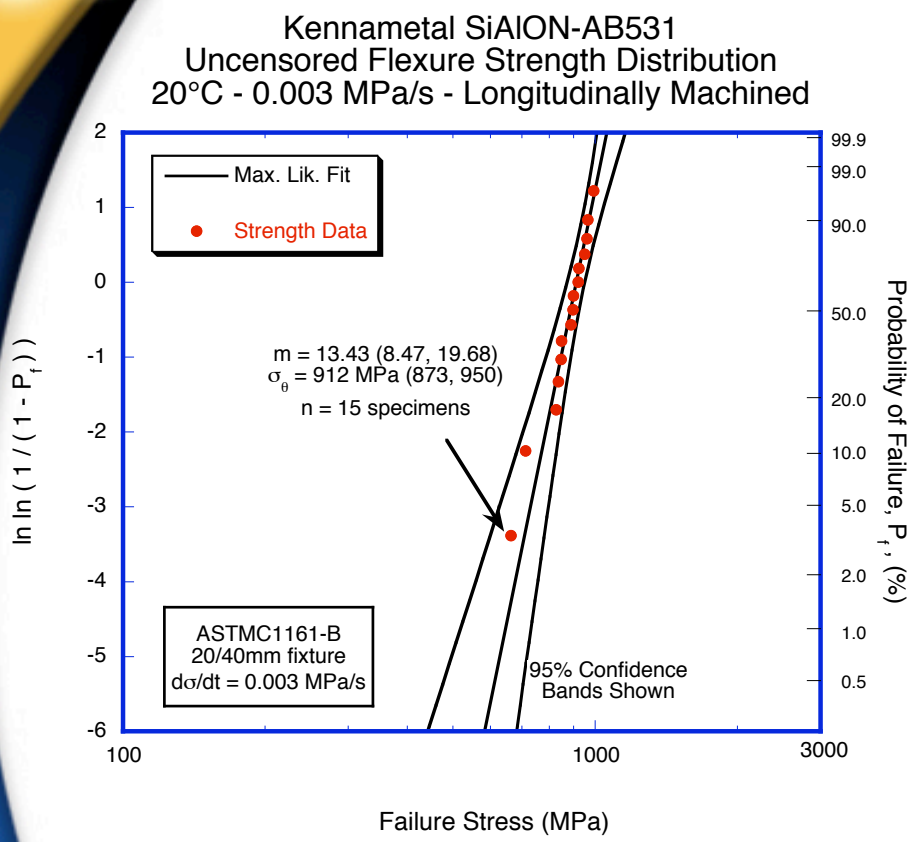
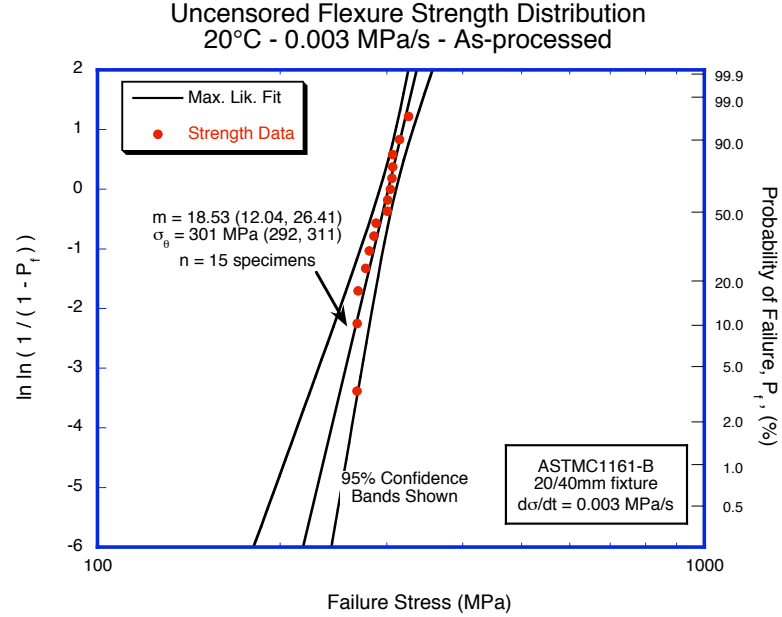
Sialon Mechanical/Corrosion Behavior

- Sialon Screening Matrix
  - Composition Matrix
    - Four with β-sialon w/ z ~ .35
    - Four with β-sialon w/ z ~ .8
    - Two intermediate
    - Two rare earth levels.

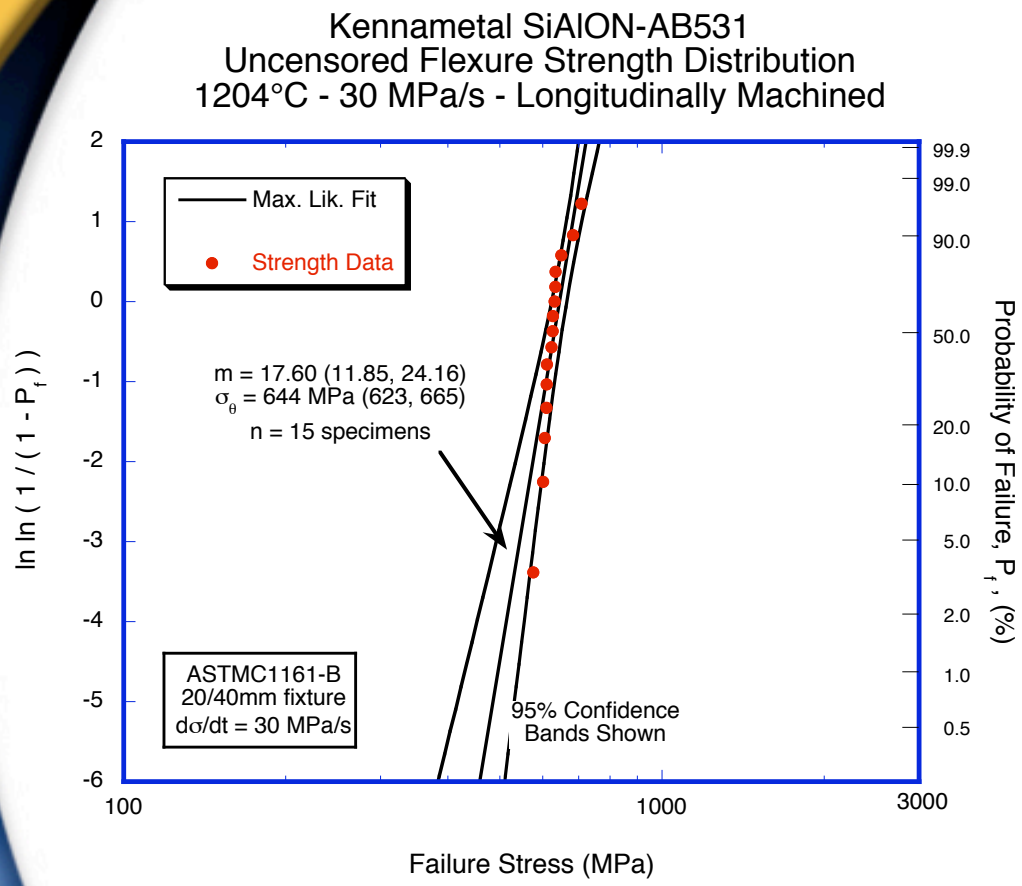
	Hvn	st dev	Kic	st dev	Fracture Strength				α	α sialon	β sialon
Sialon	GPa		MPa m1/2		RT	m	1204	m	%	x'	z'
ab831	18.35	0.13	6.89	0.26	960	8.5	607	10.5	32	0.26	0.33
ab582	19.48	0.25	4.95	0.51	567	7.2	331	8.9	58	0.40	0.79
ab581	18.19	0.24	4.59	0.30	455	8.3	383	6.7	36	0.42	0.78
ab581	18.31	0.33	4.09	0.13					37	0.42	0.77
ab531	17.62	0.33	5.83	0.26	801	10.7	497	24.0	28	0.41	0.92
ab832	18.72	0.35	7.36	0.22	783	6.1	540	6.3	44	0.25	0.38
ab532	17.38	0.58	5.73	0.16	651	5.9	393	9.8	36	0.41	0.84
ab132	18.21	0.25	6.91	0.11	878	6.4	507	8.6	39	0.35	0.57
ab131	17.39	0.20	6.58	0.16	733	6.3	466	10.3	28	0.37	0.64

