20/09/2021

DEVELOPMENT OF A CONTINUOUS NUCLEAR DEMAGNETISATION REFRIGERATOR (CNDR)

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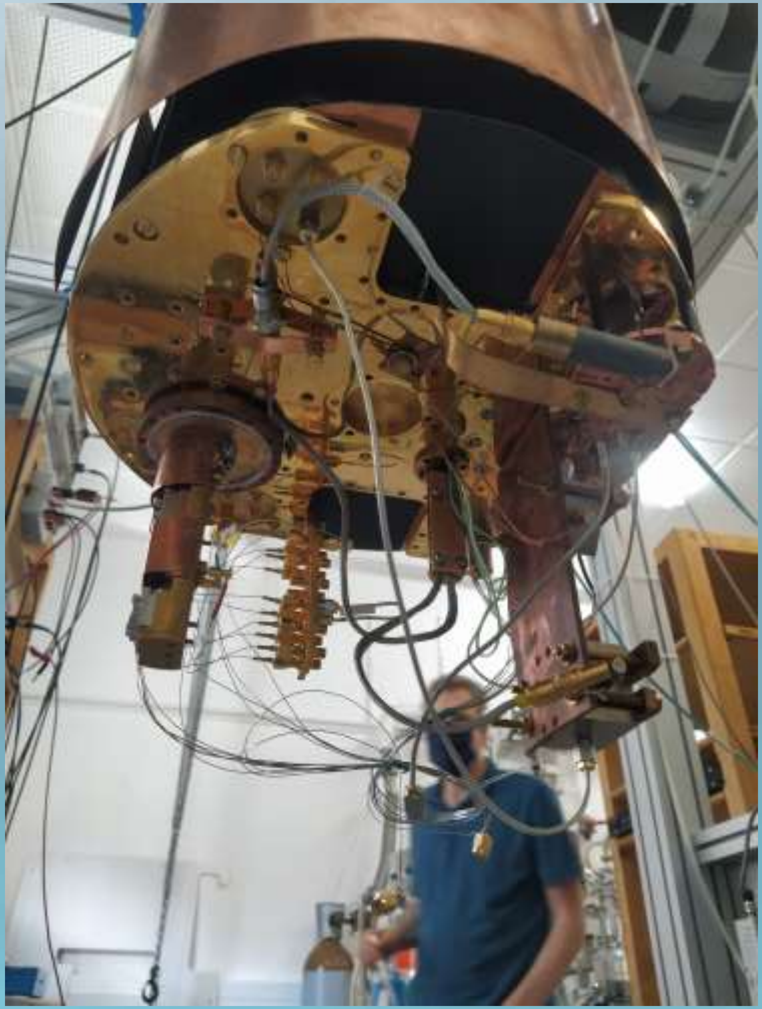
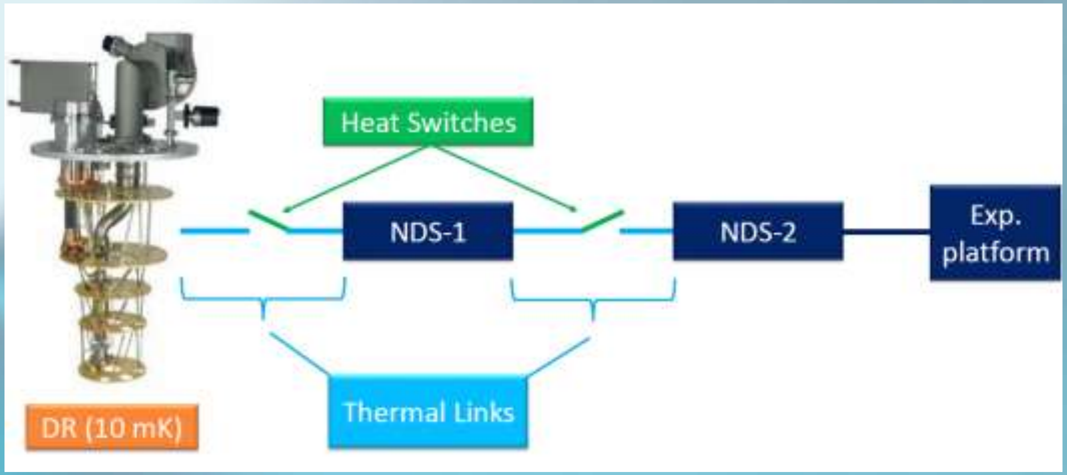
European Research Council

OUTLINE

- Overview
- Thermal resistance issue
- Contact / Welding / Heat Switch issue
- Summary of resistance measurements so far
- Heat Switch thermal characterization: normal and SC states
- Test of the Heat Switch with a single NDS
- Conclusion



- Goal is to achieve 1 mK continuously
- Serial configuration is preferred to parallel (refer to David's talk)
    - Twice fewer heat switches
    - More compact
    - Second stage (Nuclear Demag Stage 2 - NDS2) with lower fields



