

Accessories for electron microscopy: cryo-holder



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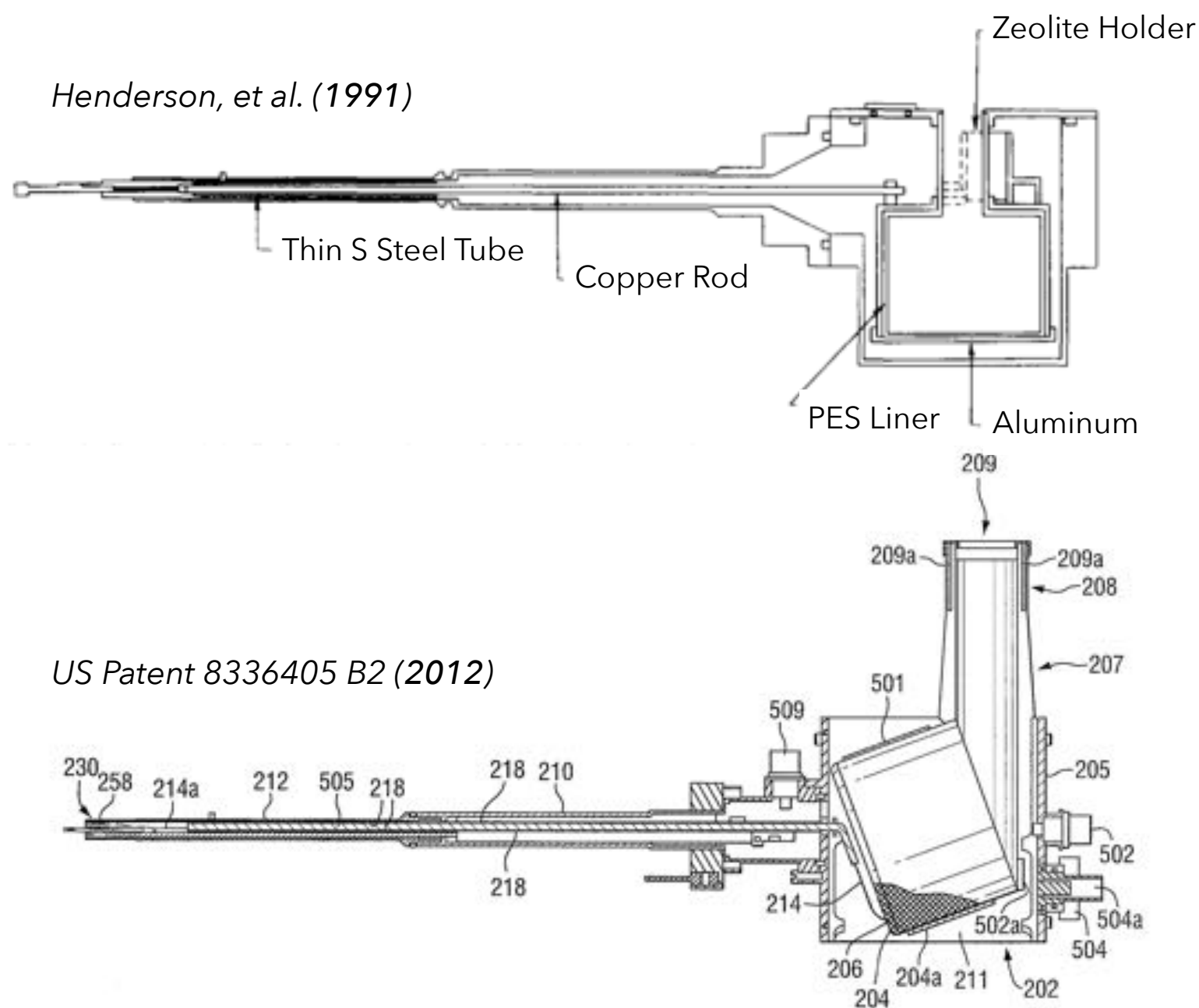
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Motivation

Cryo-holders allow for the imaging of TEM specimens at very low temperatures ($\leq 130\text{K}$). The current cryo-holder market is dominated by Gatan cryo-holders, which are expensive and limited for Hitachi microscopes. The in-house design is expected to provide significant cost savings and is made especially for Hitachi microscopes.

Prior Art & Research

Existing cryoholder designs:



Choice of material to be in contact with the liquid nitrogen is key to high resolution. Minimizing boiling minimizes vibrations at the sample.

