Additive Manufacturing of SiC Freeform Optical Elements

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OptiPro Systems

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Background

- Contract activated 7/27/2018
- UNC Charlotte (UNCC) chosen as subcontractor for their expertise in chemistry, materials science and current work developing additive techniques for ceramic materials.
- UNCC is experimenting with a powder bed additive manufacturing of silicon carbide (SiC), using a liquid sodium hydroxide (NaOH)-water solution as a binding agent. Which also promotes secondary crystal growth during sintering to reduce porosity and increase strength
- OptiPro has received molded samples which we processed with similar techniques as we would standard SiC.
- These samples were hand molded in a way to attempt to mimic the 3D printing process.

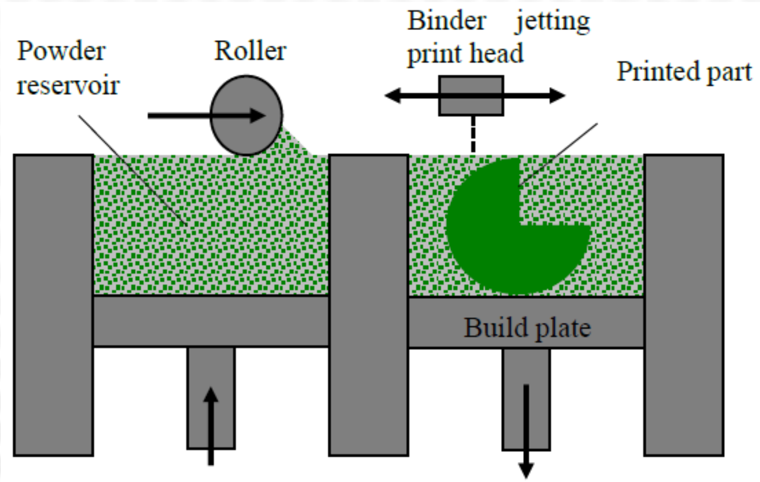

Figure 1: Schematic for powder bed liquid binder jetting.

Initial work

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- After initial molded samples sent to OptiPro, UNCC has been experimenting with different mixtures of SiC powder particle sizes, pressures of compacting the powder, binder solutions and sintering profiles. To develop the best recipe for a ‘SiC-Like’ material.
- The powder and the binder have proven harsh on the standard commercial print head and roller.
- Optipro procured ZCorp powder bed printer, similar model to which UNCC has. Allowing us to mimic work done by UNCC while using our expertise in machine design to develop and integrate more robust components to allow full function of the machine using SiC powder and NaOH solution binders.
- UNCC did some preliminary math on speeds and flowrates for applying binder.

Initial Work

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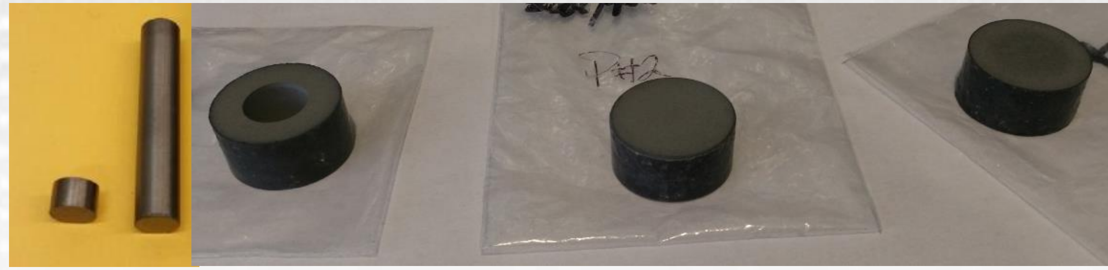
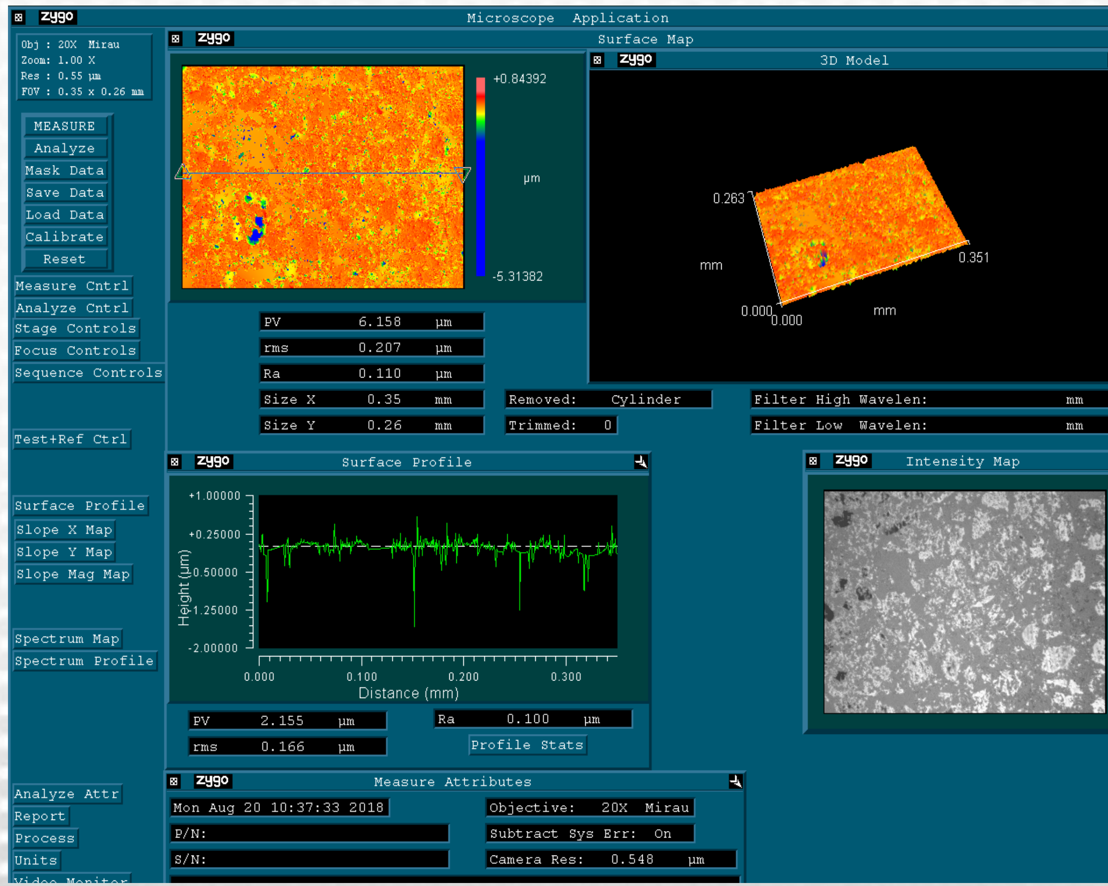
- OptiPro received 3 samples from the original molded test parts.
- On each one we ground a few millimeters of stock off the top and bottom to get thru the ‘skin’ to a more homogenous layer.
- We experimented with both Optisonic and traditional CNC grinding. We did not iterate or experiment to improve surface yet.
- Right is a screen shot from our Zygo NewView of the Optisonic ground surface





Planned work

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- UNC Charlotte is continuing to work with different recipes and processes to make more sample parts.
  - Investigating a method of using a more standard binder in the print head to print a 'green state' part and applying the NaOH afterwards. If feasible would it would bring us to printing test parts quicker.
- OptiPro is refurbishing the Zcorp powder bed printer acquired to retrofit it to be able to print with the powders and binders prescribed by UNCC
  - Investigate micro jets, sprayers and droppers to create a print head that can deliver the NaOH binder and plan to integrate it on our printer.
  - Investigate improvements to the roller, to be able to handle and deliver consistent layers of SiC powder.
- Once we are able to manufacture test samples we will start further evaluation of strength and material properties, including sending them out for CTE evaluation.
- Once OptiPro is able to start processing samples we can work with both the initial recipe and grinding/polishing processes to attempt to create an optical surface on a grown part.

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Effect of processing parameters on microstructure density, and mechanical properties.

Presented by

Ahmed El-Ghannam, PhD

Amazon crystals are initiated at the silica gel layer

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- A (1-40µm) SiC powder was mixed with 6.25 wt% (< 80 nm) nano powder.
- The surface of the SiC particles was chemically treated with 500µL of 5 wt% NaOH and shaped manually into discs (10 mm dia and 25mm height)
- The SiC discs were heated (1 C/min) in air at 900C for 2 hrs

Effect of NaOH Concentration on the surface chemistry

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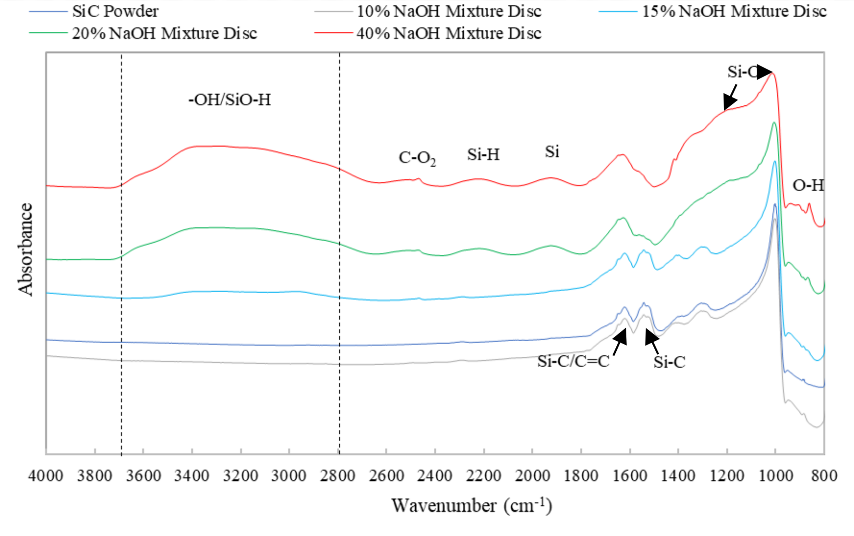