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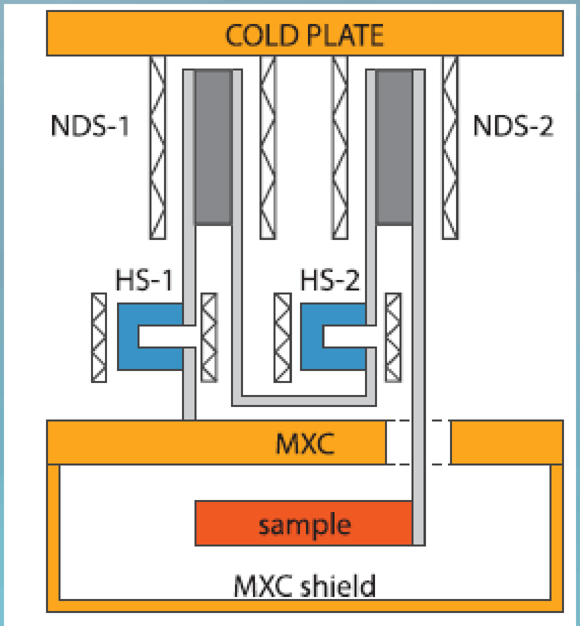
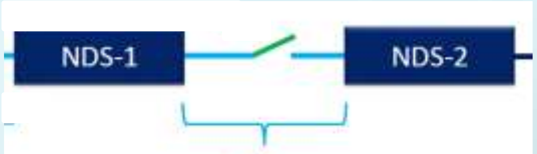
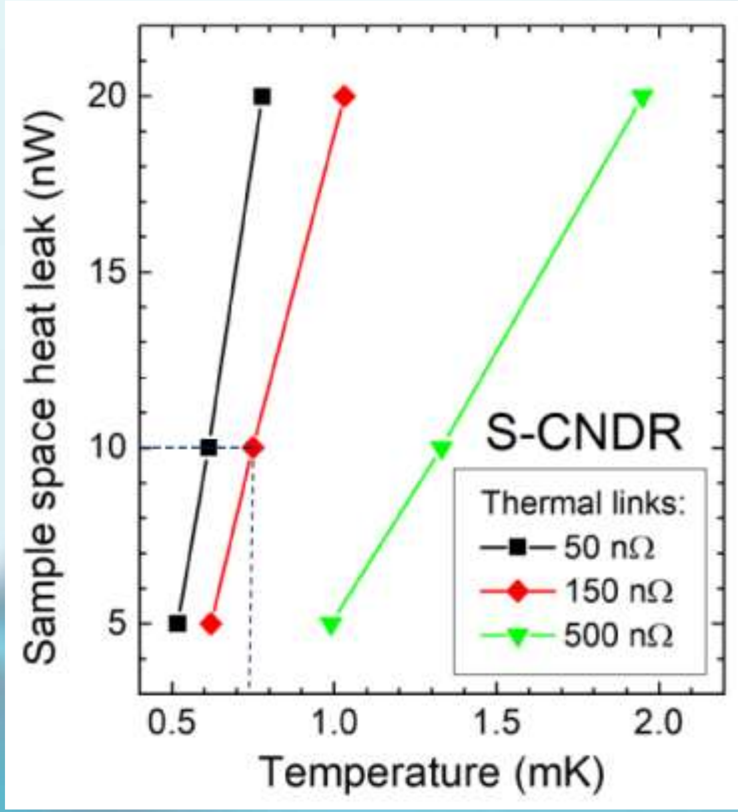