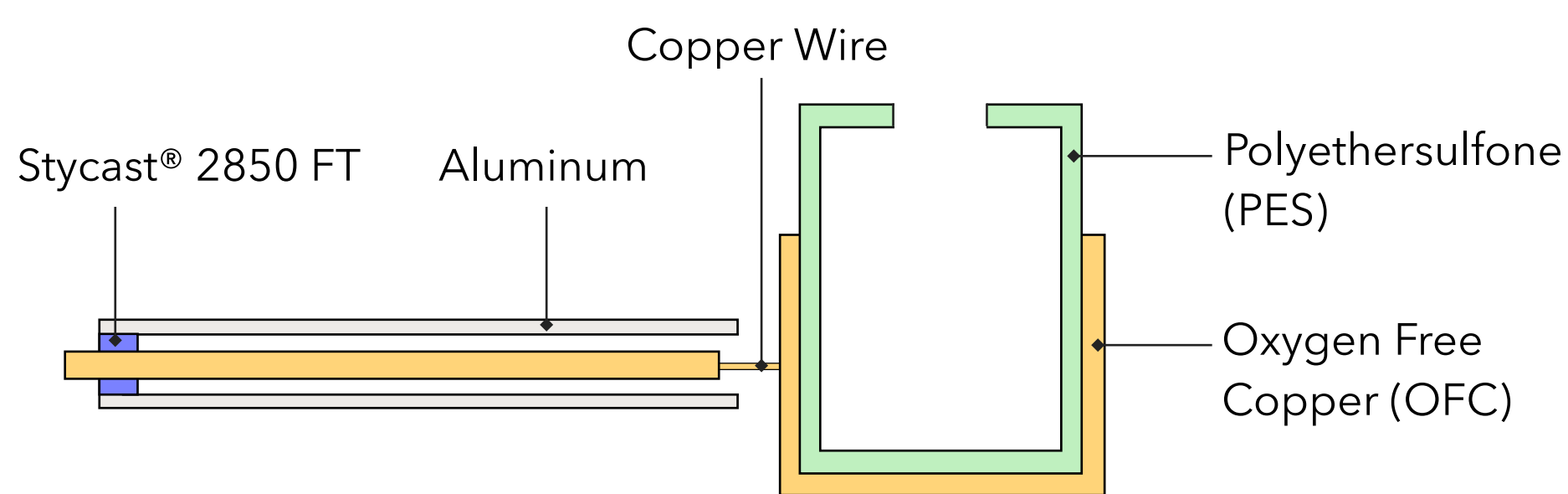
No nucleation	Mild Boiling	Strong Boiling
Copper filings	Stycast 2850FT epoxy	Iron filings
Brass filings	Sugar crystals	Nickel filings
Titanium filings	Ice crystals	Aluminum filings
Dow Corning 340 heat sink compound		White plastic thermo-couple wire (Cu/Ni)
Low-temperature varnish		Araldite epoxy
Blue plastic thermo-couple wire (Cu)		PPS (fibre-glass-reinforced plastic)
Brass screws		"Crosmares" epoxy
Teflon		NaCl crystals
PES		Coffee, granulated
Tissue Paper		Perspex powdered
Ethanol		Zeolite (molecular sieve 4a)

Henderson, et al. (1991)

The prior art shows the principle design features of a cryoholder. Liquid nitrogen in a dewar is used to cool a sample through a rod with very high thermal conductivity. A ribbon connects the liquid nitrogen to the copper rod to minimize vibrations caused by the bubbles in the liquid nitrogen. The material in contact with the liquid nitrogen must not cause nucleation of gas bubbles. Increased bubbling, would lead to more vibrations at the sample tip, therefore reducing the resolution.