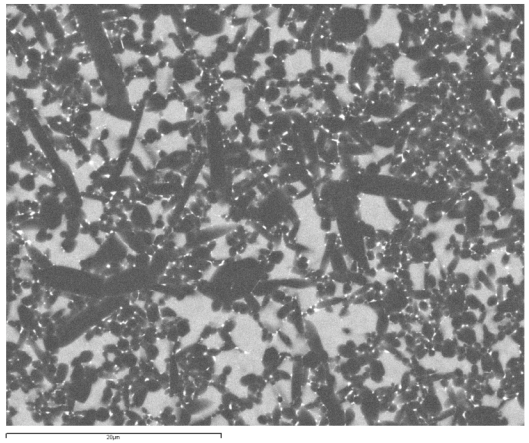
Strength Sialon ab531



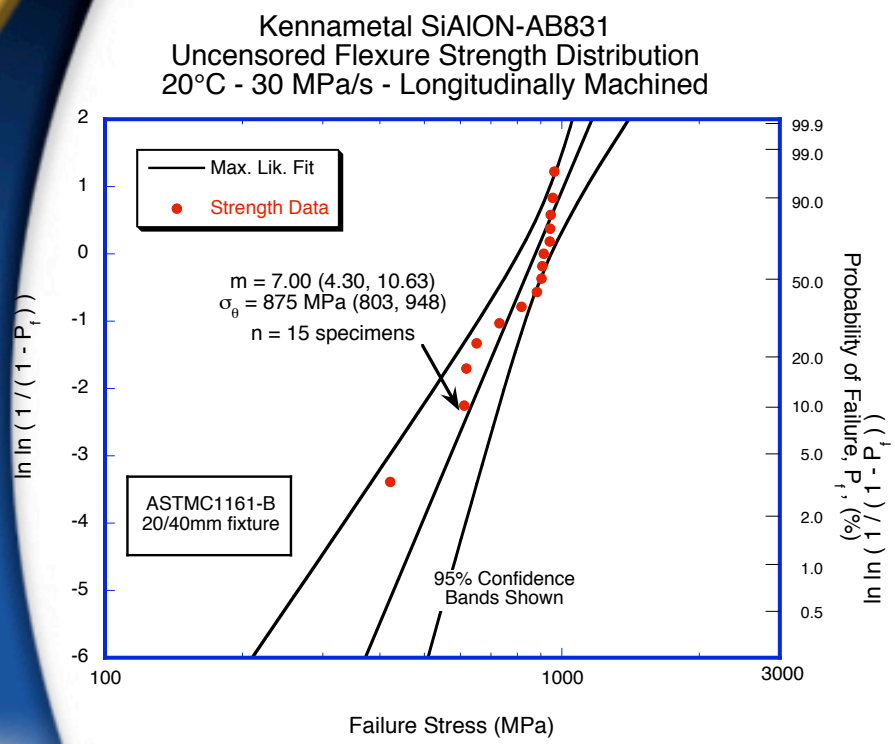


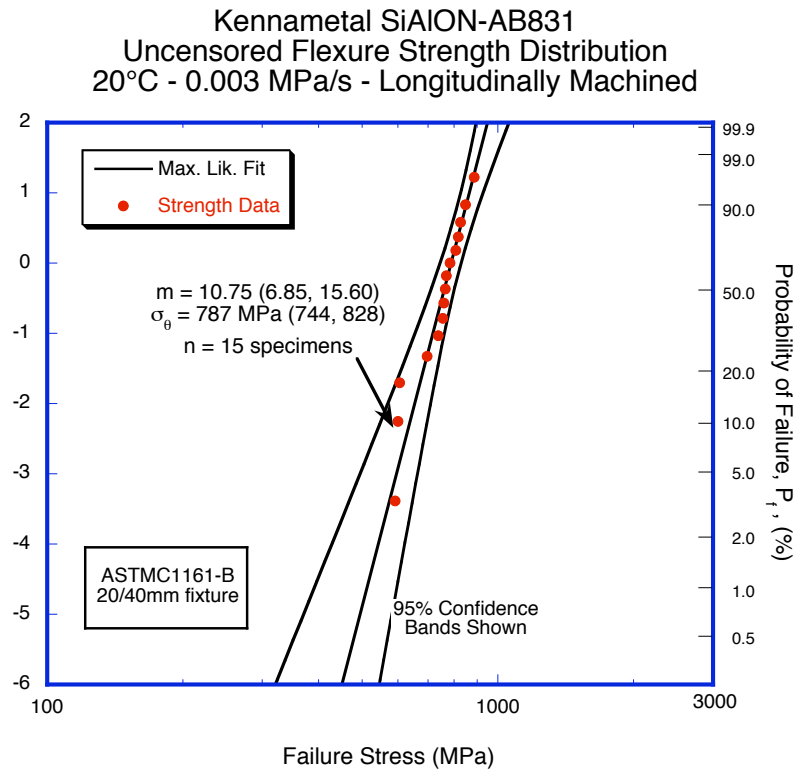
1204 C Strength Sialon ab531



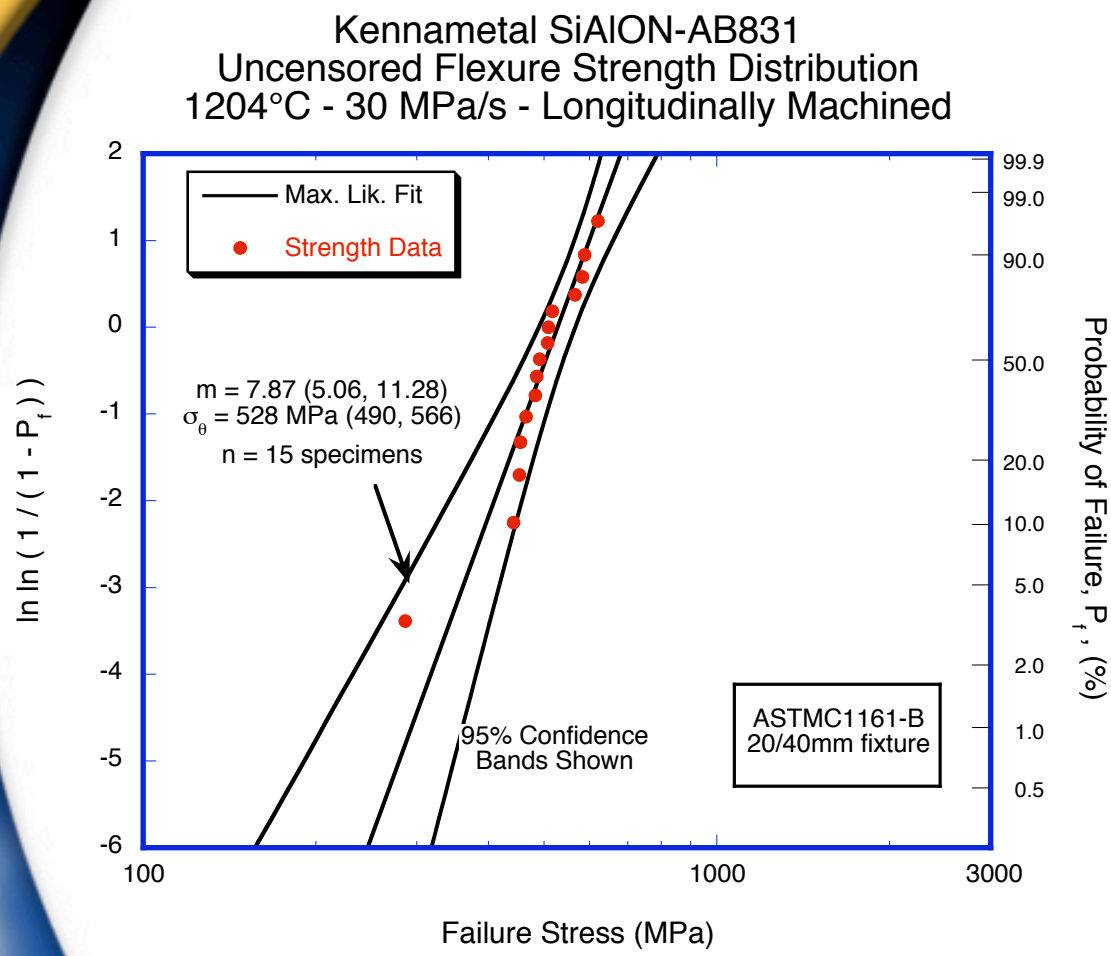


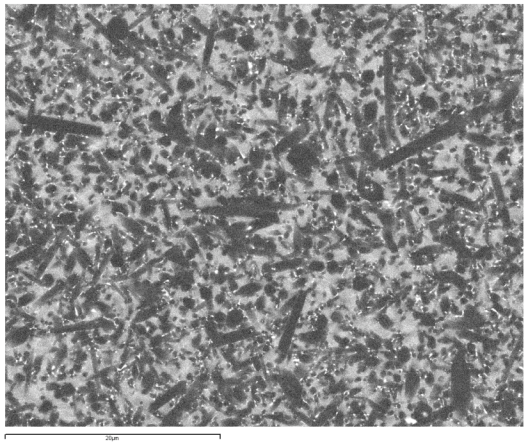
Strength Sialon ab831



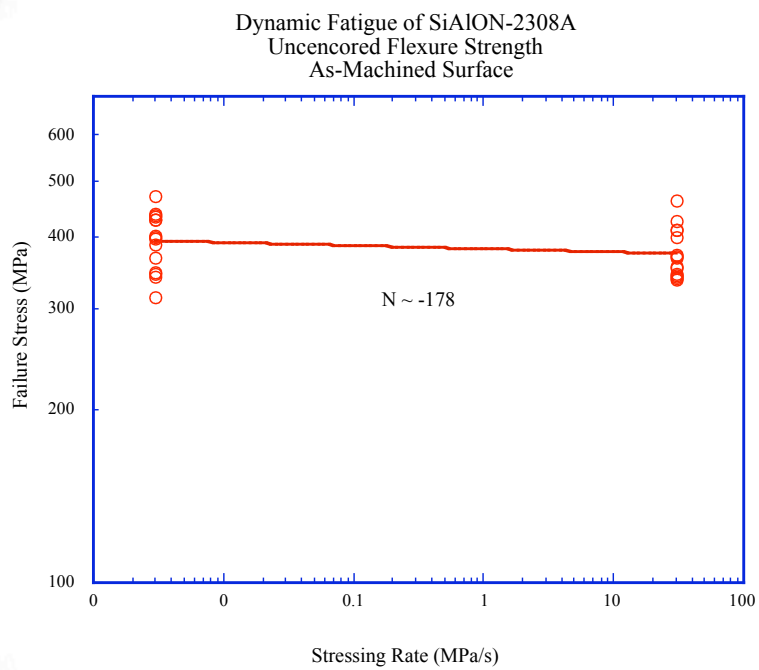


