High Temperature Water as a Clean and Etch of SiO_2 Films

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Rationale

- High temperature water (HTW) has demonstrated some interesting capabilities etching SiN (SPCC 2015)
- Given its enhanced reactivity, we were interested in the selectivity of HTW for SiN versus SiO_2

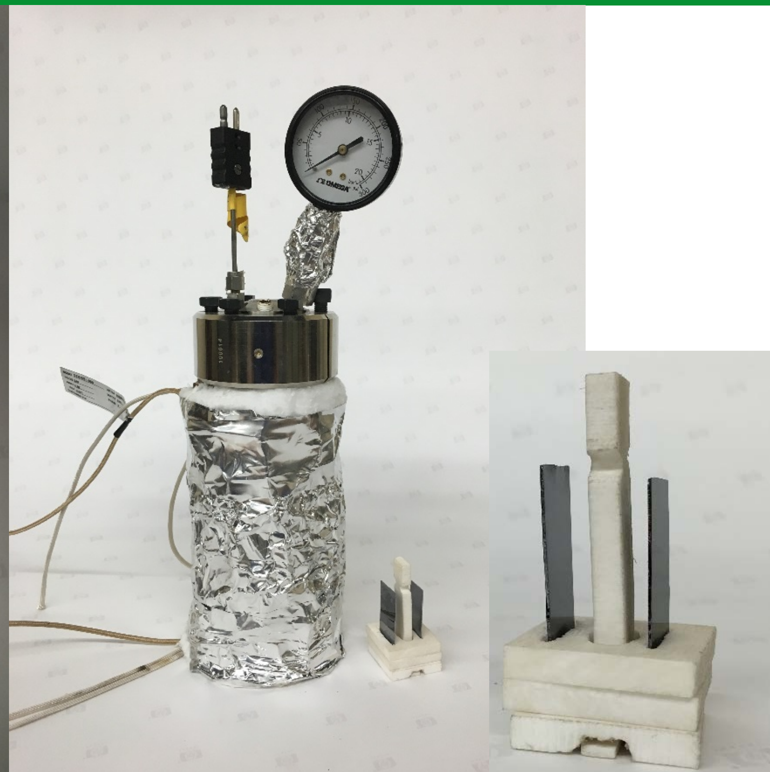
Outline

- Description of Experimental Apparatus and Conditions
- Characterization of SiO_2 wafers to examine the behavior following exposure to HTW
  - Ellipsometry—determine thickness changes
  - FTIR—changes in the chemistry and thickness
  - Profilometry—define boundaries and approximate changes in thickness