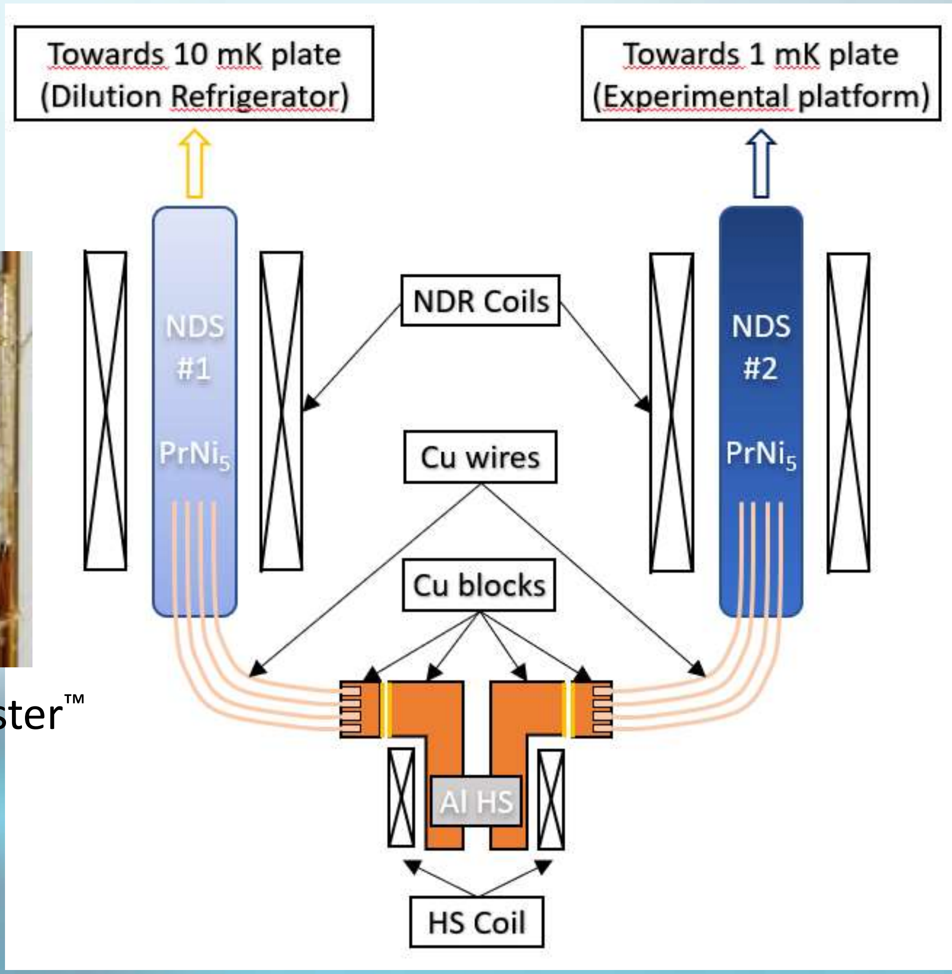


D. Schmoranzer *et al.*, *Cryogenics* **110**, 103119 (2020).

- In S-CNDR, performance is limited by the thermal resistance between the 2 NDSs (⇔ electrical R using the Wiedemann-Franz law)
- 10 nW heat load (typ. 5 nW residual losses) and 150 nΩ => ~750 µK operating T according to simulations
- With some margin, we give ourselves a goal of 150 nΩ
- Minimizing R will be helpful, even in a parallel configuration

PRELIMINARY BUDGET

Lancaster™



15 individual contributors:

- Bulk materials
- Contacts / Assemblies

NDS to Cu wires

Cu wires

Cu wires to Cu block

Cu block (dismountable)

Cu blocks connection

Cu block (HS assembly)

Cu block to Al heat HS

--------- Al HS ----------

PRELIMINARY BUDGET

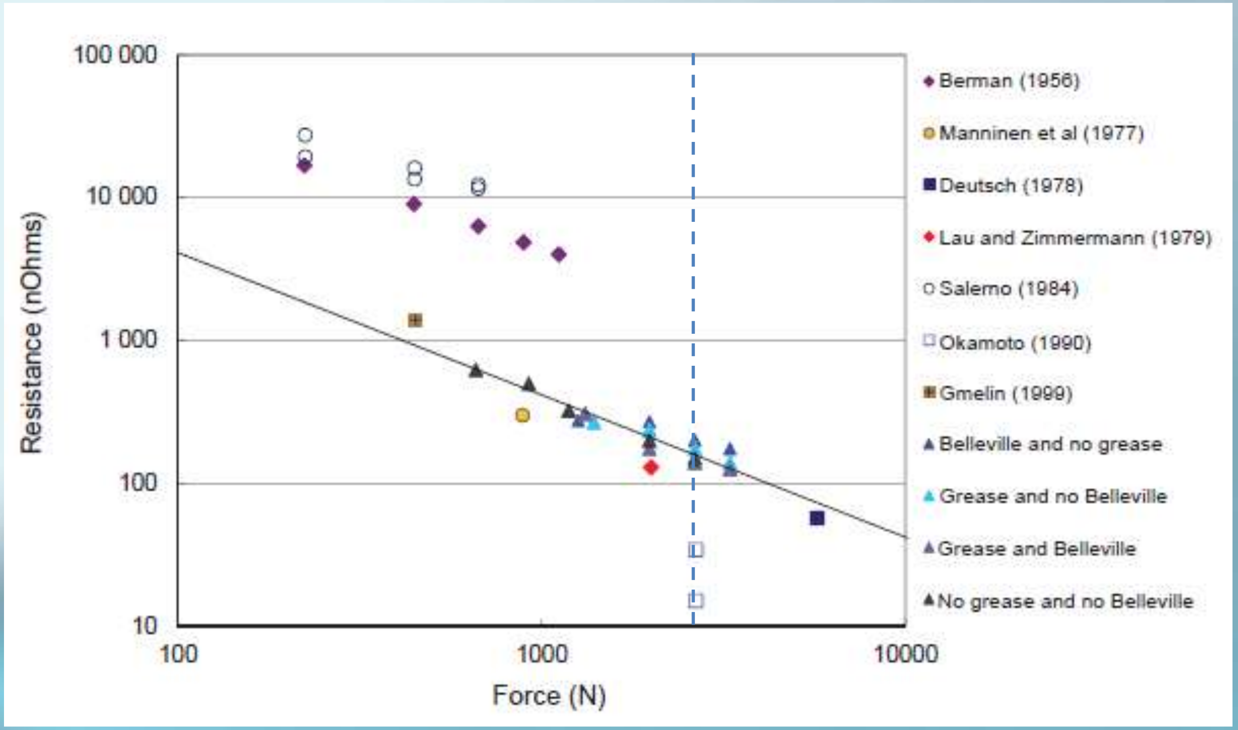
Label	Type	R (nΩ)	#	Notes
NDS to Cu wires	Brazed	5	2	10 wires => 50 nΩ per wire allowed
Cu wires	Bulk	30	2	For 10 Wires ϕ1.5 mm - L=8 cm - RRR 2500
Cu wires to Cu block	Welded	5	2	10 wires => 50 nΩ per wire allowed
Cu block (dismountable)	Bulk	5	2	Cu with RRR > 1 000
Cu blocks connection	Pressed	10	2	Goal
Cu block (HS assembly)	Bulk	5	2	Cu with RRR > 1 000
Cu block to Al heat HS	Pressed	10	2	Goal
Al HS	Bulk	5	1	Calculated 2 nΩ with RRR = 5000
TOTAL		**145**		