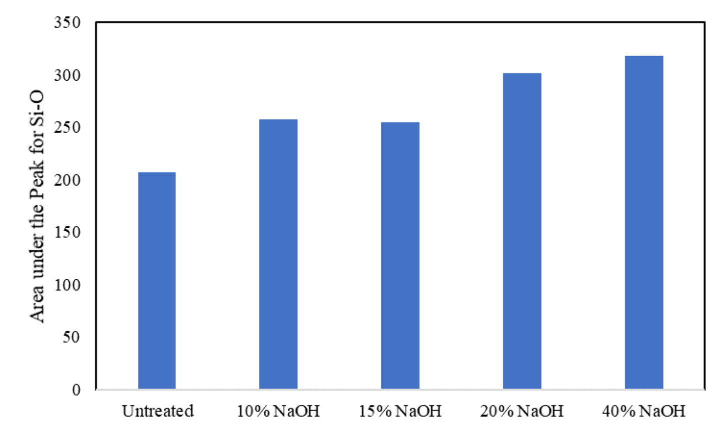

Effect of NaOH concetration on the Surface Charge

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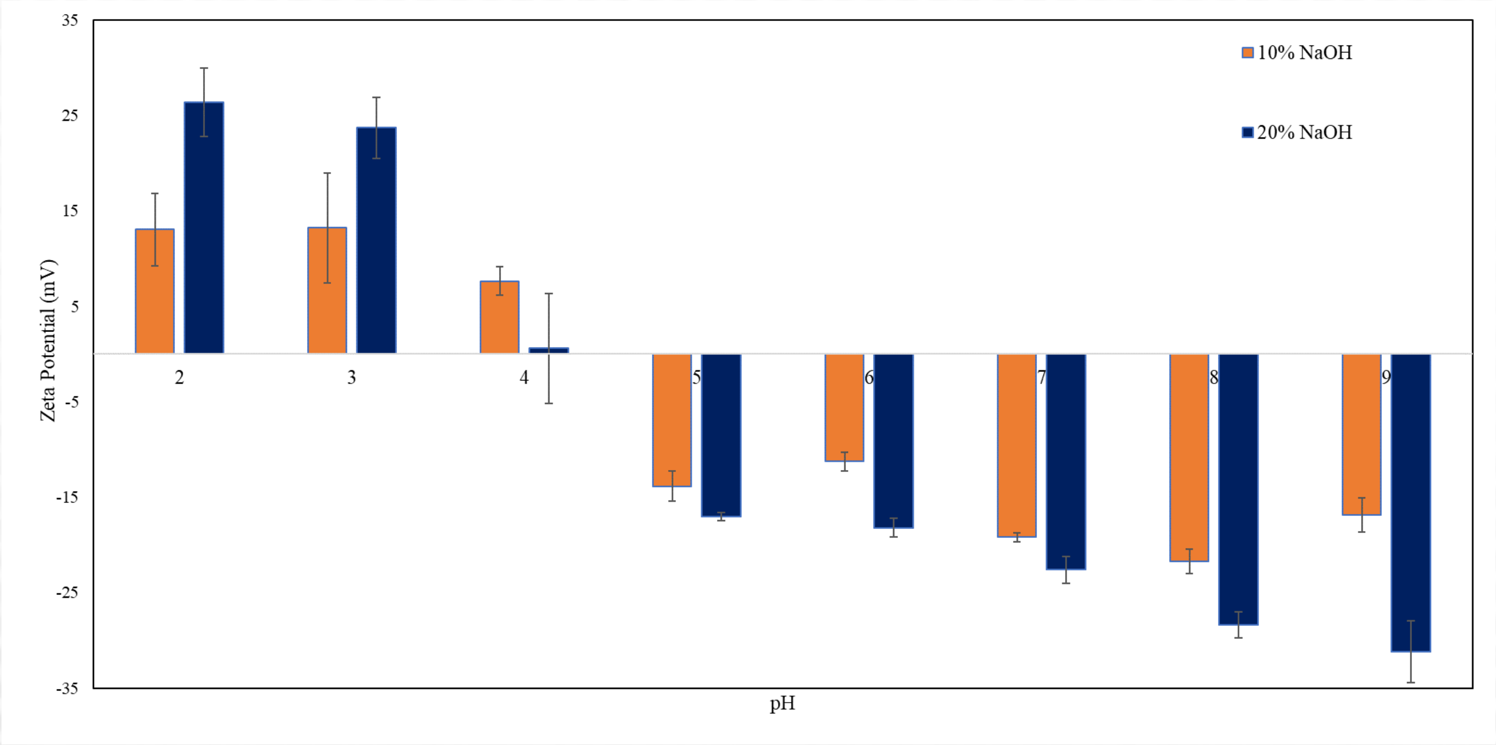

Amazon Crystals

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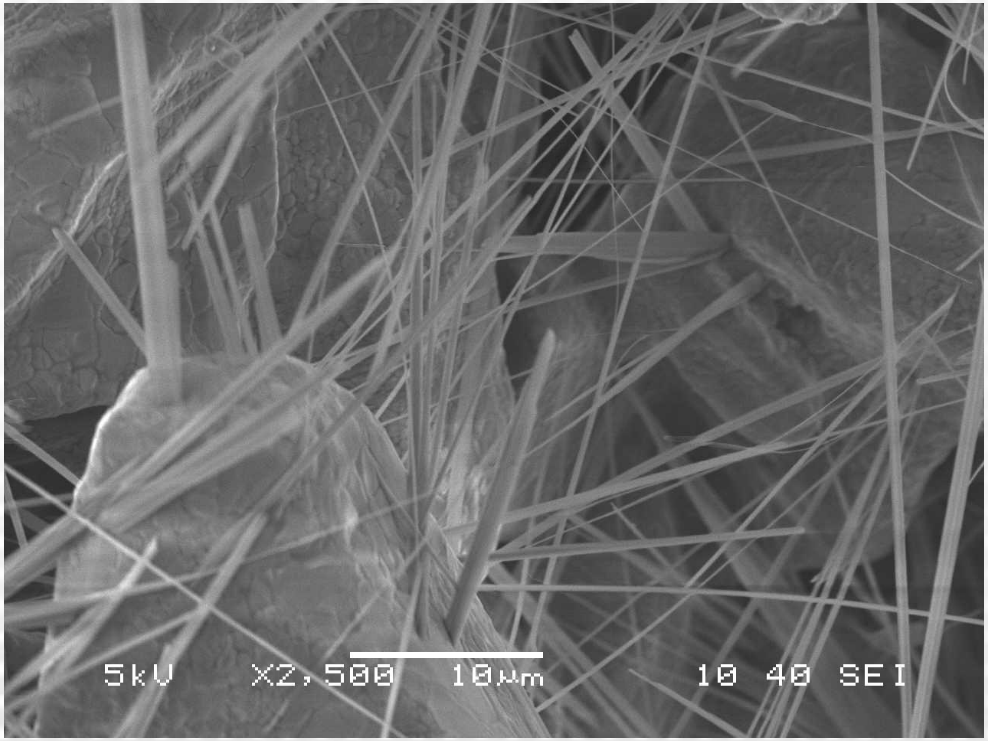
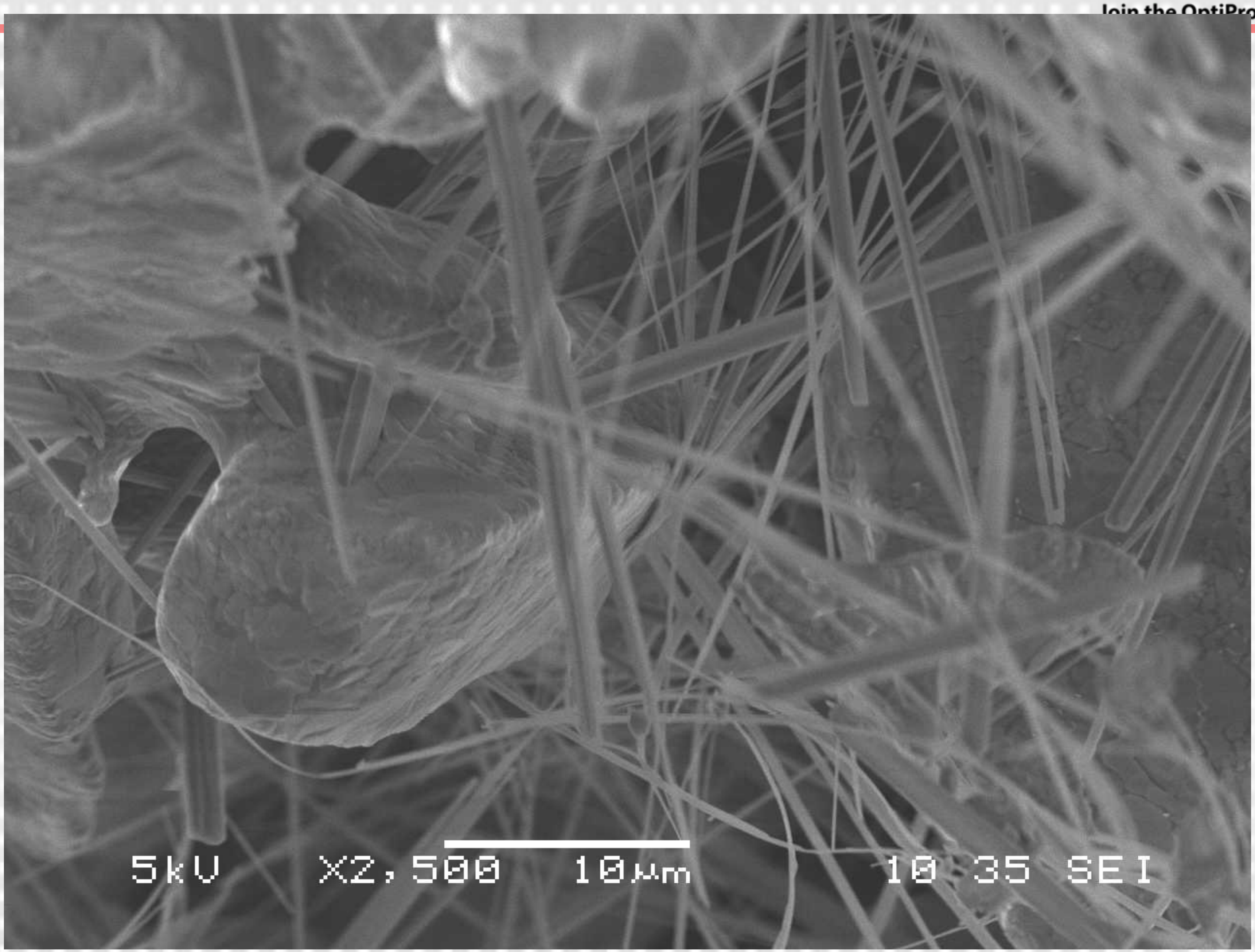
- During heat treatment, a secondary crystal growth occurs bridging the gaps between SiC particles

