

TTC 2020 Small Sample Survey

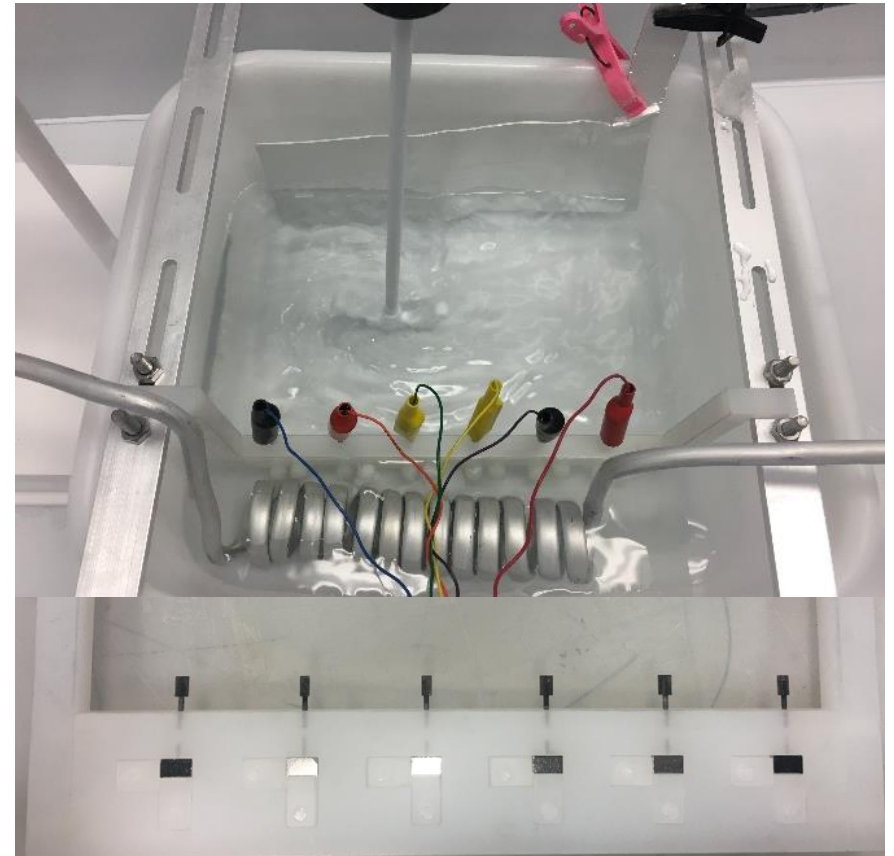
Can we as a group define the best practices for small sample preparation, cleaning and transport?

What experimental techniques are being used around the world?

Who can you contact for future collaborations? – primary contact in embedded file at the end.

Ari Palczewski, Marc Wenskat & Kensi Umemori

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JLab's temperature controlled 6x sample EP setup

Original purpose of the small sample survey

- Similar to the 2019 Cavity furnace survey we wanted to discuss as a group the best practices for small sample preparation and analysis for HighQ0/Grad research within the community.
- The goal is to drive discussion between groups to accelerate the surface science in the field.
- We received input from CEA-SACLAY, IHEP, SHINE-SARI, PKU, TRIUMF, KEK, DESY, and JLab.
- After this presentation we hope you will go into the coffee break and think about question you could ask the experts in the crowd – open discussion will follow in the next session.
- If anyone else still wants to send data this week I will integrate the information into the uploaded talk.

Questions

- Detail your sample size, surface preparation, and stock material?
- Detail the surface techniques are you using and why? – see embed file at the end
- Location of the facilities so other may collaborate? – see embed files at the end
- Do you deform samples before or after cutting? - no
- When have you ever cut-up cavities for samples? Why/why not?
- Do you HPR your samples? – only DESY
- Do you get implant standards made?
- How do you handle and transport samples?
- For doping/infusion do you put samples under a HOM pot or in an Nb box to mimic the cavity interior and then what about this Nb?
- If treated: Are you the same infrastructure as for the cavities or in their own systems?