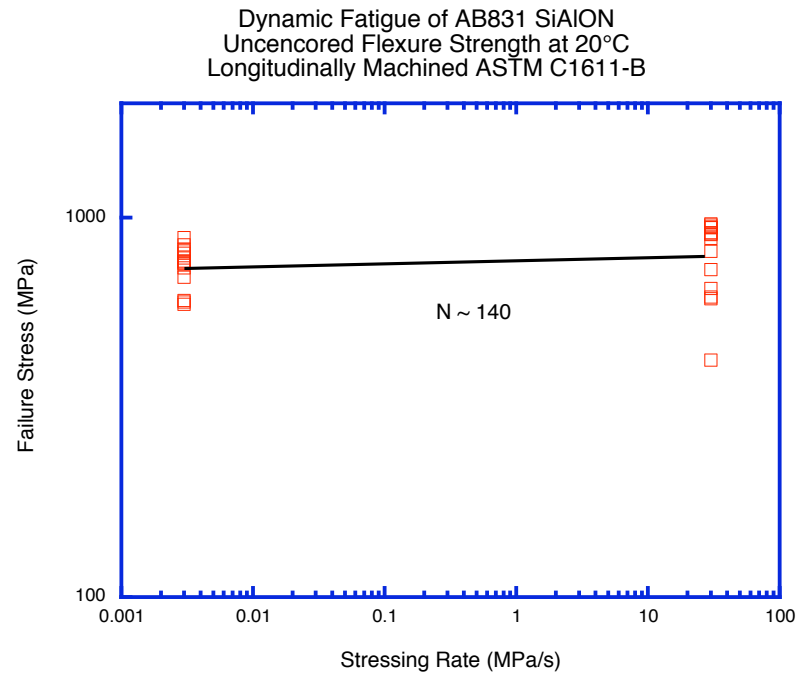
1204 C Strength ab831





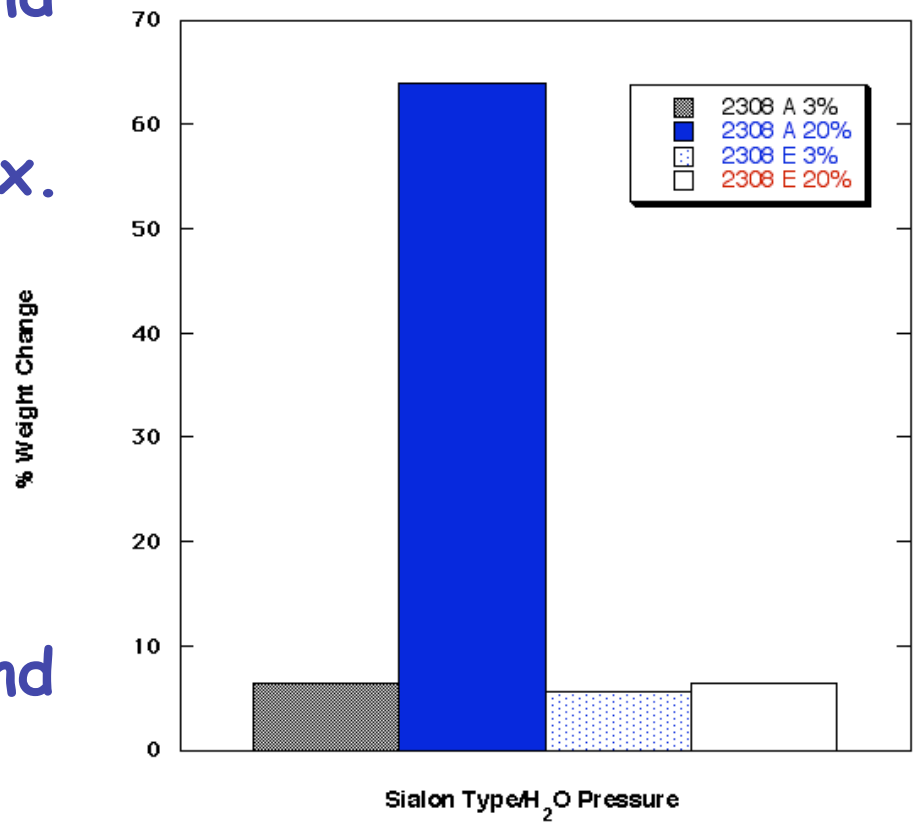
Fatigue Resistance





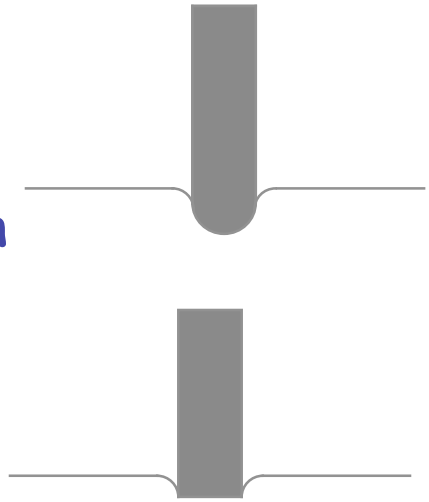
Kaiser Rig Results

- Sialon ab832 and ab582 from screening matrix.
- Sialon ab582 comparable to silicon nitride
- EBC's required
- Sialon ab531 and ab831 samples provided.