Major uncertainties = contact resistances

Most critical one = Al to Cu

CONTACT ISSUE

- Is it possible to make a Cu-Cu or Cu-Al joint with a contact res. (R_c) in the nΩ range ?

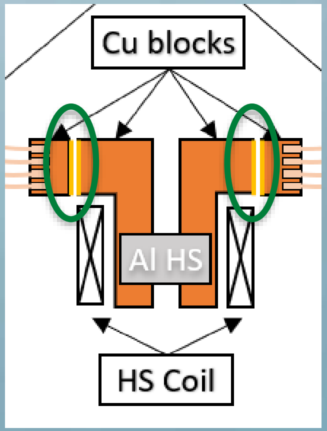


Review of Cu-Cu contacts

F. Blondelle *et al.*, *Journ. Low Temp. Phys.* **175**, 877 (2014).

- For Cu-Cu contacts at 3 kN (M4 compatible), R_c usually > 100 nΩ. Some authors report R_c < 10 nΩ including for Cu-Al contacts.

CONTACT ISSUE

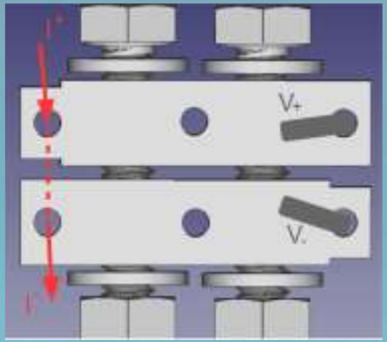


Goal: dismountable Cu-Cu joint

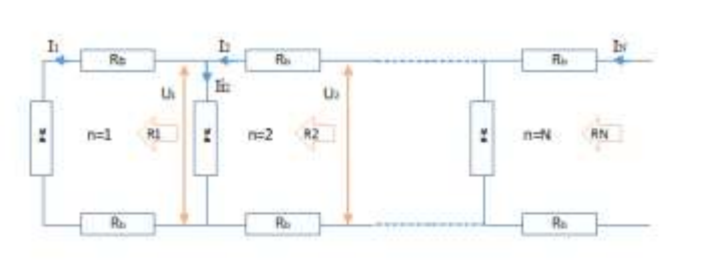
Simple design for preliminary tests

Parameters:

- Rough Polish (RP ⇔ P800) or fine polish (P ⇔P2000)
- Coating (Ag or Au) or not
- Electro (Ag + Au) or electroless (Ag) plating or evaporation (Au)
- Application of extra compression force (7.5 kN) or not

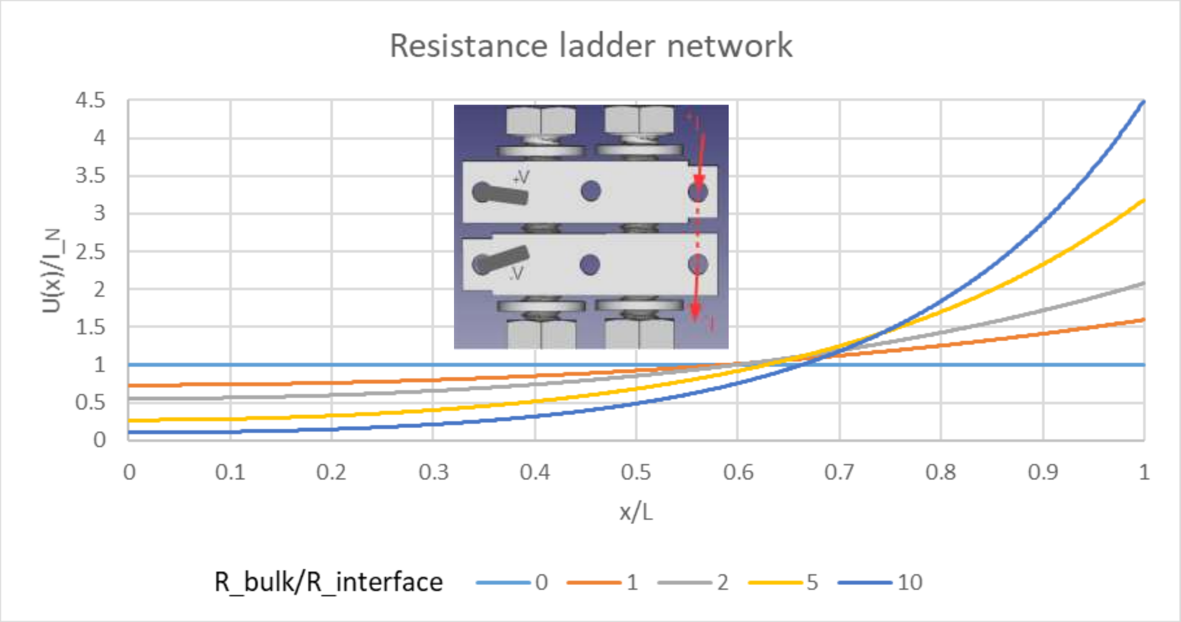


CONTACT ISSUE



Notes:

- Use of high RRR bulk Cu is mandatory
- 4 wire measurements can be tricky



Apparent R as function of V pickups position for various R_{bulk} / $R_{contact}$ ratios

Current injected at x/L = 1

- Our measurements (x=0 and x=0.5) showed no discrepancy at 4.2 K

CONTACT ISSUE

Results:

Sample	R at 4K	Sample	R at 4K
Cu-Cu RP 7.5 kN	$435 \pm 7\ n\Omega$	Au Dalic P	$5.5 \pm 8.4\ n\Omega$
✓ Au Dalic RP	$3.9 \pm 6.1\ n\Omega$	Au Evap P	$13.7 \pm 8.7\ n\Omega$
Ag Dalic RP	$5.2 \pm 5.6\ n\Omega$	Cu-Cu RP	$44 \pm 9\ n\Omega$
Electroless Ag RP	$47 \pm 9\ n\Omega$		

- Extra compression not recomended
- Rough polish is sufficient
- Dalic electro plating provides the best results. Au preferred to Ag
- Below 50 nΩ feasible for Cu-Cu contacts with no coating or a simple electroless coating

CONTACT ISSUE

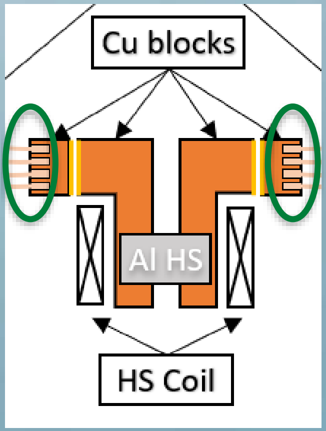
Impact of multiple connections / disconnections:

	RP AuDalic	RP AgDalic	P AuDalic
Dismounted+0 09/12/20	$3.9 \pm 6n\Omega$	$5.2 \pm 6n\Omega$	$5.5 \pm 8n\Omega$
Dismounted+2 10/06/21	$6.7 \pm 7.2n\Omega$	$5.7 \pm 6n\Omega$	$8 \pm 7n\Omega$
Dismounted+3 18/06/21	$5.5 \pm 14n\Omega$	$5 \pm 10n\Omega$	$3.9 \pm 14n\Omega$
Dismounted+0 23/06/21	$4.7 \pm 7n\Omega$	$4.7 \pm 5.3n\Omega$	$7.1 \pm 5.3n\Omega$
Dismounted+1 *air 30/07/21	$2.8 \pm 3.3n\Omega$	$8.9 \pm 3.9n\Omega$	$6.9 \pm 3.4n\Omega$

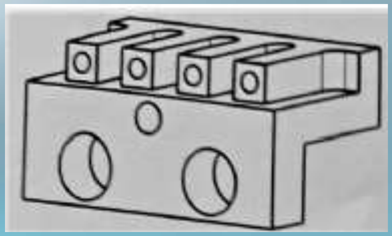
No measurable change in 6 months including after 1 month with the 2 parts in contact with air

WELDING ISSUE

Goal: low resistivity Cu wire to Cu block assembly



Selected design for a 4 wire assembly after a series of basic tests:



WELDING ISSUE

Results:

Sample	After Annealing & Heat treatment				
	Resistance at 4K for 1 weld [$n\Omega$]				4-wire resistance [$n\Omega$]
	weld n°1	weld n°2	weld n°3	weld n°4	
CuCuWeld7	59 ± 8	69 ± 7	39 ± 17	52 ± 17	13.1 ± 3.6
CuCuWeld8	21 ± 18	32 ± 7	22 ± 14	29 ± 9	6.3 ± 3.5

- Up to 50% discrepancy between the different welds
- Resistances are comparable to that of the bulk material => no major contribution of the welding process

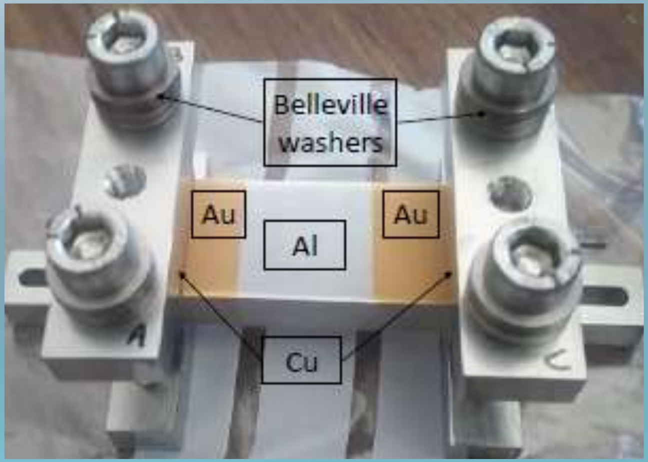
- Global R in agreement with our needs

HEAT SWITCH ISSUE

Idea (inspired from Shigematsu): chemical etching followed by plasma etching + e-beam Au deposition (evaporation)