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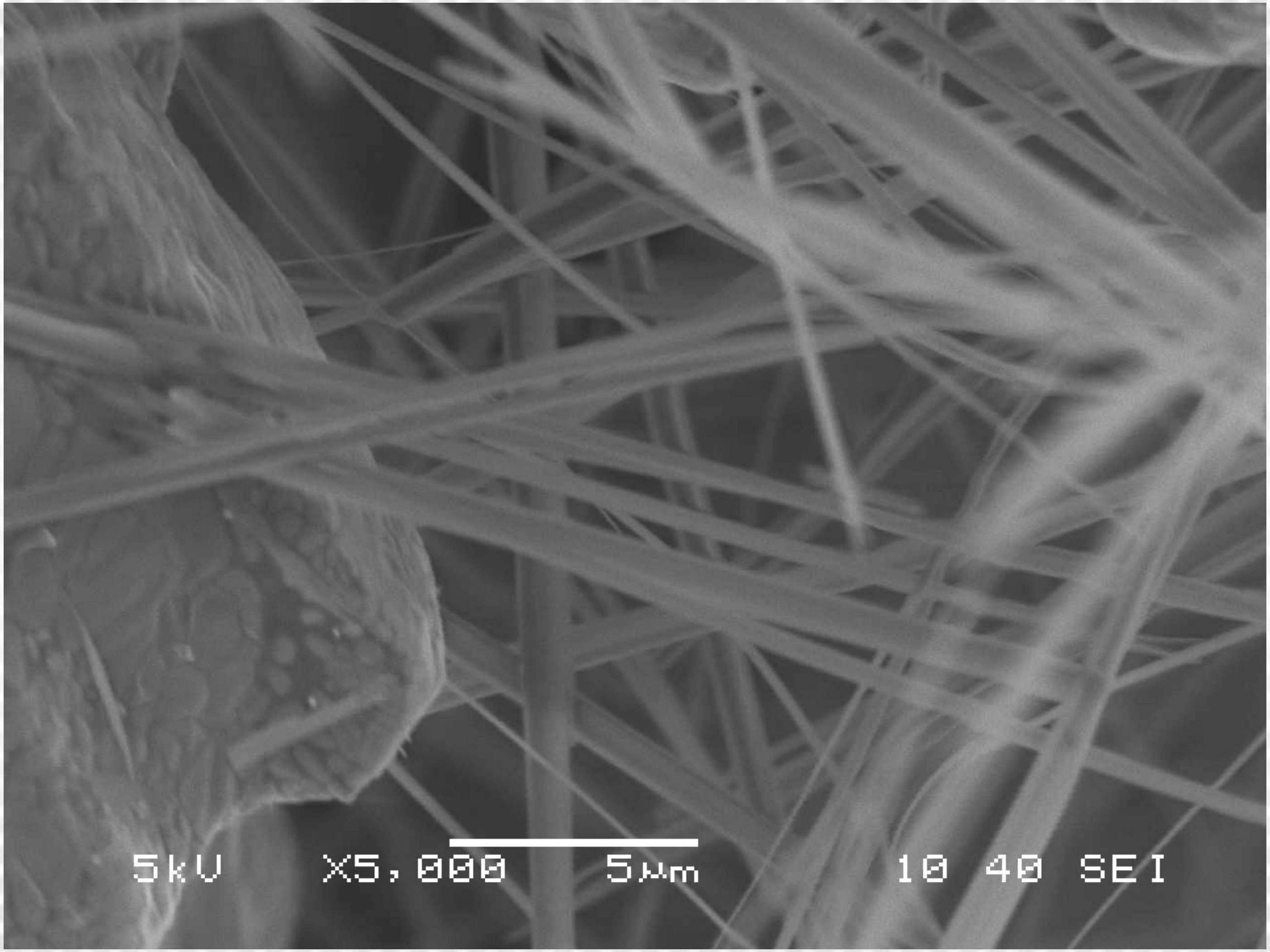
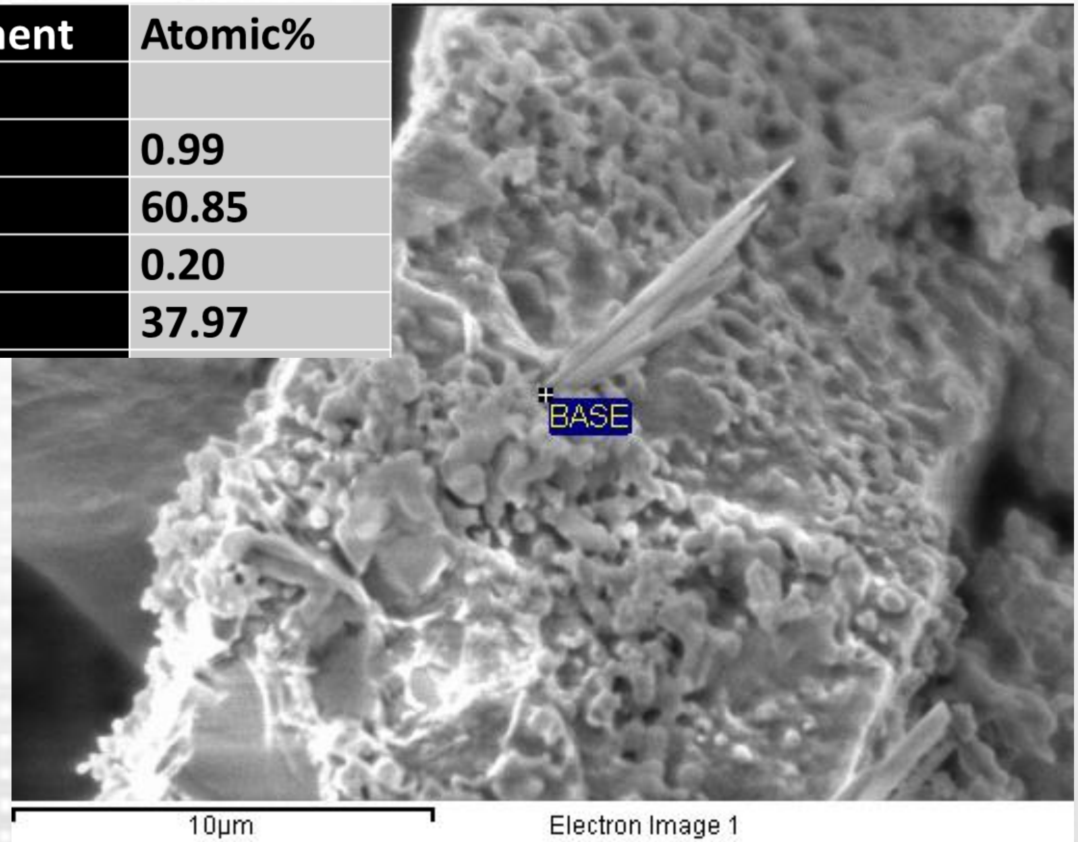