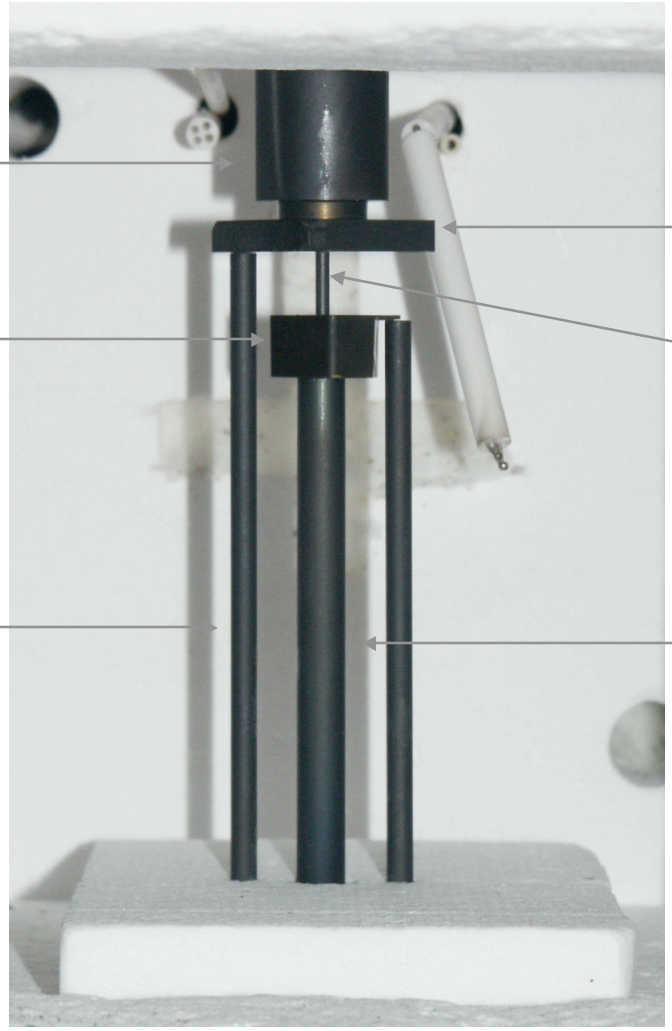
Impression creep

- Advantages of using a small indenter to measure creep behavior:
  - Small sample size
  - Multiple measurements per specimen
  - Minimal specimen machining



- Indenter geometry
  - Hemispherical
    - Reduced dependence on alignment
    - Contact area changes with depth of penetration
  - Flat-ended cylinder
    - Stress on the specimen does not change significantly with time or depth of penetration
    - Gives a steady state penetration velocity at constant load

Impression creep system



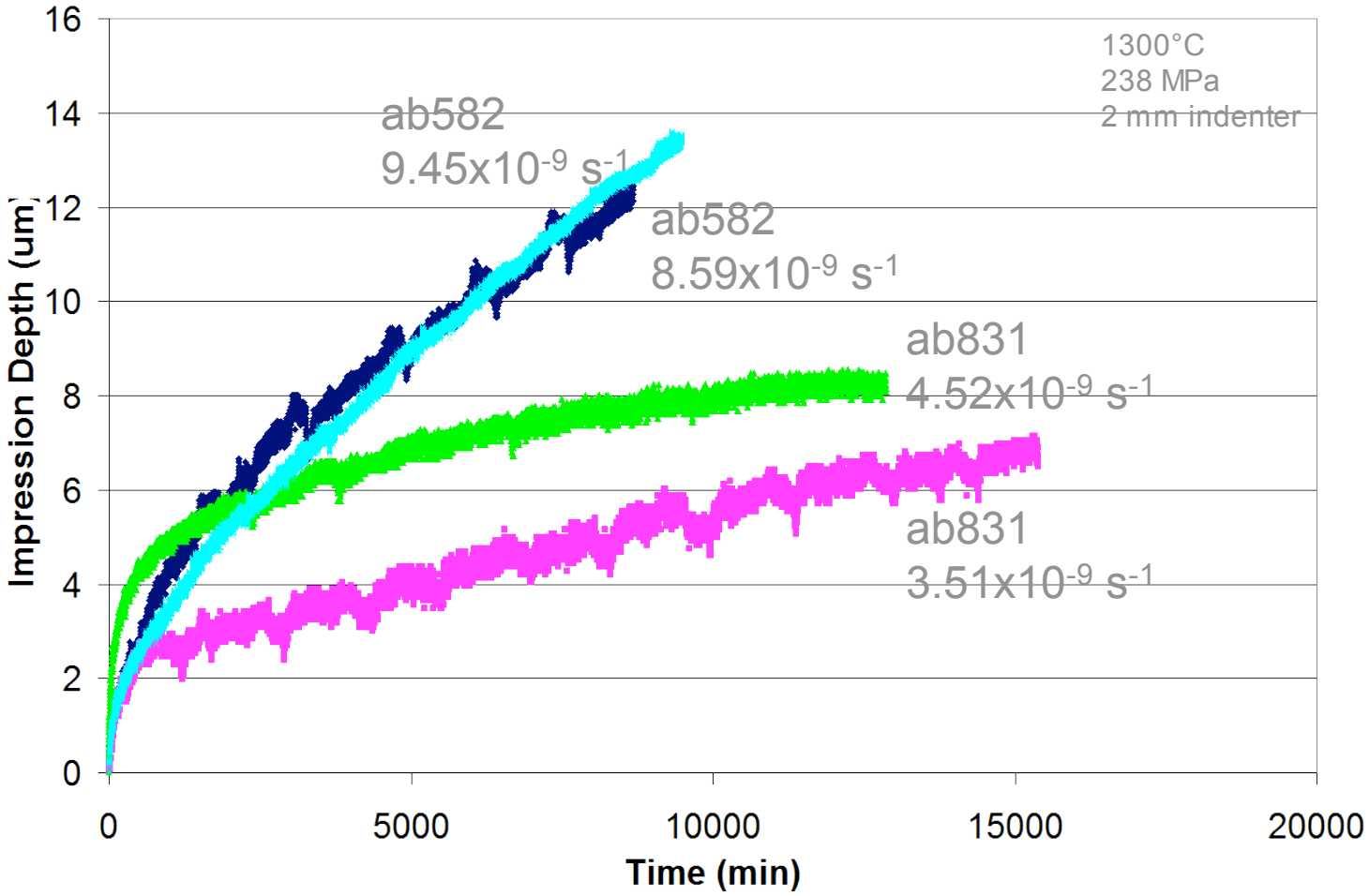
Impression Creep

Table 4-1. Initial results of impression creep testing of NT154. Two tests were run at the test conditions shown.

Test conditions	Strain rate measured in this study	Strain rate reported by Wereszczak (for compression)[28]
1399°C, -300 MPa	$-3.19 \times 10^{-9} \pm 0.10 \times 10^{-9}\ s^{-1}$ $-5.64 \times 10^{-9} \pm 0.17 \times 10^{-9}\ s^{-1}$	$-2.1 \times 10^{-9}\ s^{-1}$

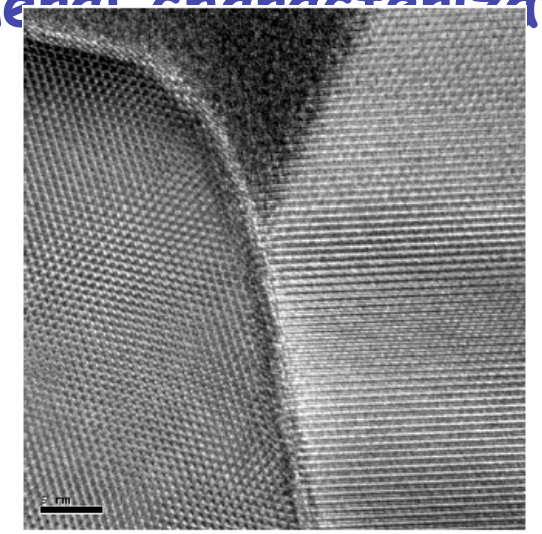
- Impression creep rates comparable to Compression creep
- Determine activation energy and stress exponent two sialons.