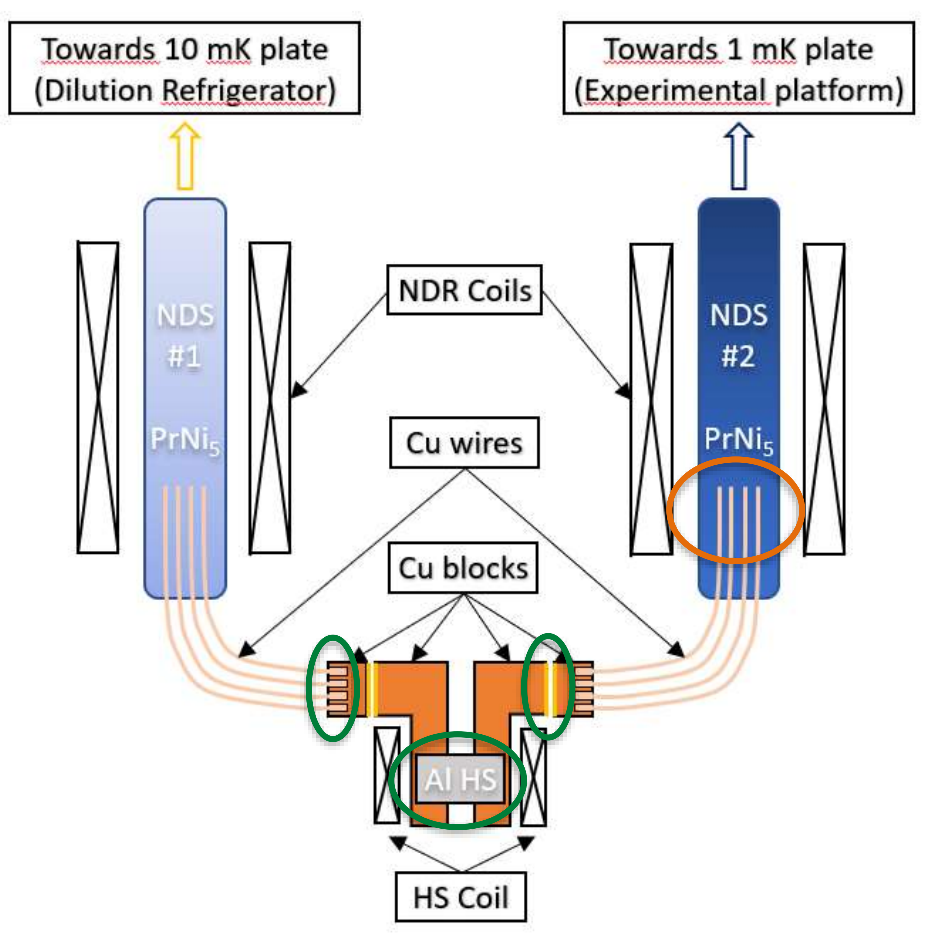
- Preliminary tests on a basic Al to Cu assembly:
- Different configurations tested (cf. article)

S. Triqueneaux *et al.*, *Journ. Low Temp. Phys.* **203**, 345 (2021)



- Contact resistance as low as **3 nΩ** at 4.2 K could be achieved ✓

SUMMARY



Most of the contact resistances are validated

SUMMARY

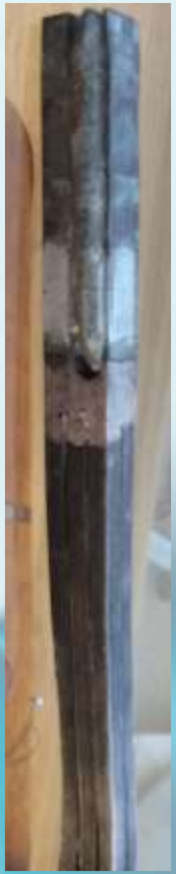
Label	Type	R (nΩ)	#	Notes
NDS to Cu wires	Brazed	5	2	Same as ini
Cu wires	Bulk	30	2	Same as ini
Cu wires to Cu block	Welded	5	2	Measured (~ 50 nΩ per weld)
Cu block (dismountable)	Bulk	2	2	Measured
Cu blocks connection	Pressed	5	2	Measured
Cu block (HS assembly)	Bulk	3	2	Measured
Cu block to Al heat HS	Pressed	1	2	Measured
Al HS	Bulk	1	2	Measured (3 nΩ equivalent R)
TOTAL		**103** ✓		