Experimental Apparatus

Heating mantle and reactor

Reactor can be easily removed from mantle

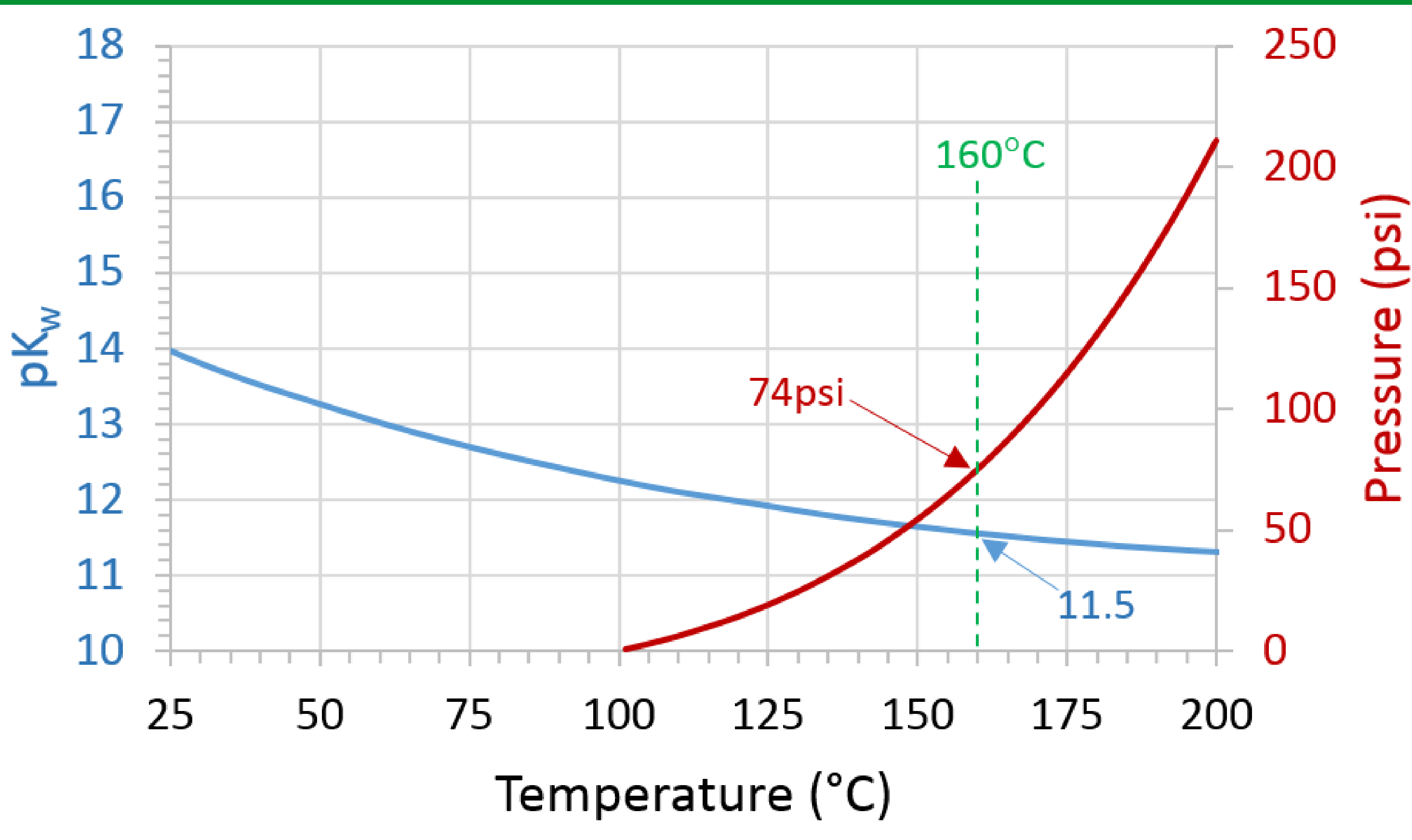


Reactor and sample holder

Experimental Conditions

- DI water heated to 98°C
- SiO_2/Si samples placed in the reactor and reactor closed (~45sec)
- Temperature set to 160°C for 5, 10, and 20 minutes (heat up takes ~17min)
- Reactor removed from heat and quenched in water for 2 minutes
- Reactor opened (~45sec)
- Sample stand is removed
- Samples rinsed with DI water and allowed to dry.