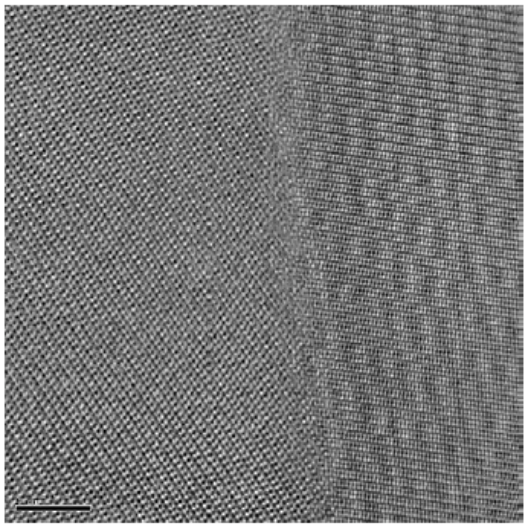
Impression creep data comparison



Microstructure Characterization

- Primary goal; correlate Yb content in alpha to XRD models
- General characterization.





High-resolution TEM micrographs of grain boundaries between Two alpha sialon grains in ab831(left) and ab531 (right).

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Composition predicted by XRD vs. EPMA Results

Composition	Phase	at.% Yb, XRD using x from *a*	at.% Yb, XRD using x from *c*	at.% Yb, EPMA
ab531	alpha	1.45% ± 0.01%	1.59% ± 0.01%	1.29% ± 0.04%
ab831	alpha	0.93% ± 0.01%	1.04% ± 0.02%	1.17% ± 0.01%
ab581	alpha	1.42% ± 0.00%	1.55% ± 0.01%	1.33% ± 0.03%

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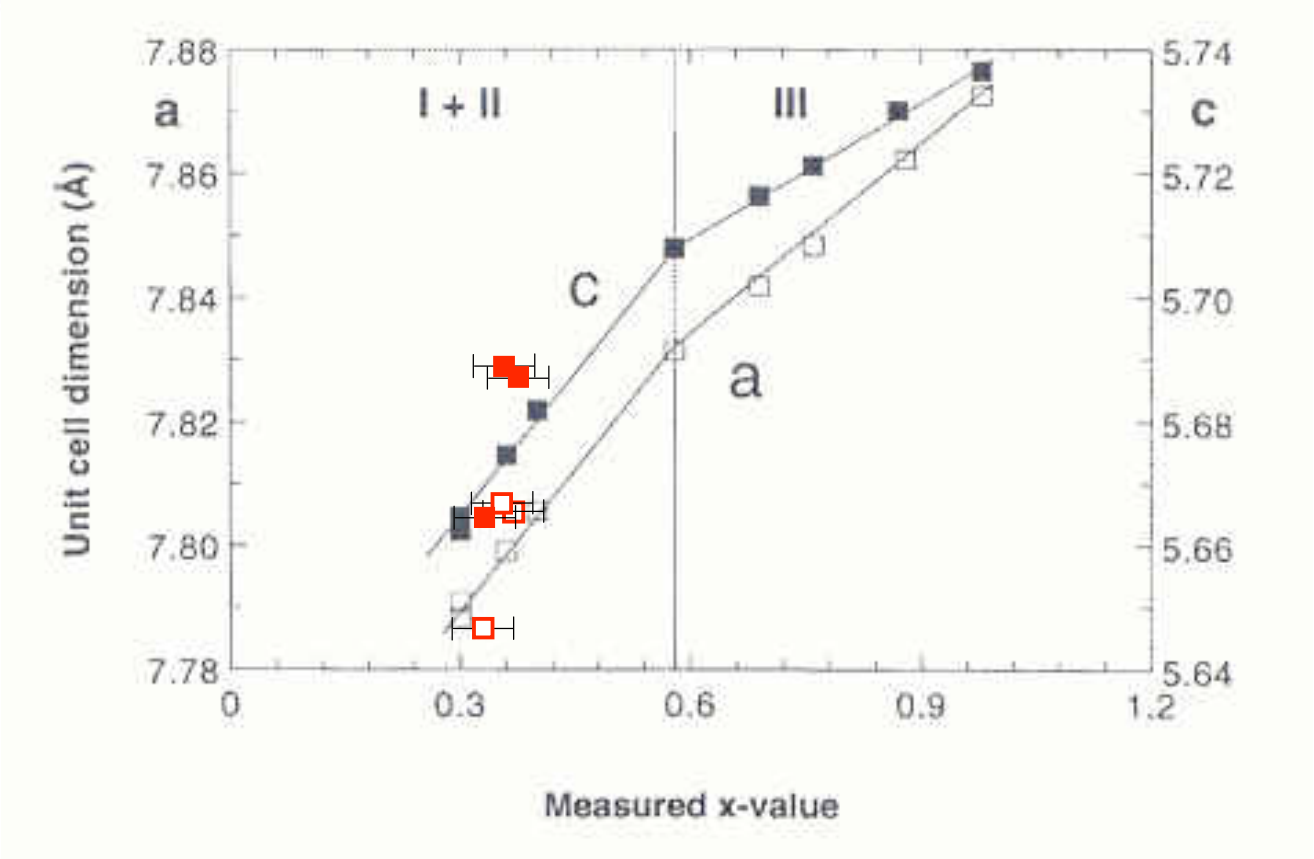
Lattice parameters/composition relationship



- Data points in red are from the Kennametal SiAlONs.
- Data points in black are from the published work.

Shen, Ekstrom and Nygren, J. Phys. D: Appl. Phys., **29** 893-904 (1996).

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Colliodal Processing

- Goal; slip formulations that would be suitable for complex shape forming
- Use passivation – dispersion approach to stabilize the different powders
- Studied water and IPA solvents.