5kV X5,000 5μm 10 40 SEI

Amazon crystal growth on the surface of SiC

5% NaOH - 900/2hrs – EDX analysis of the bas

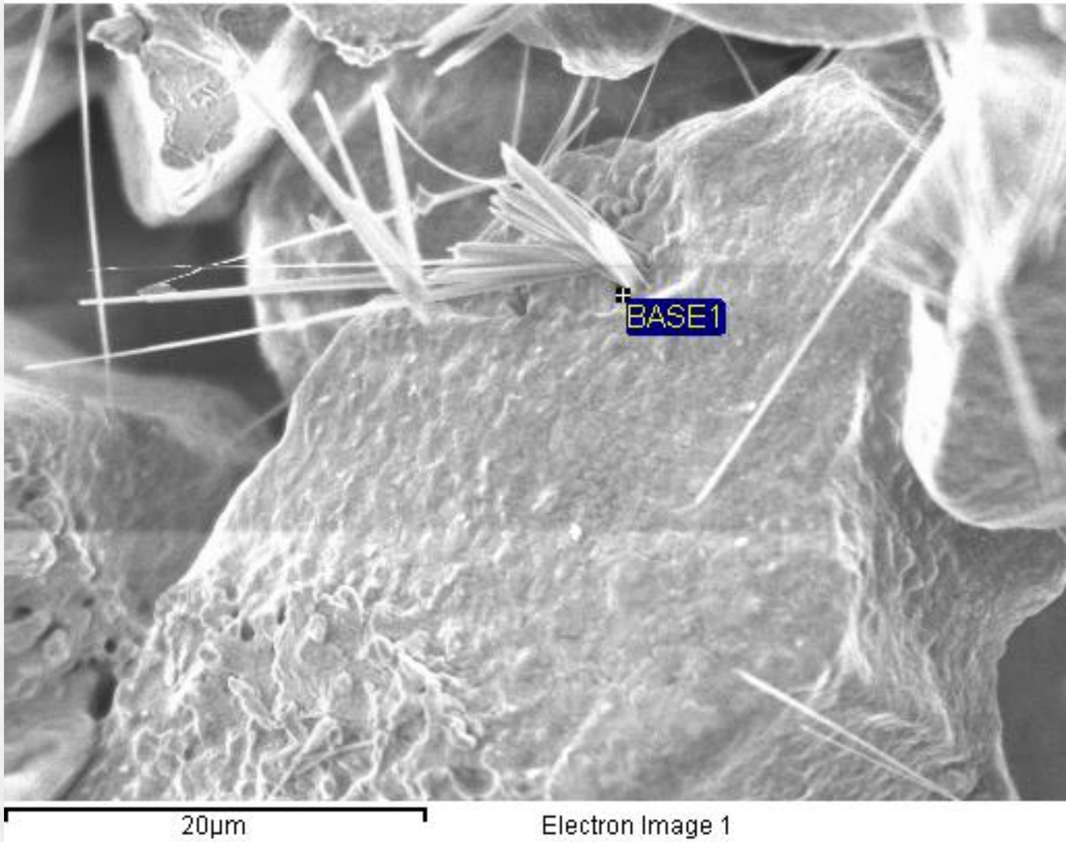
Element	Atomic%
C	0.99
O	60.85
Na	0.20
Si	37.97



- These amazon crystals originate from the gel layer rather than from the silicon carbide particle itself

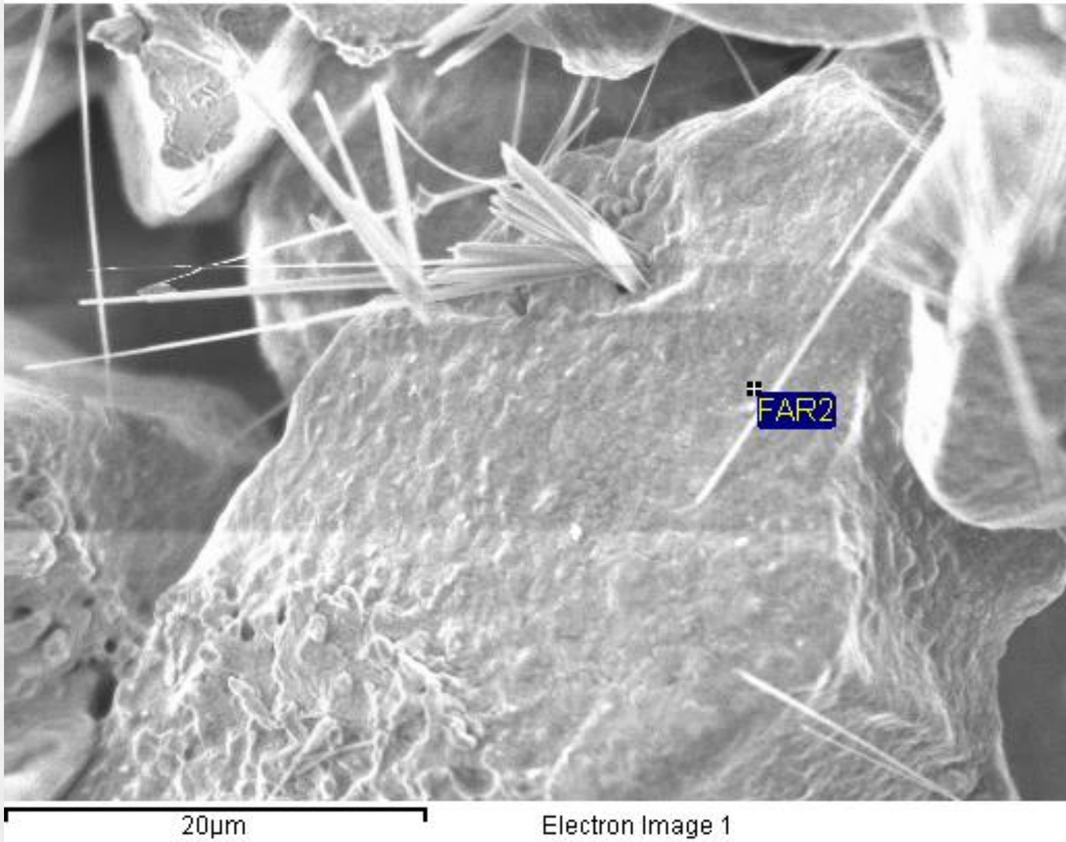
5% NaOH 900/2H - Base of crystal

Element	Atomic%
C	5.13
O	58.55
Na	0.77
Si	35.54



5% NaOH 900/2H - Away from crystal growth (6.812 µm)