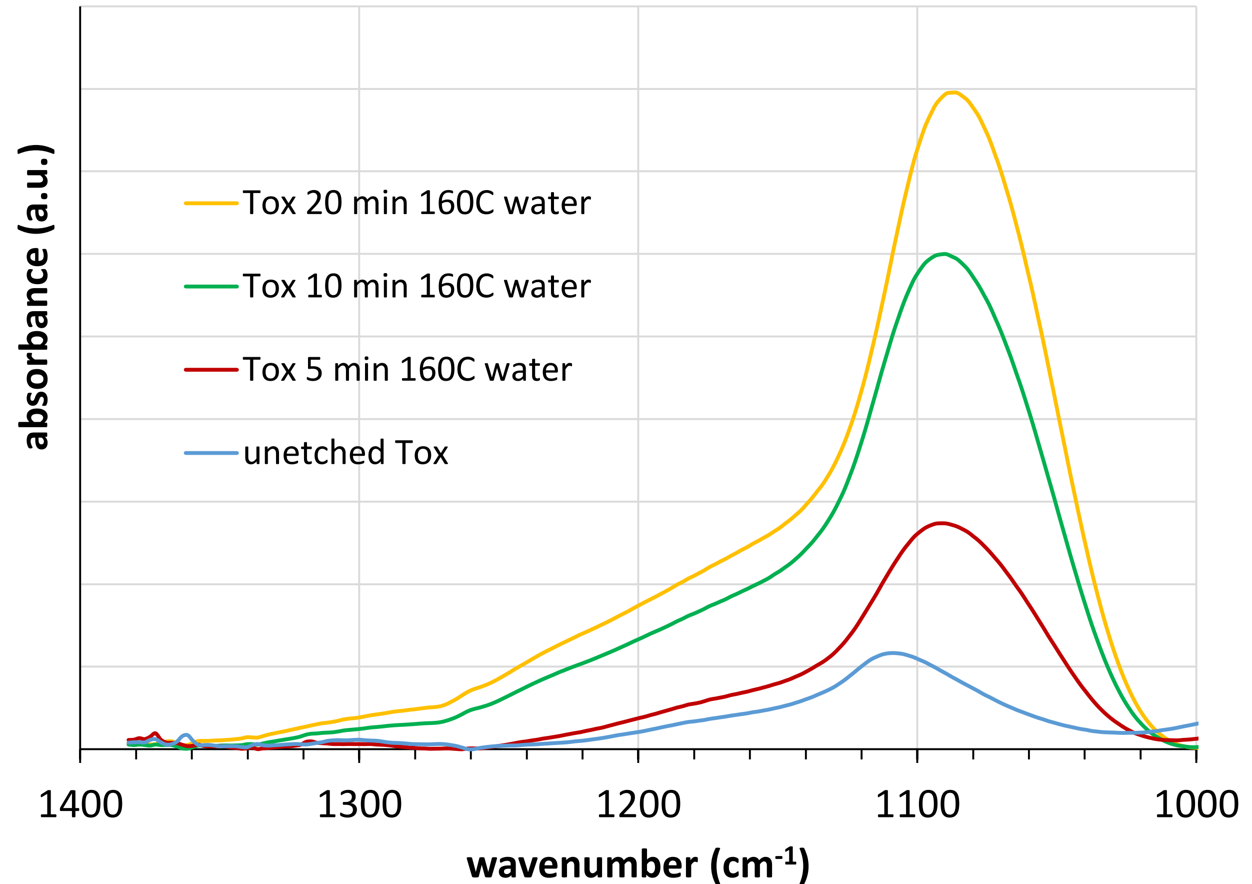
What are the Experimental Conditions?



- $pK_w = -\log[K_w] = -\log [H^+][OH^-]$: at 160°C, water has more ionic species than at room temp

FTIR of oxide exposed to 160°C steam

- FTIR (trans) of the top of the sample (above the water level)
- Shows SiO_2 thickness increasing with longer exposures