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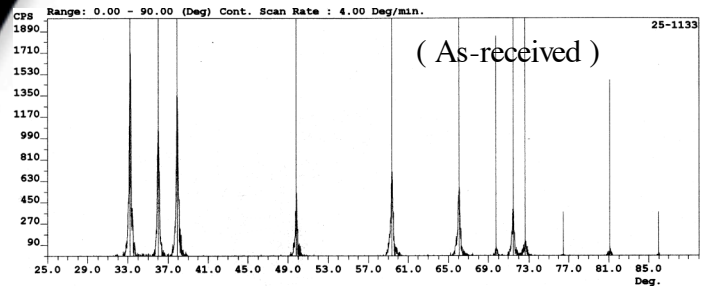
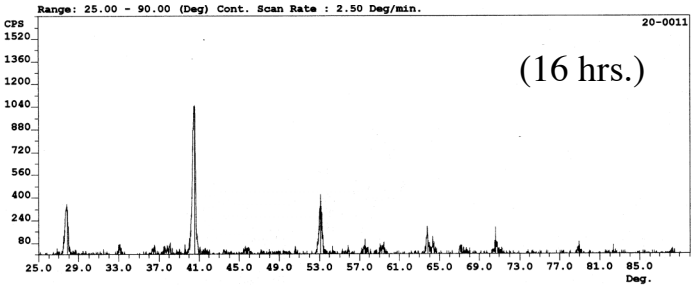
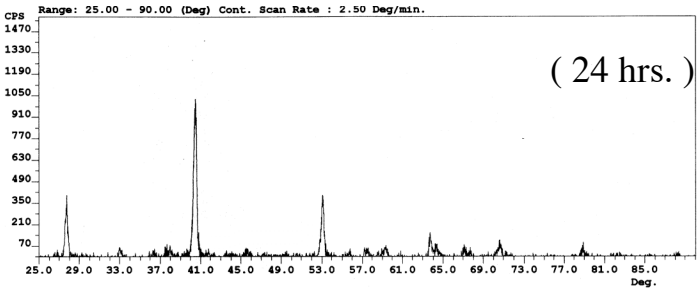
Most Difficult Component AlN

ALN in H_2O (50 w/o)