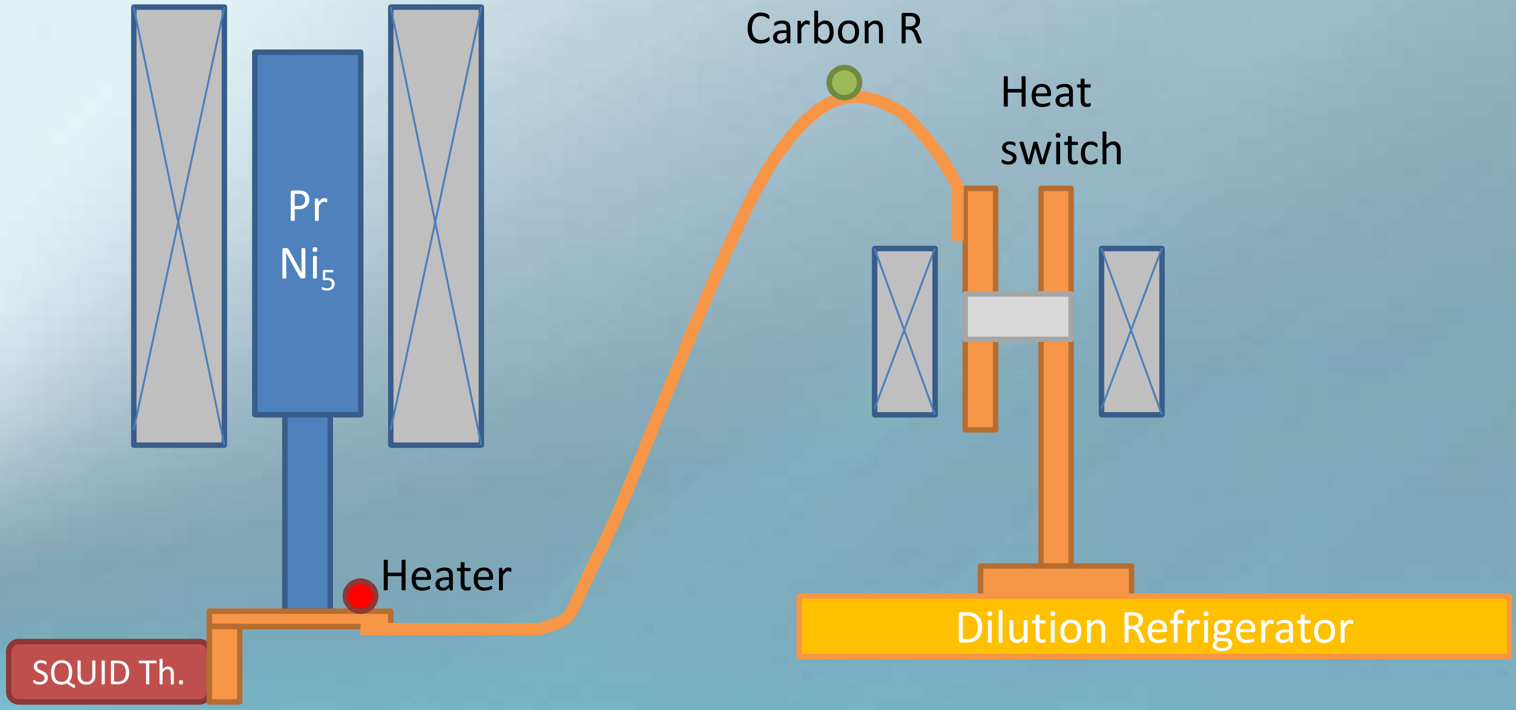
Relaxes some constraint on the wires

NDS to wires contact R to be validated

TEST OF HS + NDS

Further step = HS combined to a single NDS:



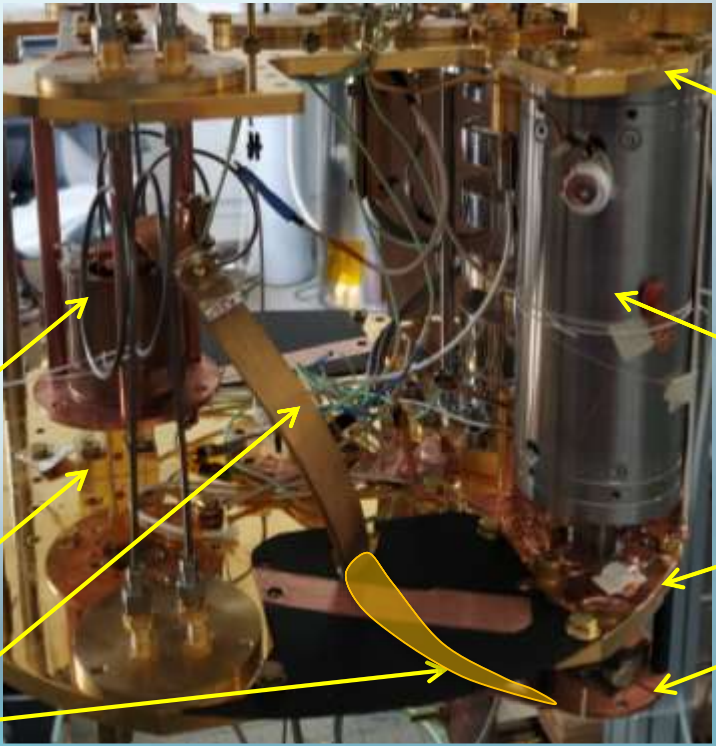


Existing $PrNi_5$ stage (Parpia)