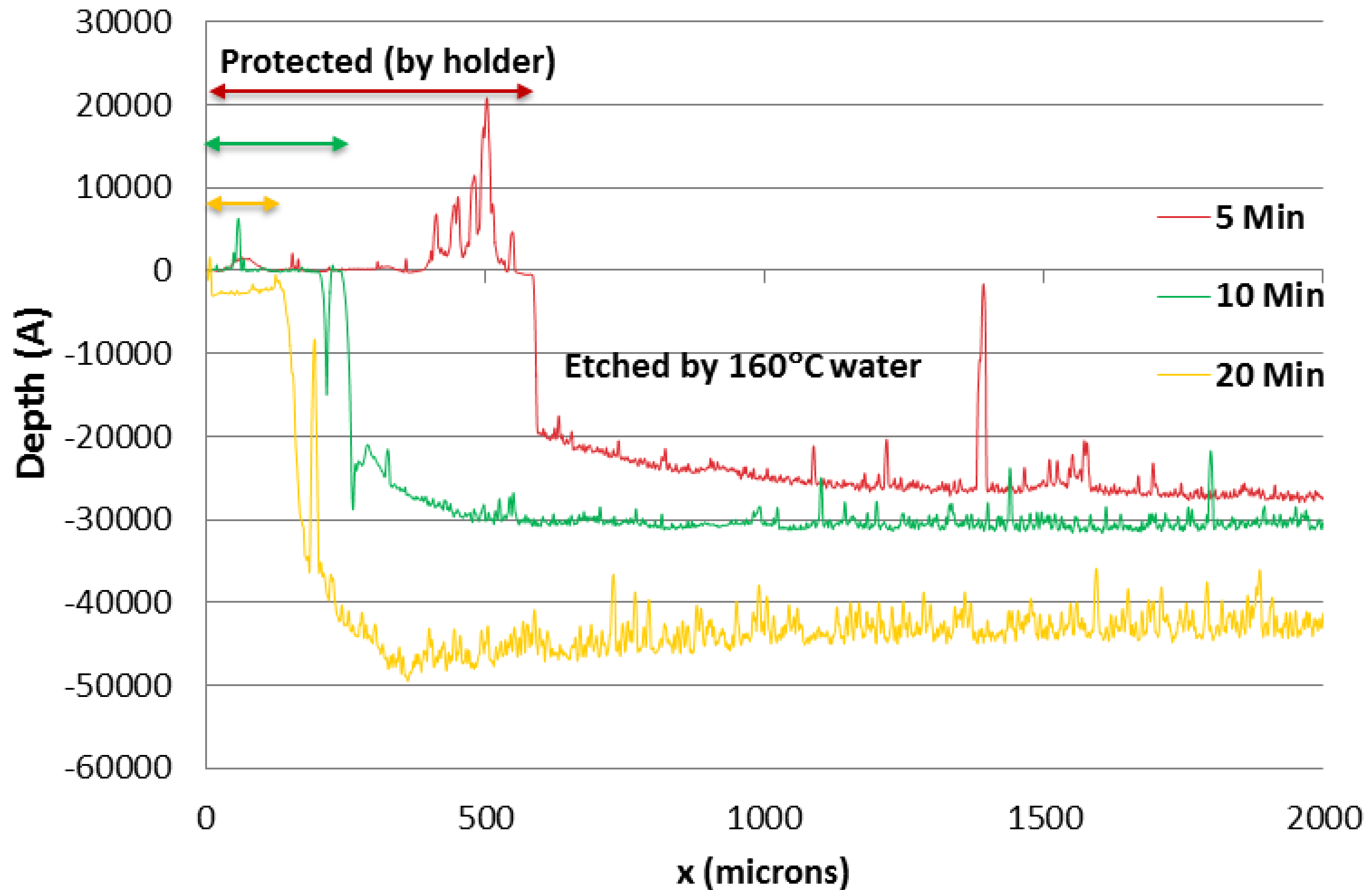


Ellipsometry: oxide exposed to 160°C steam

- Tox oxidized and ↑ thickness
- Δthickness after 5 minutes → ~13nm
- Samples were too roughened after 10 and 20 minutes to model