Element	Atomic%
C	6.60
O	59.11
Na	0.50
Si	33.78



Effect of NaOH conc: Mechanical properties

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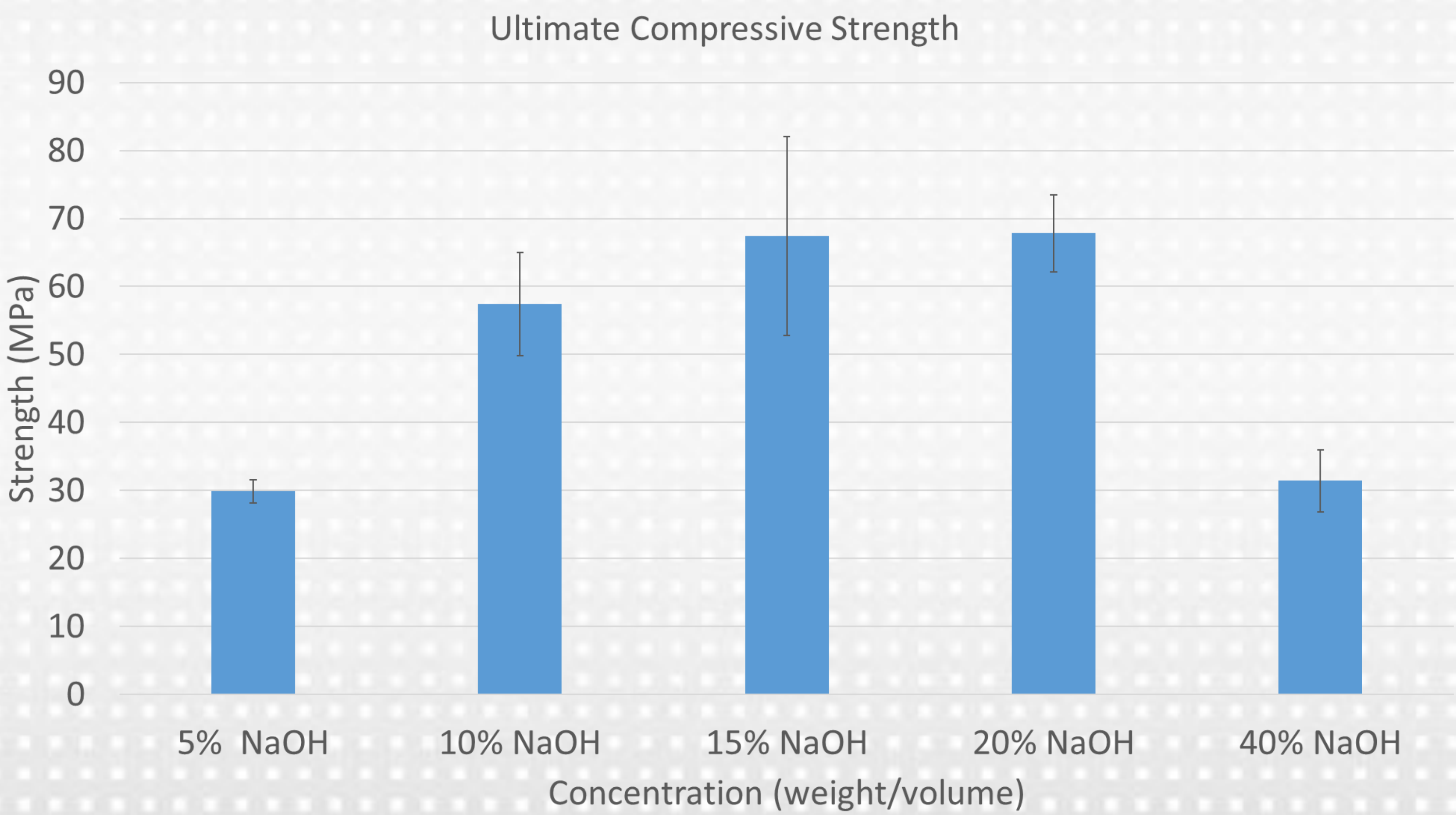
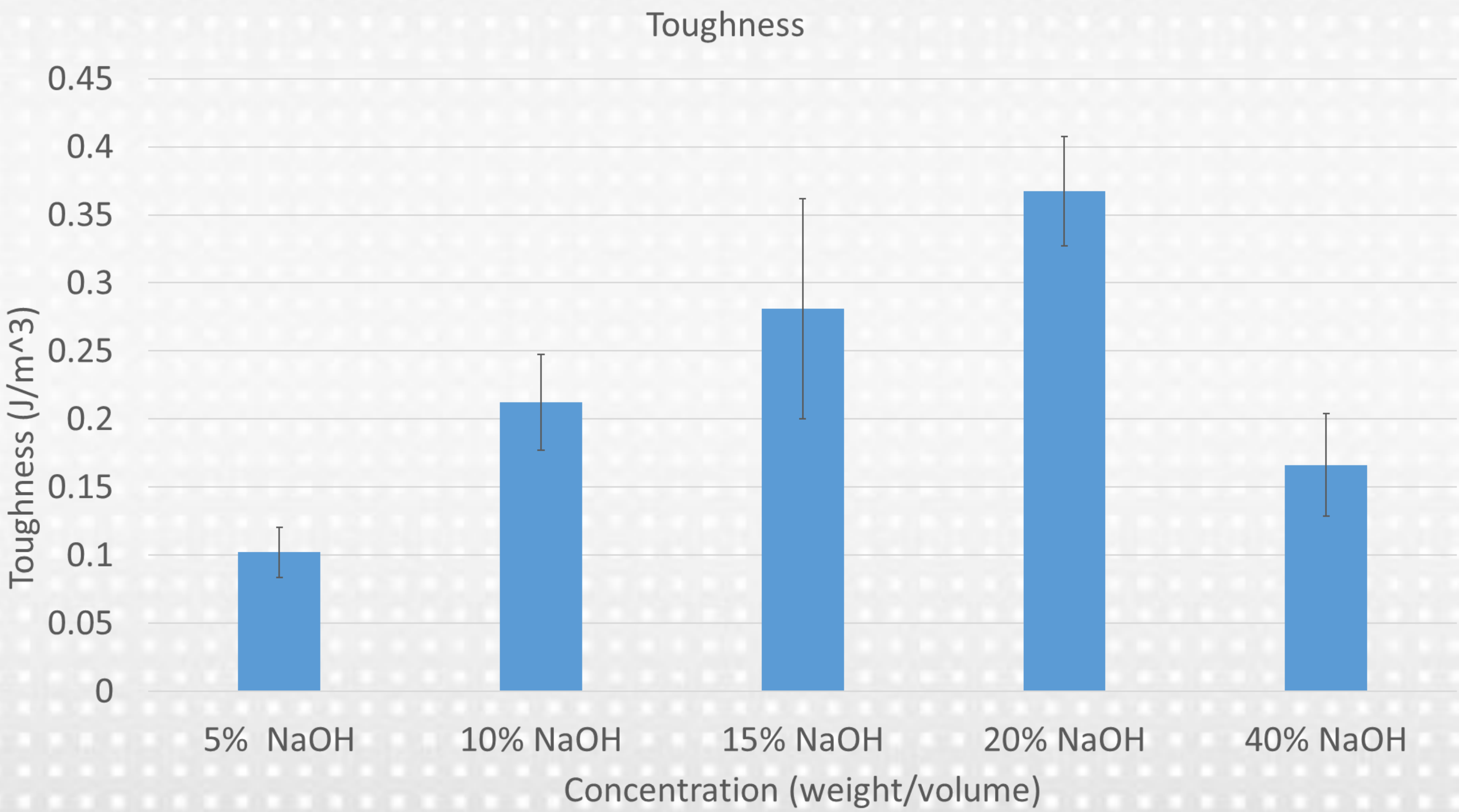
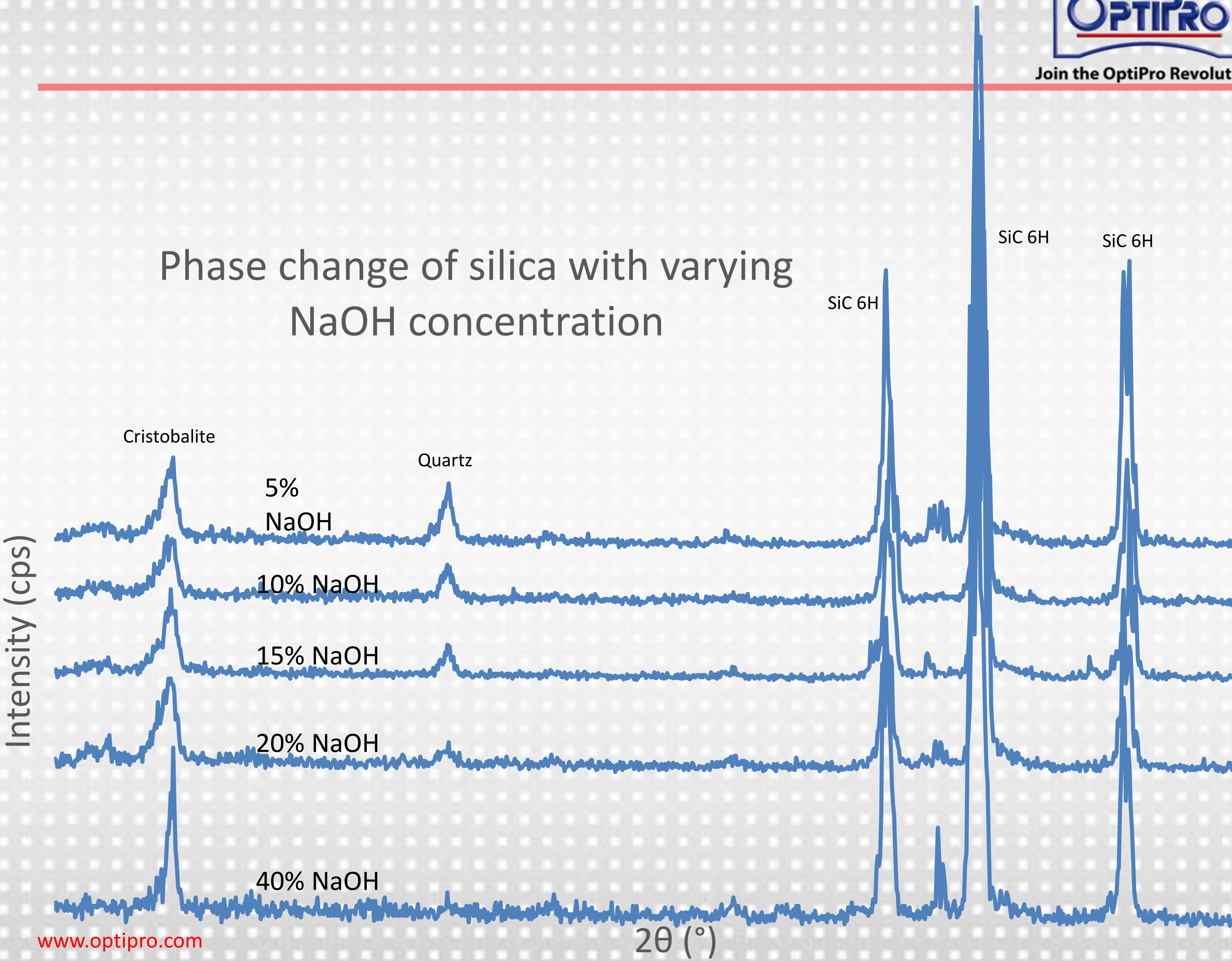