How are samples made? (1)

	JLab	DESY(FG)	DESY(LG)	DESY(SC)	KEK
Sample size, Nb material	6x10mm (TD LCLS-II stock) 6x10mm (high RRR LG from CBMM and Heraeus) 10x10mm LG	Flat conic, 12mm base, 2.8mm thick, 10mm top (XFEL sheets, FG-TD, NX, Plansee)	Round, d=10mm, h=2.8mm thick (LG disk RRR>300)	Round, d=10mm, h=2.0mm thick (Single crystal high purity from company SPL))	7x7x3mm (TD FG) 7x7x3mm (SC from TD LG) 2mm sphere (TD-FG)
Niobium pre-preparation	usually pre-anneal at 900-1000C depending on stock material, moving forward 100%, but not in the past.				
Cutting/cleaning	cut Wire EDM	Waterjet cutting at DESY + Turning	Waterjet cutting at DESY + Turning		cut out by Milling cutter
Chemical removal 1 /cleaning	100um BCP – 111 fast with agitation Label samples after BCP Nanopolish to 10nm surface roughness and 40/20 scratch dig or better	Coarse BCP or EP Ethanol rinse / US cleaning / HPR	BCP(50-100um) Nanopolish		BCP 100 um or Chemical mechanical polishing Ra ~0.4nm or EP 100um
Heat treatment		800C		2000 C for 6h in UHV	(depend on process)
Chemical removal 2 cleaning	20 - 40 micron EP at 22 C or lower cleaning in ISO 6, ISO 4 UHV	Fine chemistry Rinsing Ethanol / US Cleaning / HPR	Flash BCP Rinsing Ethanol / US cleaning		Ultra-sonic rinsing
Experimental device	SEM, EBSM, XPS, SIMS, TOF-SIMS	SEM/EDX, XRR, XRD, XPS, SIMS, TEM, EBSD, PALS/PAS, UHV AFM + cooling, SQUID, RRR			TOF-SIMS, XRD/XRR, SEM/EDX/EBSD, SEM/EDX, SQUID
Comment	Nanopolish http://www.surfacefinishes.com/ After delivery we perform SEM analysis on 10% of the samples				BCP for SQUID MPMS and EBSD CMP for XPR and (SIMS) EP for SIMS, SEM, XPS

How are samples made? (2)

	CEA-SACLAY	IHEP	SHINE-SARI	PKU	TRIUMF
Sample size, Nb material	4x100, 10x10, 10x20, 20x20mm(TD FG) 10x10, 10x20, 20x20 (TD LG) 100mm diameter (cut out during ESS cavity fabrication)	10x10x2.8mm (Ningxia LG/FG) 5x5x2.8mm (Ningxia LG/FG) 3x3x0.5mm (Ningxia FG)	5x5.2.8mm (Ningxia LG) 2x2x2.8mm (Ningxia FG) 2x2x2.8mm (TD FG)	5x5x2.8mm (Ningxia)	Ellipsoidal samples of size 1-5 cm major axis length. Flat samples of cylindrical and rectangular shape 10x10 to 25x25 mm.
Niobium pre-preparation					
Cutting / cleaning	Cut by supplier or ext. contactor	Cut out samples of different shapes	Cut wire EDM US cleaning Sample labeling after preparation procedure	Cut wire EDM US degrease	Machine / cut Hand polish with sand paper
Chemical removal 1 /cleaning	BCP/EP	~150um BCP USR and stored in vacuum-sealed bags	Over 100um BCP before furnace	BCP 250um Clean by UPW	BCP 120um
Heat treatment	Heat treatment (same procedure with cavities)	(Furnace heat treatment for N-doping study?)	(Furnace heat treatment for N-doping study?)	Apply N-doping step	1400C
Chemical removal 2		Different thickness EP after N-doping	Different thickness EP/BCP	EP or not (depend on purpose)	BCP 5um
Cleaning				Clean by UPW	US cleaning
Experimental device	SIMS, Tunnel Spectroscopy, Magnetometry, XPS, XRD, Confocal microscope, Transport measurement (Tc, RRR)	TOF-SIMS, SEM/XRD, PPMS/MPMS/AFM/EDS, SQUID, the two-coil mutual inductance device	SEM, EPMA, AFM, TOF-SIMS, XRD	TOF-SIMS, XPS, MPMS	SEM, TEM, muSR, betaNMR, SQUID Magnetometry, TOF-SIMS, Low energy-mu SR

When have you ever cut up cavities for samples? Why/why not?