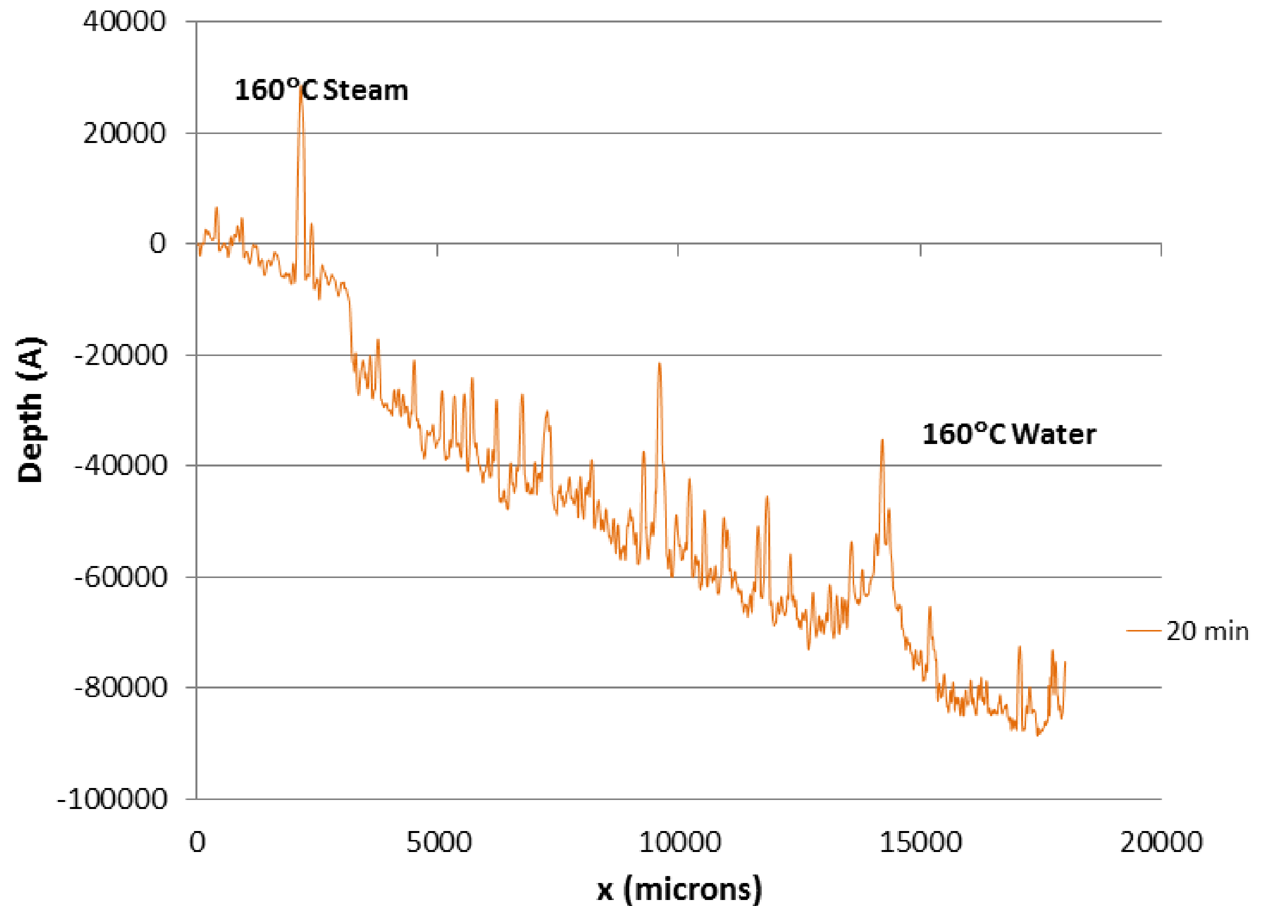
Profilometry Profile: from protected to exposed to 160°C Water