Effect of NaOH conc: Mechanical properties

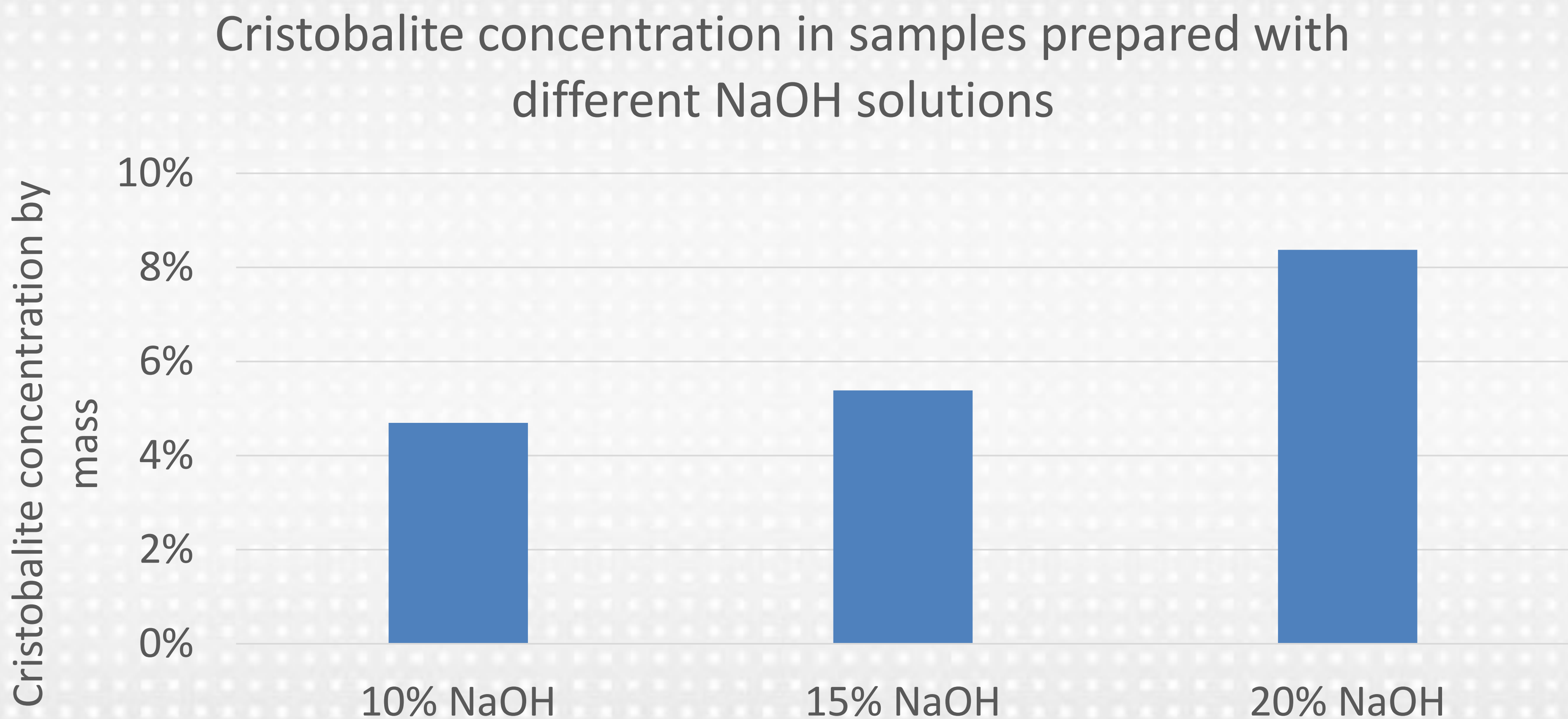


Phase change of silica with varying NaOH concentration



Phase change of silica dependence on NaOH concentration

NaOH Concentration	Percent increase in the intensity of Cristobalite XRD signal compared to that of Quartz
5% NaOH	140%
10% NaOH	180%
15% NaOH	250%
40% NaOH	771%



Cristobalite concentration in samples prepared with different NaOH solutions
Cristobalite concentration by mass
10%
8%
6%
4%
2%
0%
10% NaOH
15% NaOH
20% NaOH

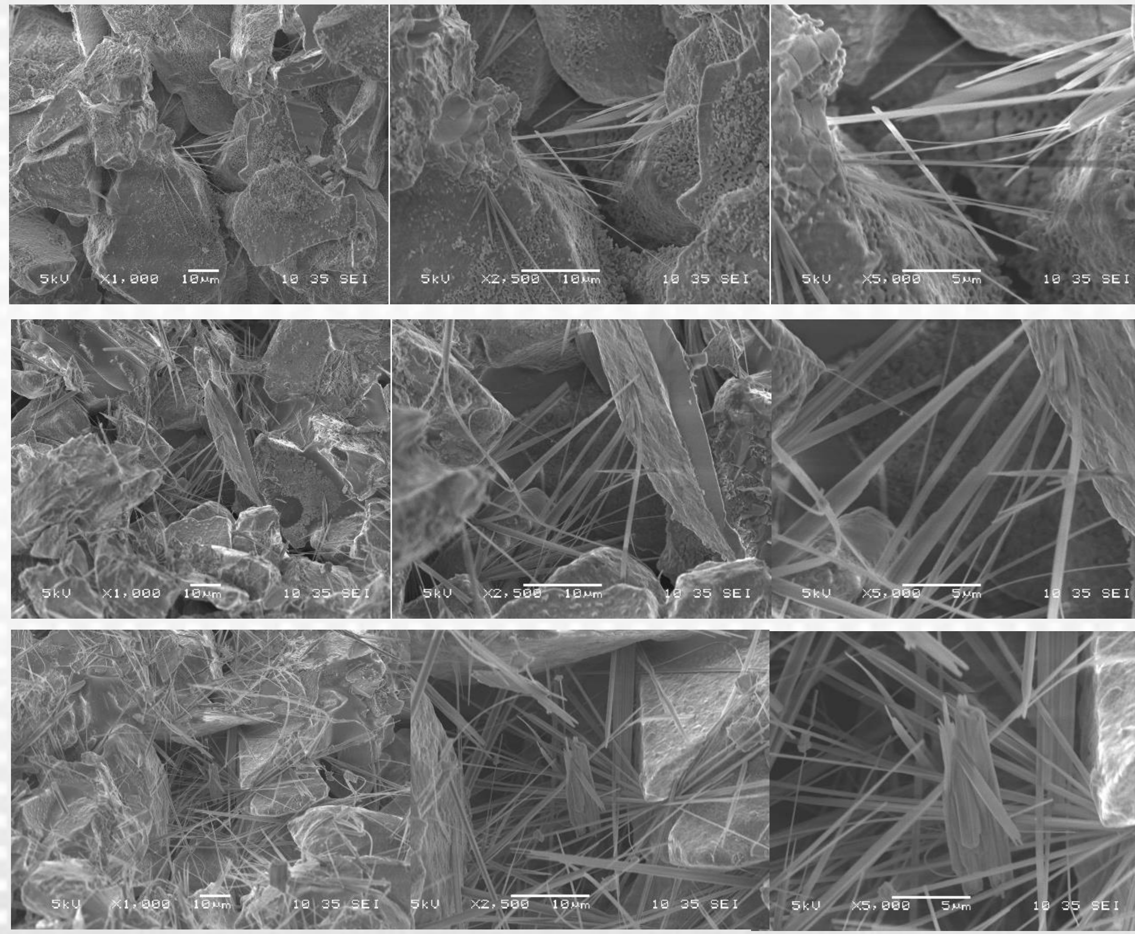
Fracture Surface Analysis after the mechanical test showing Amazon crystal growth in SiC samples treated with 5, 10 and 15% NaOH

1000X 2500X 5000X

5% NaOH 900C/2h

10% NaOH 900C/2h

15% NaOH 900C/2h



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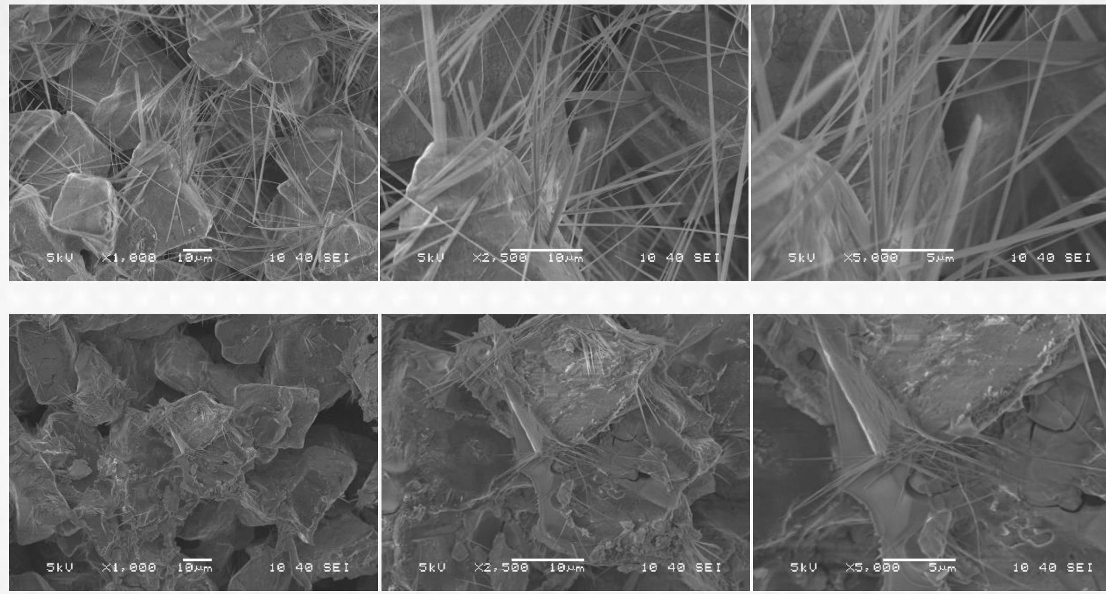
1000X 2500X 5000X