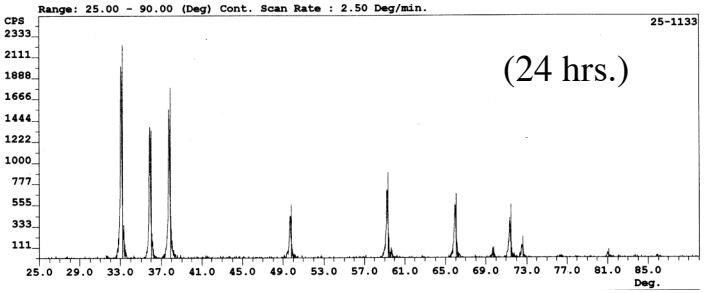


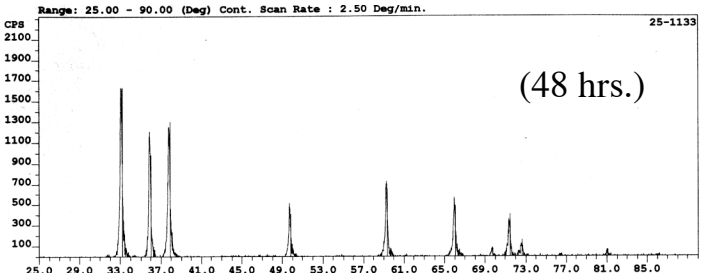


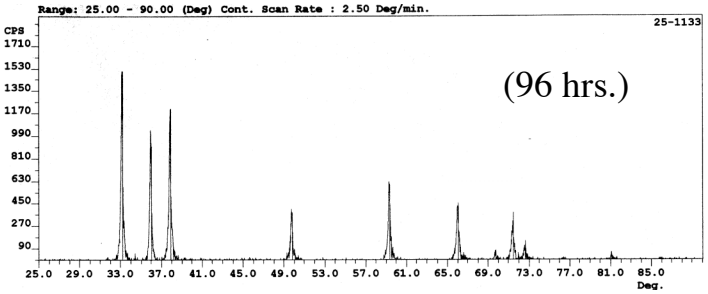


Aluminum Hydroxide/ Bayerite

ALN (50 w/o) + 2 w/w Succinic Acid in H_2O







Aluminum Nitride

The Passivation - Dispersion Approach

Heterogeneous Powders

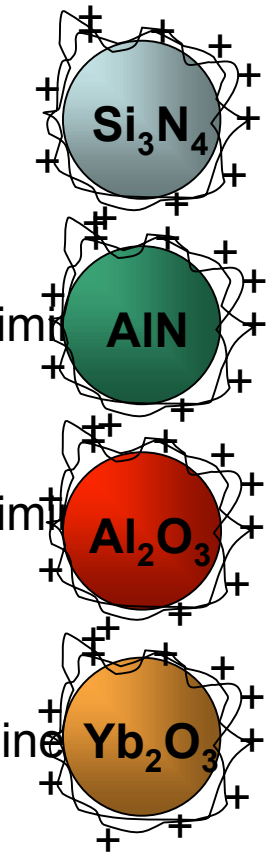
Uniform Dispersion

Si_3N_4 → pH Control, Polyethyleneimine

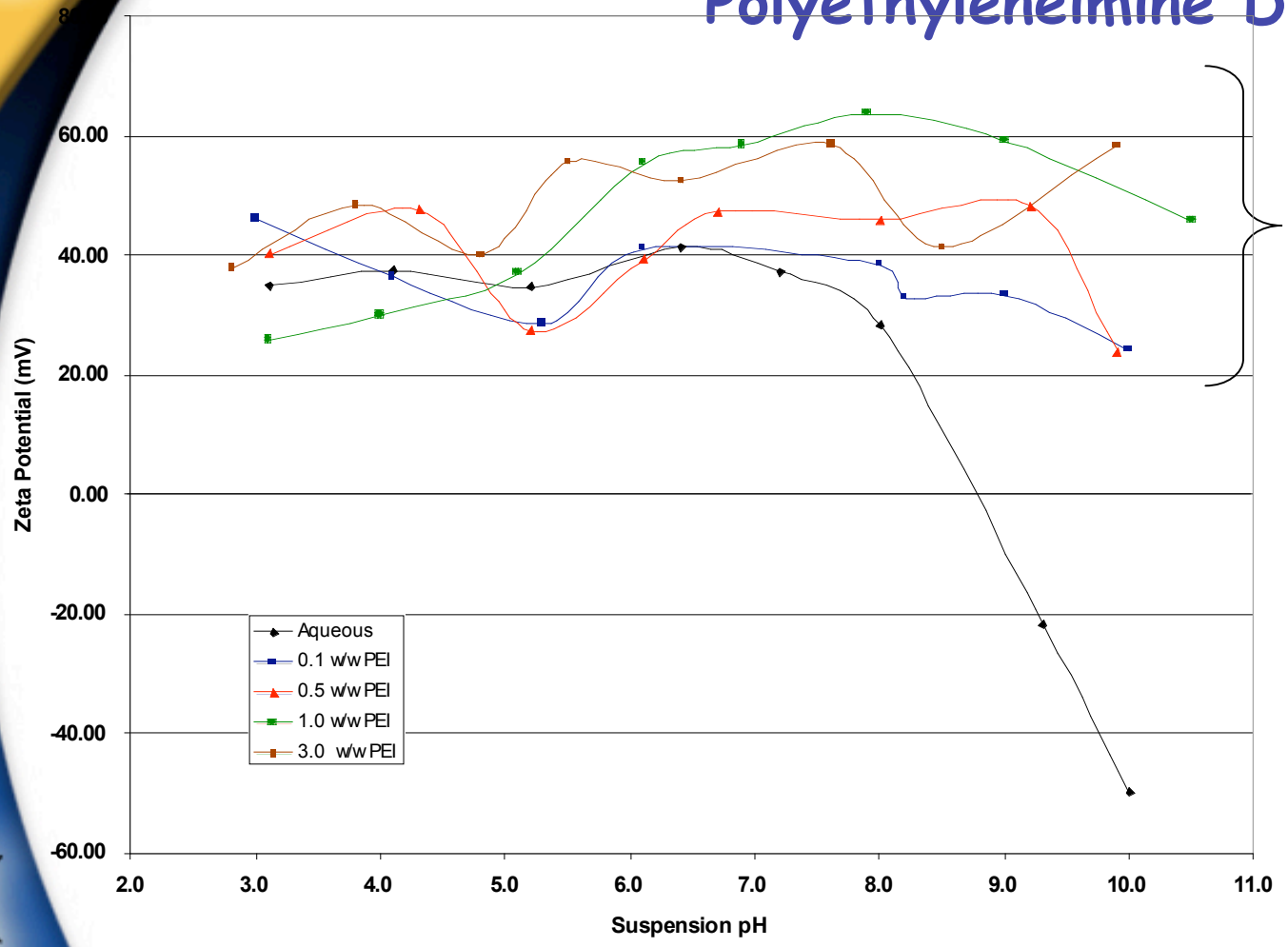
AlN → pH Control, Succinic Acid, Polyethyleneimi

Al_2O_3 → pH Control, Succinic Acid, Polyethyleneimi

Yb_2O_3 → pH Control, Oxalic Acid, Polyethyleneimine



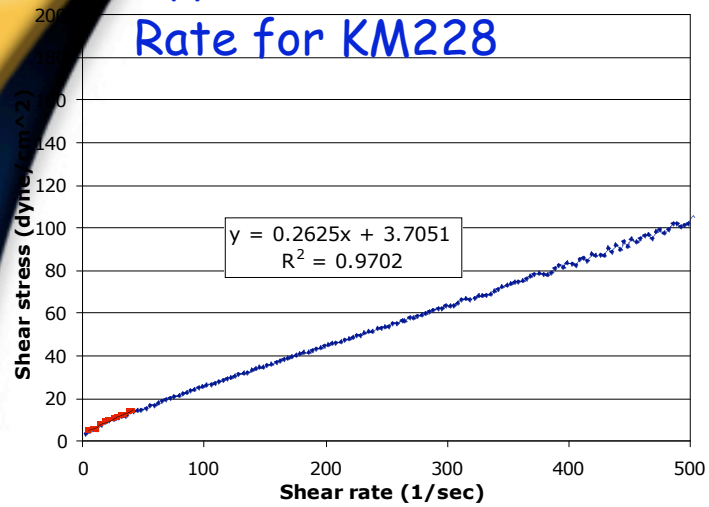
Zeta Potential of Silicon Nitride in Water with Various Concentrations of the Polyethyleneimine Dispersant



Positive Charge

Results for a 40 v/o Slurry

Typical Shear Stress-Shear Rate for KM228



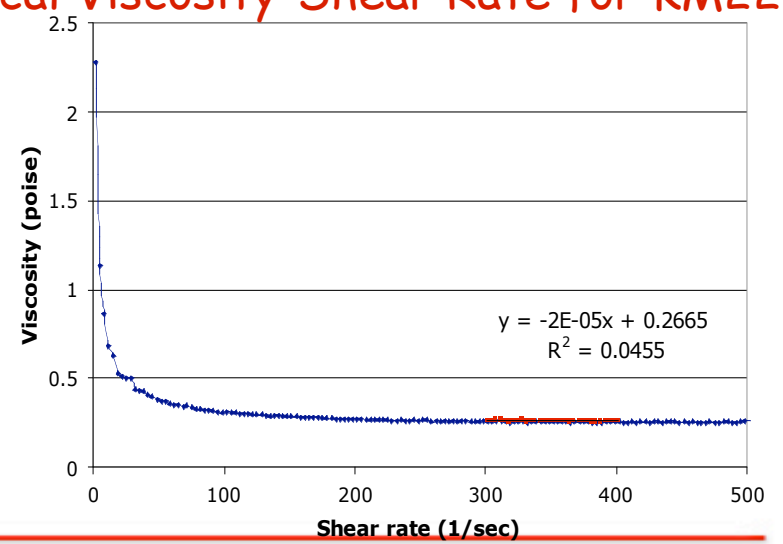
Average Bingham Yield Point ± 95% Confidence Interval

Trial	τ_B (dyn/cm^2)
1	5.5
2	3.7
3	4.2
Average	4.5±2.2

Average Viscosity ± 95% Confidence Interval

Trial	η_a (cP)
1	23.0
2	21.8
3	26.7
Average	23.8±6.3

Typical Viscosity-Shear Rate for KM228



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Phase 1 Conclusions

- Strength and fatigue resistance meets requirements for a microturbine operating at 1050 C to 1250 C.
- Corrosion behavior comparable to silicon nitride
- The alpha – beta sialon phase stability not fully understood. A new relationship between Yb substitution and lattice parameters proposed.
- A passivation-dispersion approach successful in forming high solids slips with water or isopropanol
  - Improve sintering uniformity for complex shapes
  - Vibration casting a possibility

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