JLab	DESY	KEK	CEA-SACL	IHEP	SHINE-SARI	PKU	TRIUMF
Rarely, usually for weld defect analysis.	1DE16 – study whether carbides are forming inside and how they relate to rf performance .	No.	No.	No. We have two special sample cavities for EP testing and N-doping, respectively. And samples are often heat-treated / N-doped together with cavity.	No. In standard Cavity procedure, our samples are heat-treated / N-doped together.	none. Cavities are too expensive.	Yes, but generally no.

Do you get implant standards made for SIMS or other techniques?

JLab	DESY	KEK	CEA-SACL	IHEP	SHINE-SARI	PKU	TRIUMF
<p>Yes - carbon and nitrogen and oxygen in fine and large grain for SIMS calibration - LEONARD KROKO INC</p> <p>Now getting carbon free N samples.</p>	<p>Yes but they suck – no C, N or O seen in any sample which as been confirmed by two independent SIMS.</p>	<p>No, but we have NbN powder bought from sample vender.</p>	<p>No.</p>	<p>No.</p>	<p>Not yet. Recently we will do Nitrogen calibration for SIMS.</p>	<p>No.</p>	<p>No.</p>

How do you handle and transport samples?

JLab	DESY	KEK	CEA-SACL	IHEP	SHINE-SARI	PKU	TRIUMF
<p>small round holder with backing spider, corner holders - EDS safe tweezers -</p>	<p>MSL Samples: after chemistry in DI Water, otherwise in samples holders under air; NL the same, some experiments sample transport in dedicated chamber, but its experiment specific</p>	<p>The sample surface is wrapped in soft paper with hard sample case. However, recently, there is a lot of garbage. So we will use sticking a rubber seal on the back so that the surface does not touch anything.</p>	<p>Clean room tissues and boxes</p>	<p>Wrap the sample in niobium foil.</p>	<p>Small round holder with backing spider</p>	<p>centrifuge tube</p>	<p>Gloves, sample membrane boxes https://www.2spi.com/item/02002-bc/</p>



For doping/infusion do you put samples under an HOM pot or in a Nb box to mimic the cavity interior and then what about this Nb?

JLab	DESY	KEK	CEA-SACL	IHEP	SHINE-SARI	PKU	TRIMF
Inside a cavity or niobium box, unless trying to evaluate the furnace, then outside. Niobium box in reactor grade.	NL: Nope; MSL: Yes (Nb Box now, HOM Pot earlier). HOM Pot: RRR300, rinsed, BCPed once in the beginning, afterwards not tracked. Stopped using after ~2 runs, switched to Nb-Box. Box is RRR40, BCPed everytime.	Inside a niobium box, unless trying to evaluate the furnace, Niobium box in same grade of cavity (RRR>300).	Nb box or exposed to furnace.	Yes, we put samples in an Nb box which treated with standard processing procedures.	Not yet.	in niobium caps. reactor grade.	No.

If treated: using the same infrastructure as for the cavities or together or some own systems?

JLab	DESY	KEK	CEA-SACL	IHEP	SHINE-SARI	PKU	TRIUMF
We use the same furnace for cavity.	Both – we perform in-situ and ex-situ; NL uses mainly in-situ own infrastructure for treatments/experiments while MSL uses cavity system partially together with cavities.	We install samples with cavity at same time in the cavity furnace. Now we build sample furnace using tubular electric furnace.	We use the same furnace for cavity.	We use the same furnace for cavity.	We use the same furnace for cavity.	We use the same furnace for cavity.	We use the same furnace for cavity.

- Enjoy the coffee and come back ready to discuss.
- Embedded survey data in the file link below. Click to open in Excel



Microsoft Excel
Worksheet

SEM images by Joshua Spradlin – JLab spradlin@jlab.org