Material Selection

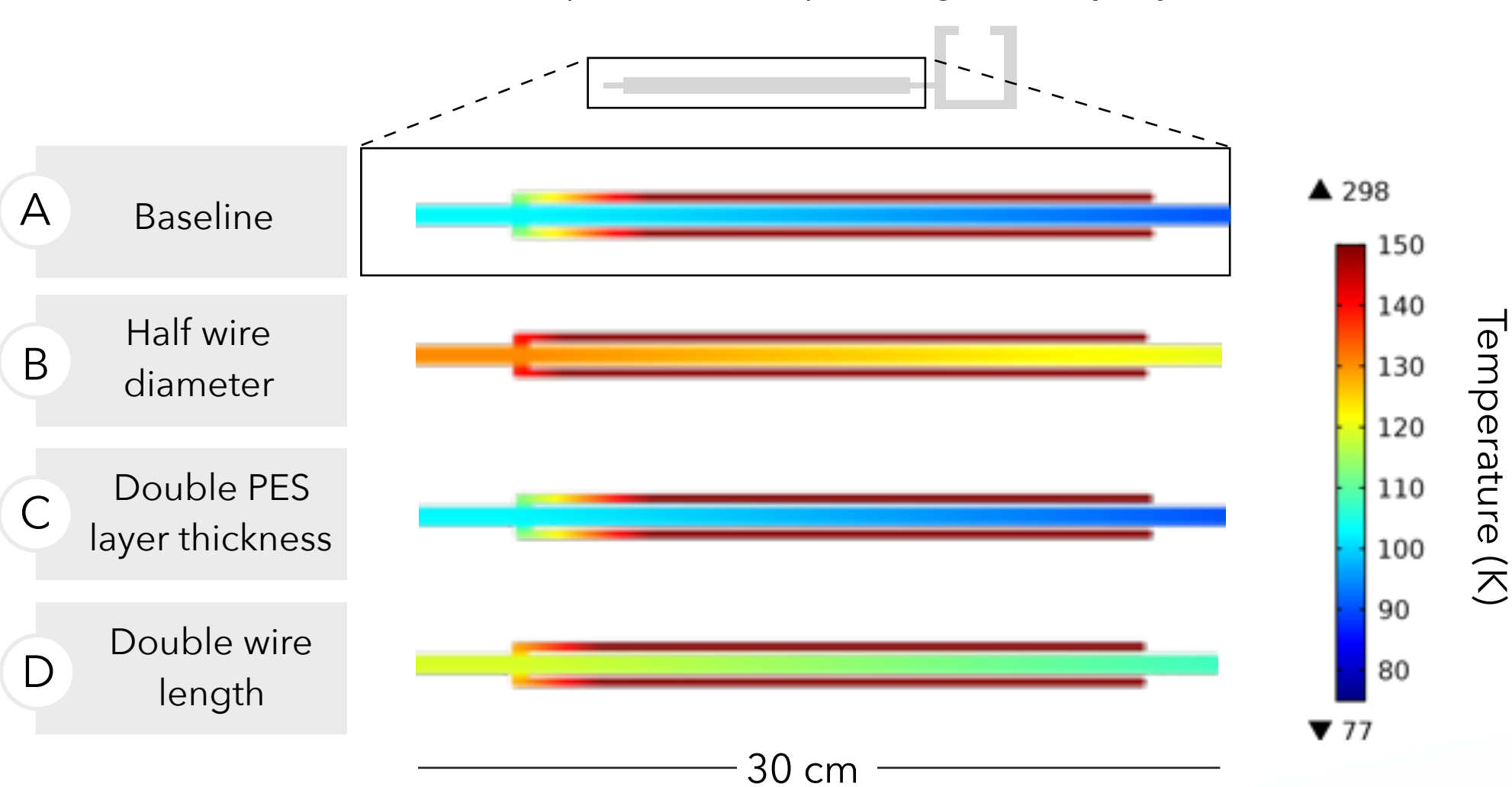
Simplified cryo-holder geometry showing key material selections:



The material selection considers heat transfer, nucleation of gas bubbles, cycling reliability and availability. PES was chosen because it does not nucleate gas bubbles in liquid nitrogen and can withstand repeated cooling cycles. OFC was chosen for its high thermal conductivity at low temperatures, as well as availability. The Stycast® 2850 FT is a commercial epoxy from Loctite® that features a low thermal conductivity and is known to withstand repeated cooling cycles.

Results

COMSOL simulations of heat profile in simplified geometry cryo-holder:



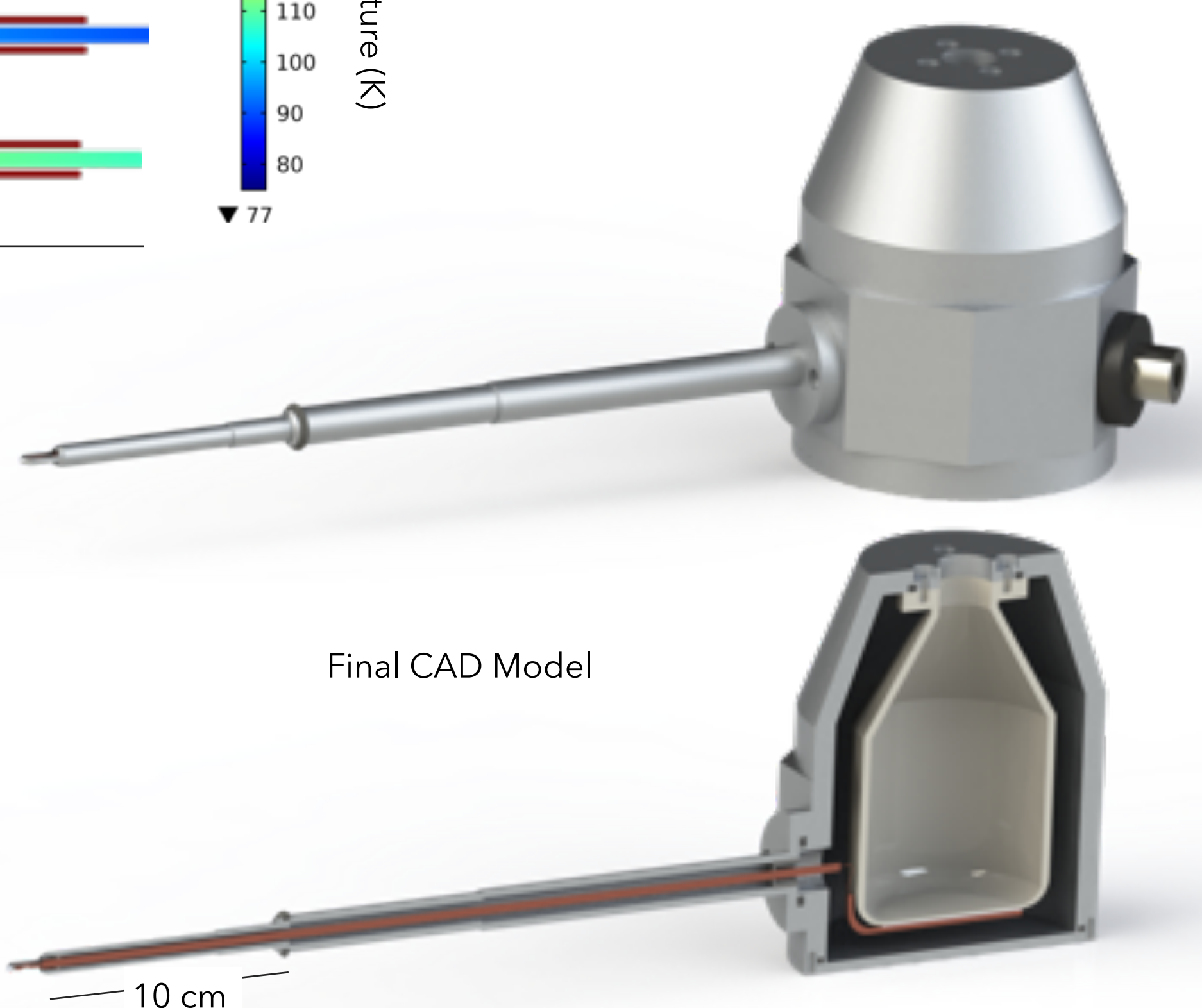
The theoretical ideal temperature at the sample would be that of liquid nitrogen (77K)

Design	Temperature at Sample (K)
A	109
B	128
C	109
D	121

The steady-state COMSOL heat simulations above show the expected temperature at the sample with different design changes. Using the results of the simulations, as well as considering manufacturability constraints, the final proposed cryo-holder design was achieved. The cross-section view shows the PES liquid nitrogen container with a copper plate beneath it, attached using thermally conductive epoxy. The copper plate is attached to the copper rod through a copper wire to minimize vibrations while maintaining heat transfer.

Future Work:

1. Simulation of the final design to verify the expected temperature at the sample.
2. Manufacturing of the final design



References

- Henderson, R., Raeburn, C., & Vigers, G. (1991). A side-entry cold holder for cryo-electron microscopy. *Ultramicroscopy*, 35(1), 45-53.
- Stabacinskiene, H., Gronsky, J., Thomas, C., Deshmukh, P., Robins, A., & Fischione, P. (2012). *U.S. Patent No. US8336405 B2*. Washington, DC: U.S. Patent and Trademark Office.