- Region exposed to HTW showed etching
- Etch depth increased with time



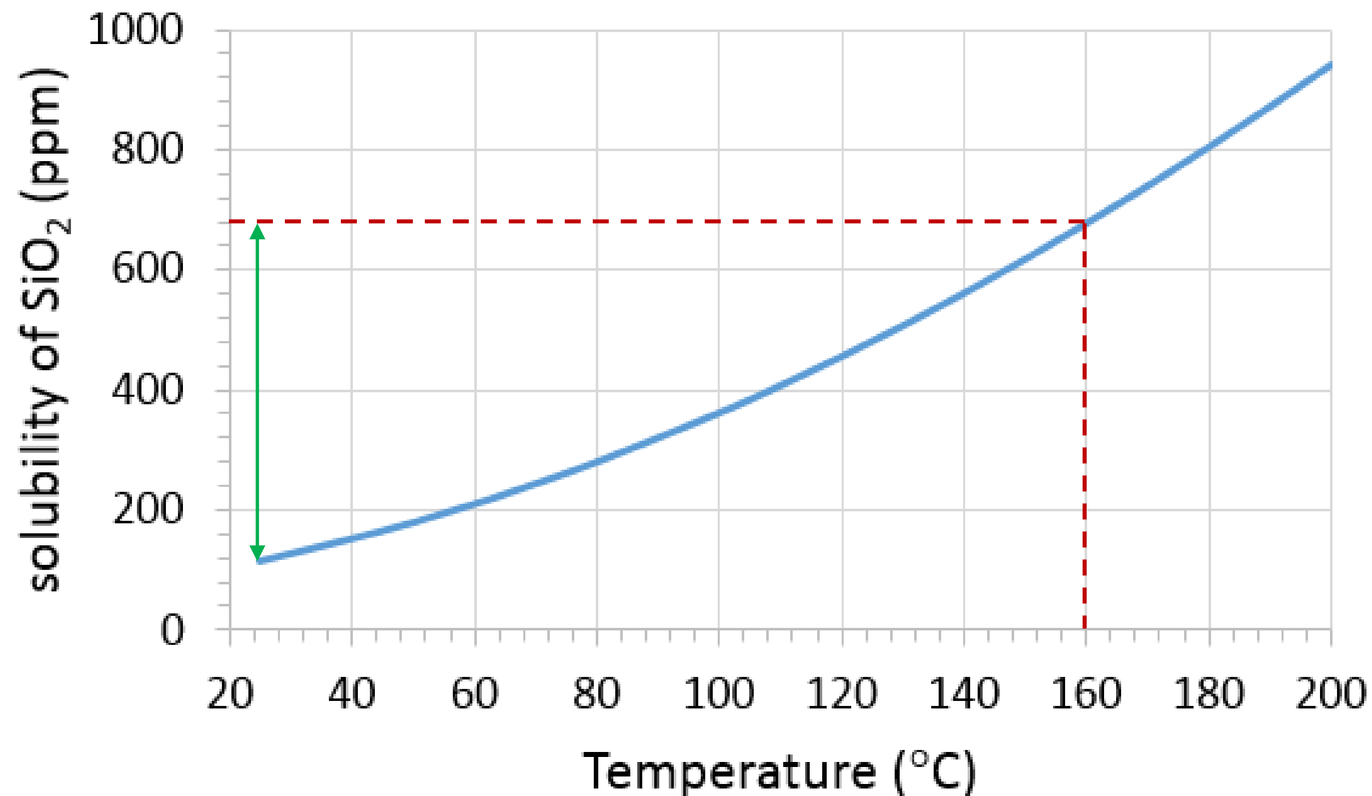
Profilometry Profile: from 160°C Steam to 160°C Water region