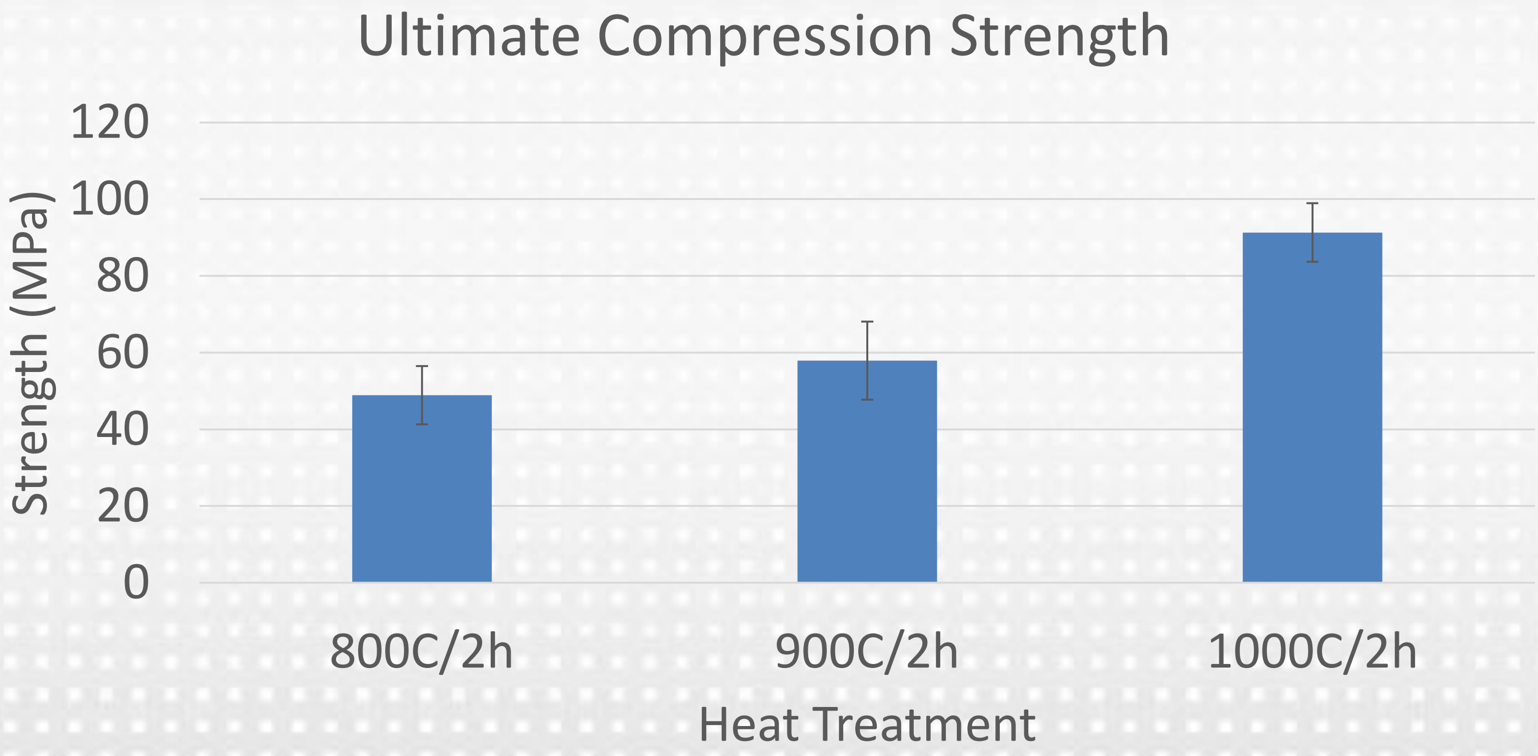
Fracture surface analysis after the mechanical test showing Amazon crystal growth in SiC samples treated with 20, and 40% NaOH