- More gradual profile change compared to film regions exposed to HTW



Previous work on SiO_2 in HTW

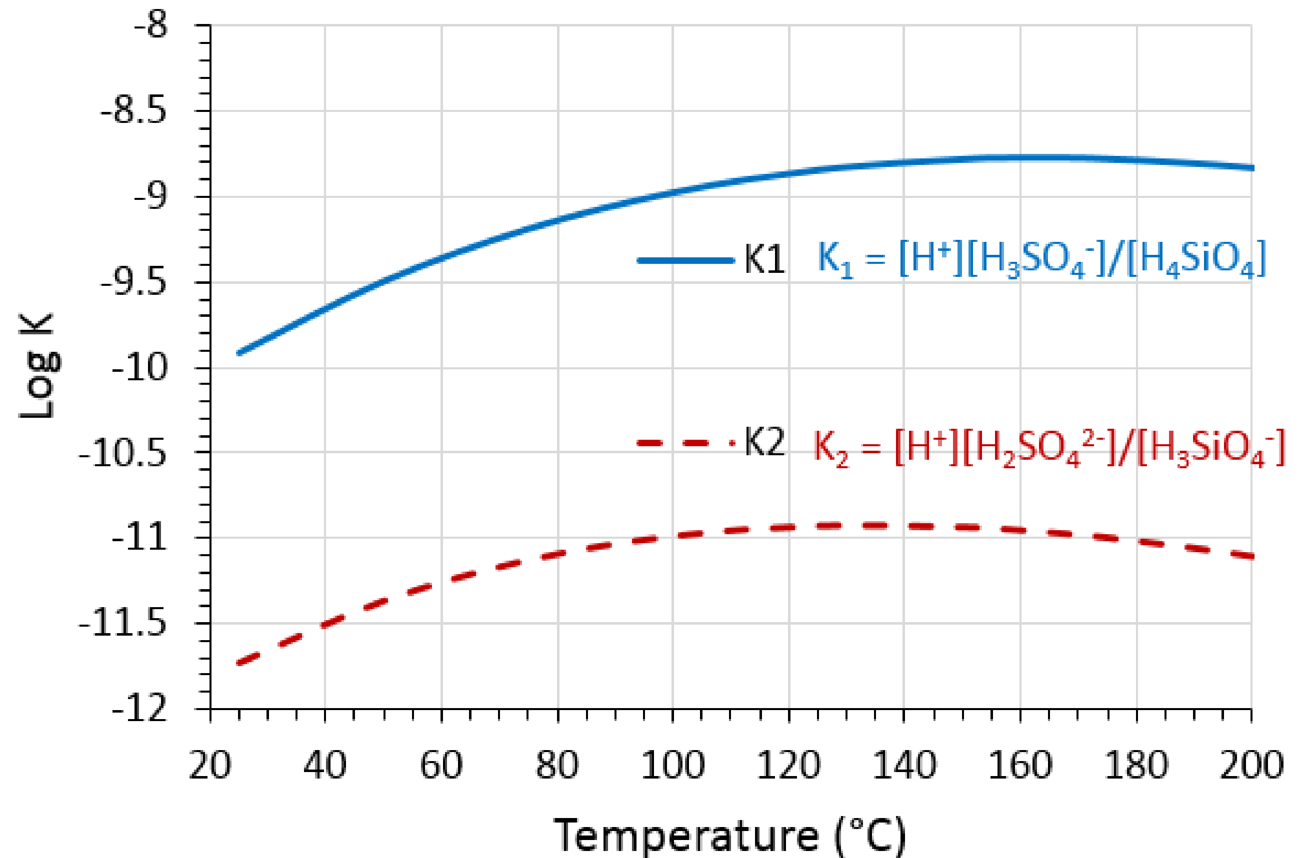
- Solubility increases ~6x at 160°C
- This solubility is sufficient to account for the loss of >1000 nm of oxide



RO Fournier and JJ Rowe, Amer. Miner, 62 1052-1056 1977

Ionization of Silica in HTW

- Ionization of silica at 160°C: ~10x greater than room temperature



Arnorsson, Sigurdsson, Svavarsson, 1982

What is Happening?

- Oxide Growth on top of sample—steam oxidation of silicon
- Regions exposed to HTW—water is more reactive (higher: pKw, solubility, ionization; lower: viscosity and surface tension)→ etching
- Because the heating time is relatively short, most of the reaction is occurring at 160°C

Summary

- The oxide sample appears to be oxidized and etched in the same reactor at the same time.
  - Steam→ oxidation
  - HTW→ etching
- Steam is like other oxidizing steams
- HTW is different from T < 100°C water
- Interesting but reaction may not be easy to control like SiN
- Future work:
  - Confirm etching mechanism
  - Determine etching rate as a function of temperature
  - Determine oxidation rate as a function of temperature

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