20% NaOH
900C/2h

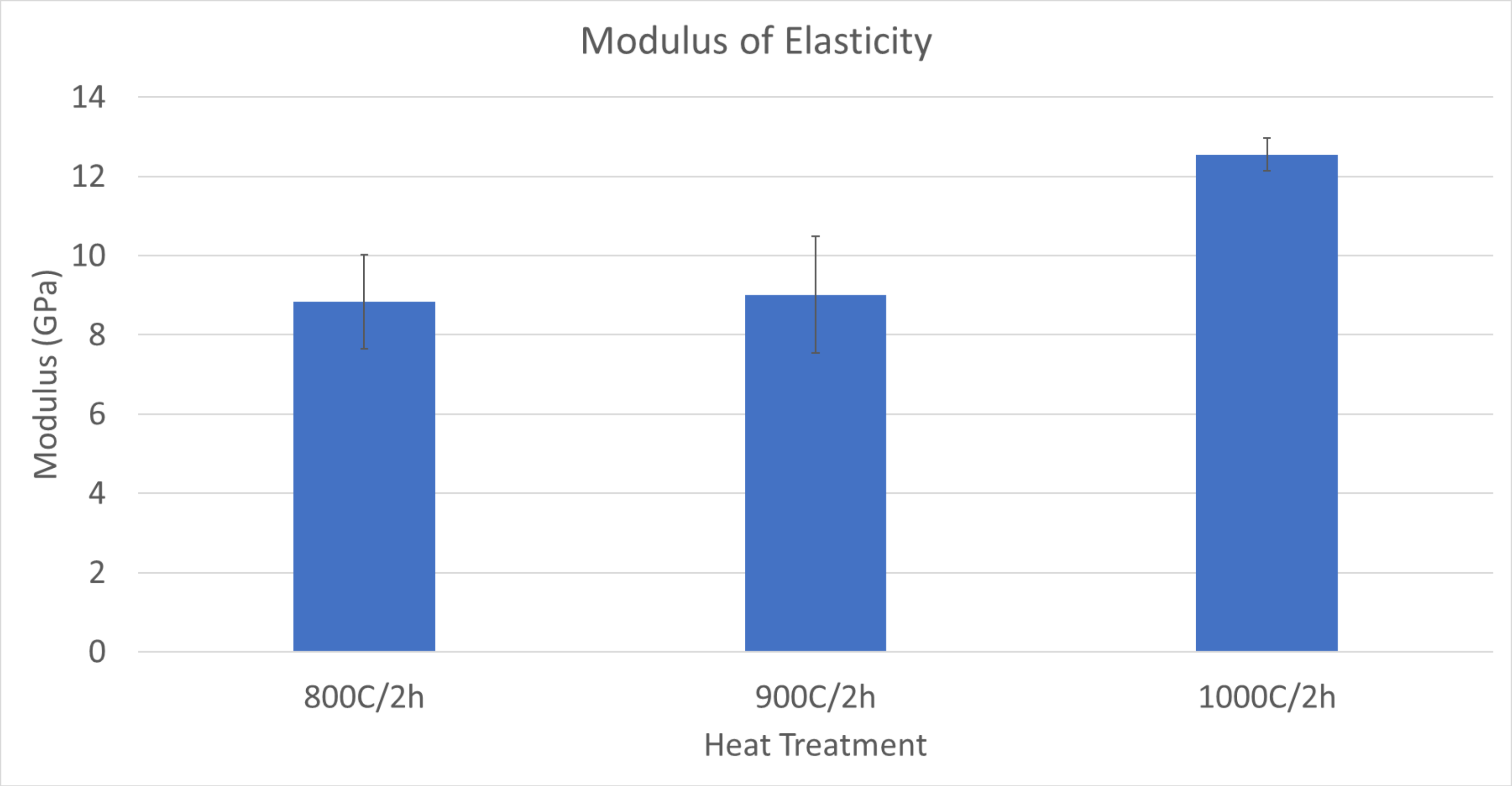
40% NaOH
900C/2h



Effect of heat treatment temperature: Mechanical properties



Effect of heat treatment temperature: Mechanical properties



Effect of heat treatment temperature: Mechanical properties

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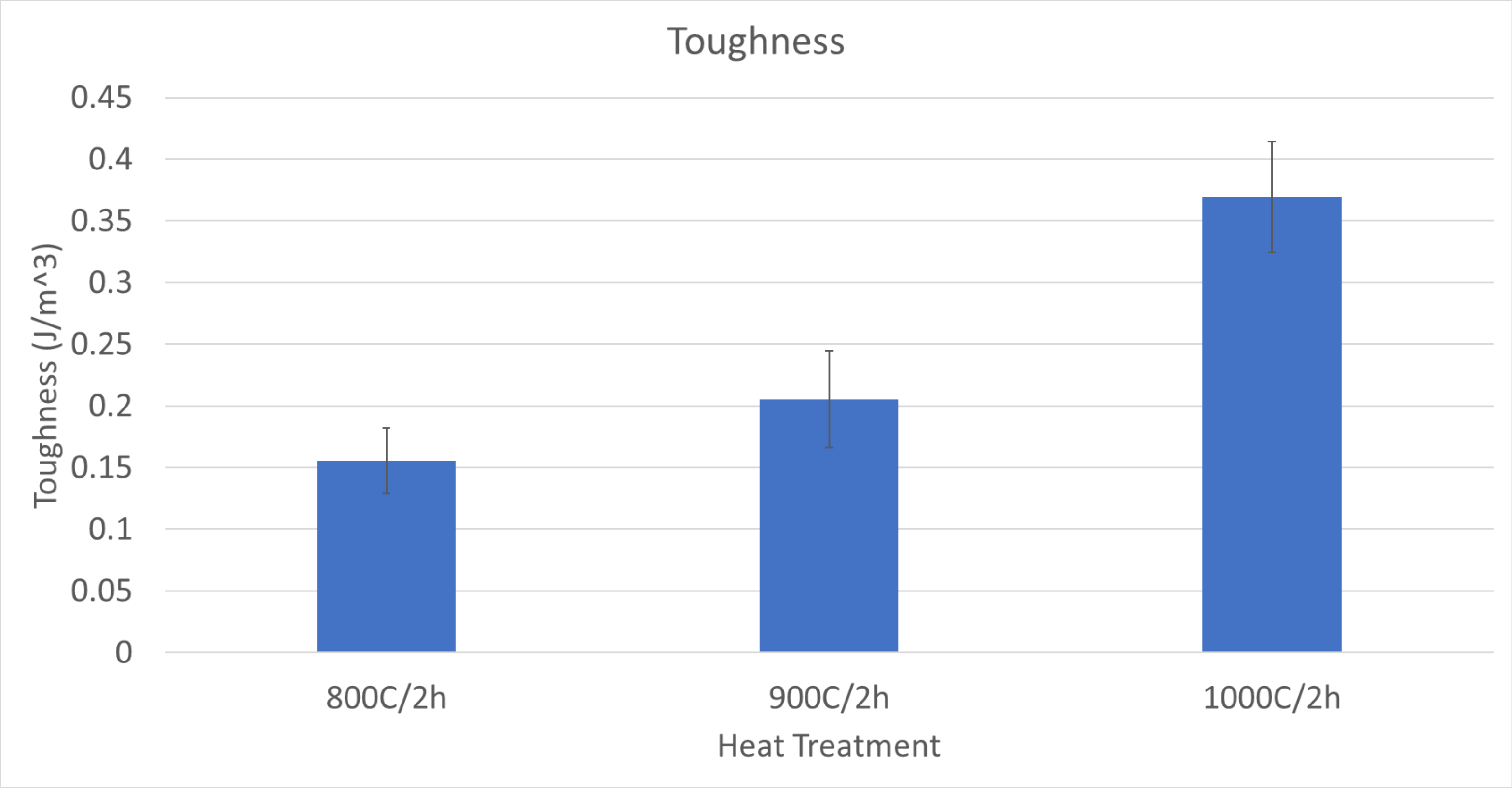
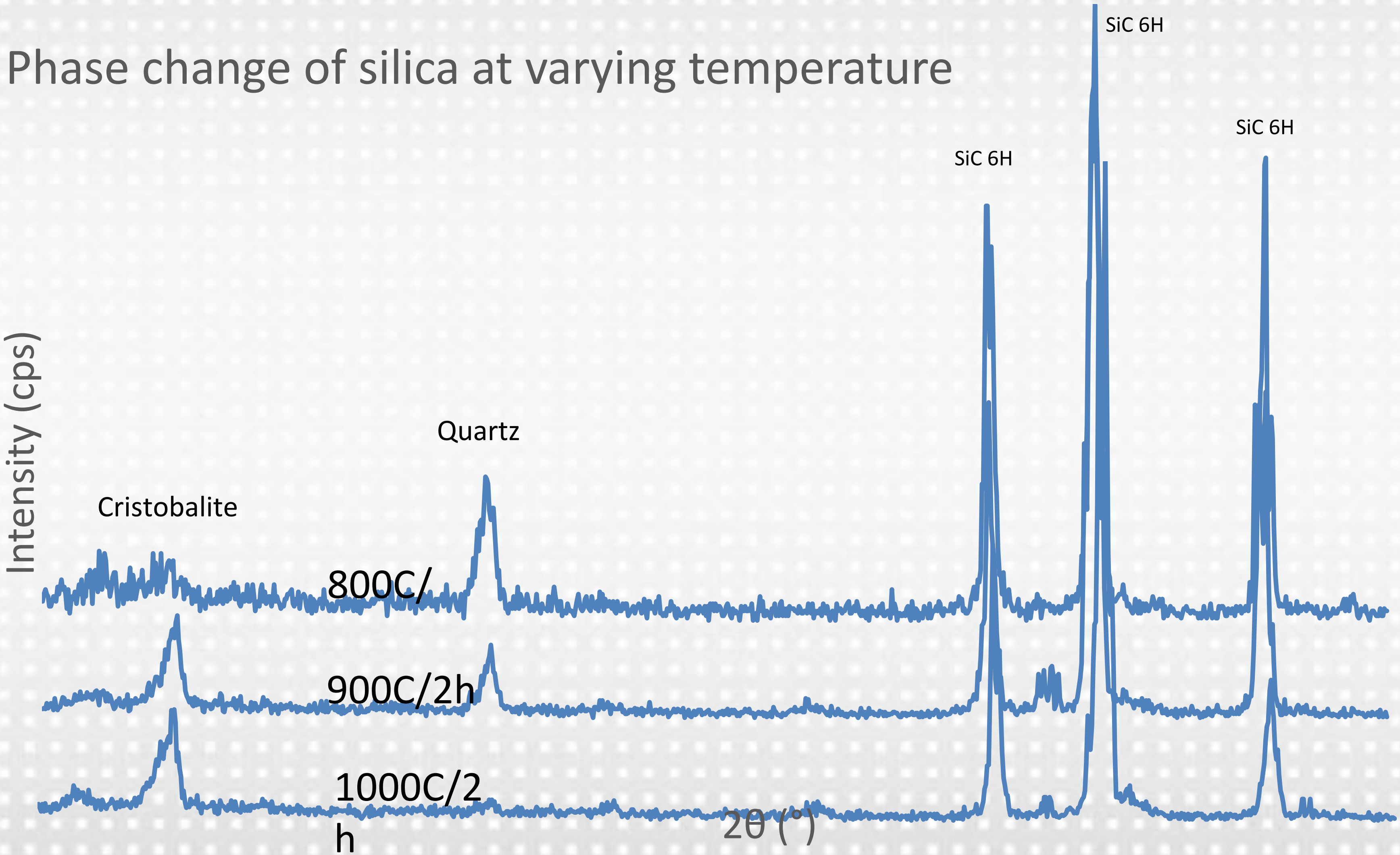


Phase change of silica at varying temperature



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Phase change of silica dependence on heat treatment temperature

Heat Treatment Temperature	Percent increase in the XRD intensity of Cristobalite compared to that of Quartz
800C/2h	42%
900C/2h	139%
1000C/2h	625%

Fracture Surface showing crystal growth at different temperatures

	1000X	2500X	5000X

800C/2h
10% NaOH
Quartz

900C/2h
10% NaOH
Quartz and Cristobalite

1000C/2h
10% NaOH
Cristobalit
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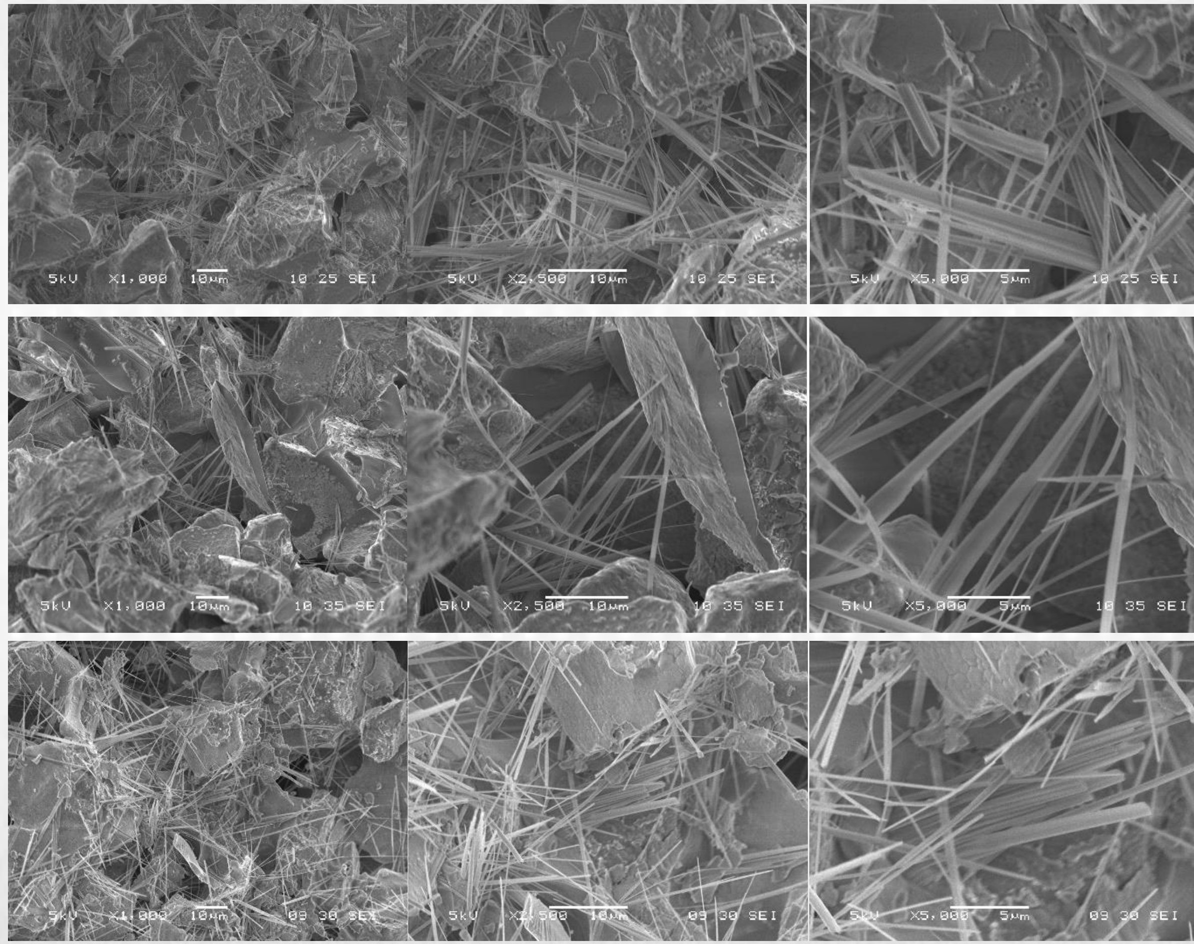

Conclusion

- Growth of silica crystals is favored at lower temperature
- Transition of Quartz to Cristobalite occurs at temperature between 800-900 C
- Future work would include growing the crystal first at low temperature then transforming them to Cristobalite