$PrNi_5$ thermalized onto an Ag rod via cadmium soldering

TEST OF HS + NDS

Once mounted onto Andrew's (Bluefors) DR:



100 mK plate

NDS Coil

HS Coil

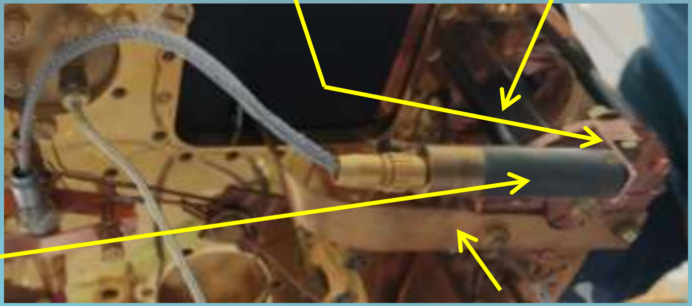
HS

10 mK plate

1 mK plate

NDS

Thermal link
(not critical)

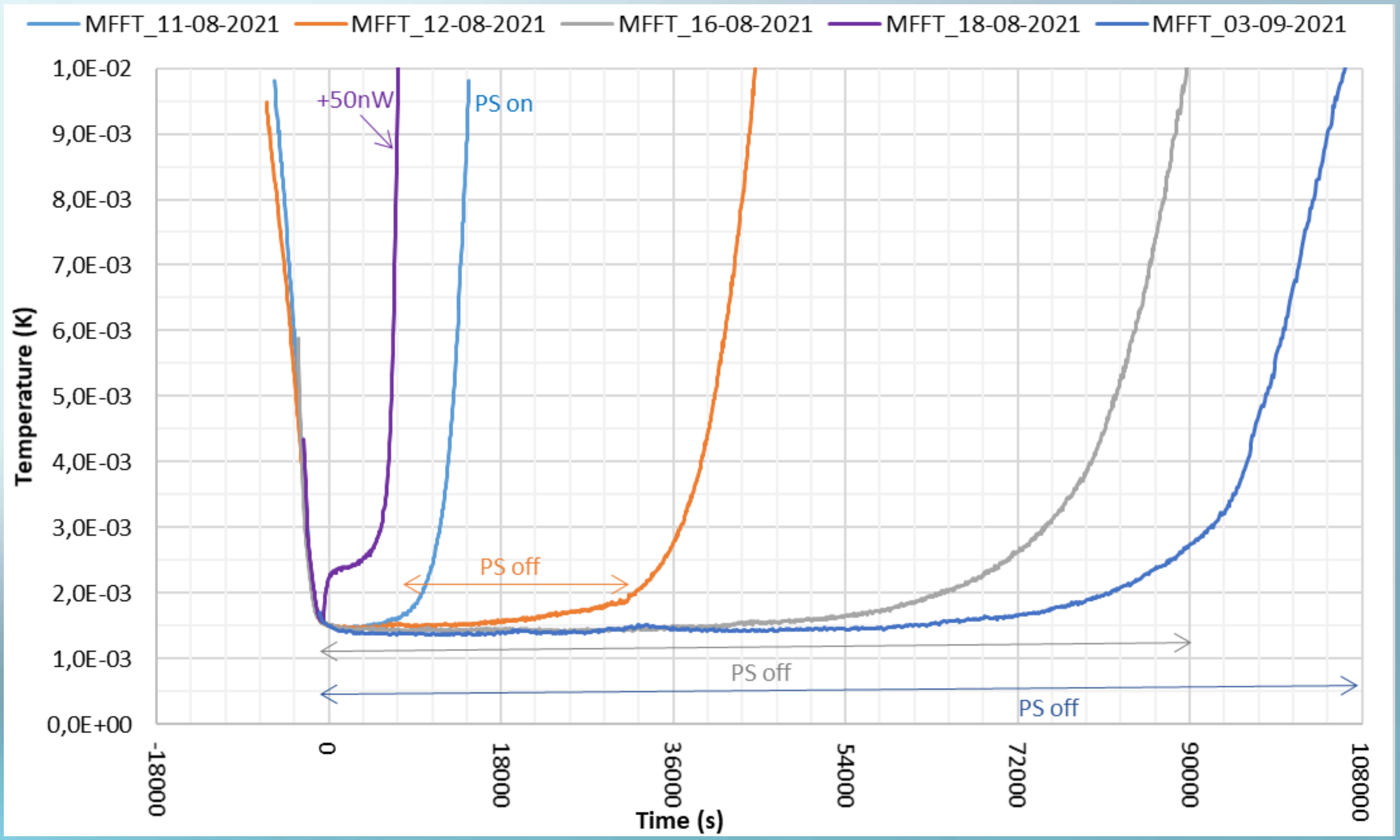


SQUID
Thermometer

Thermal link

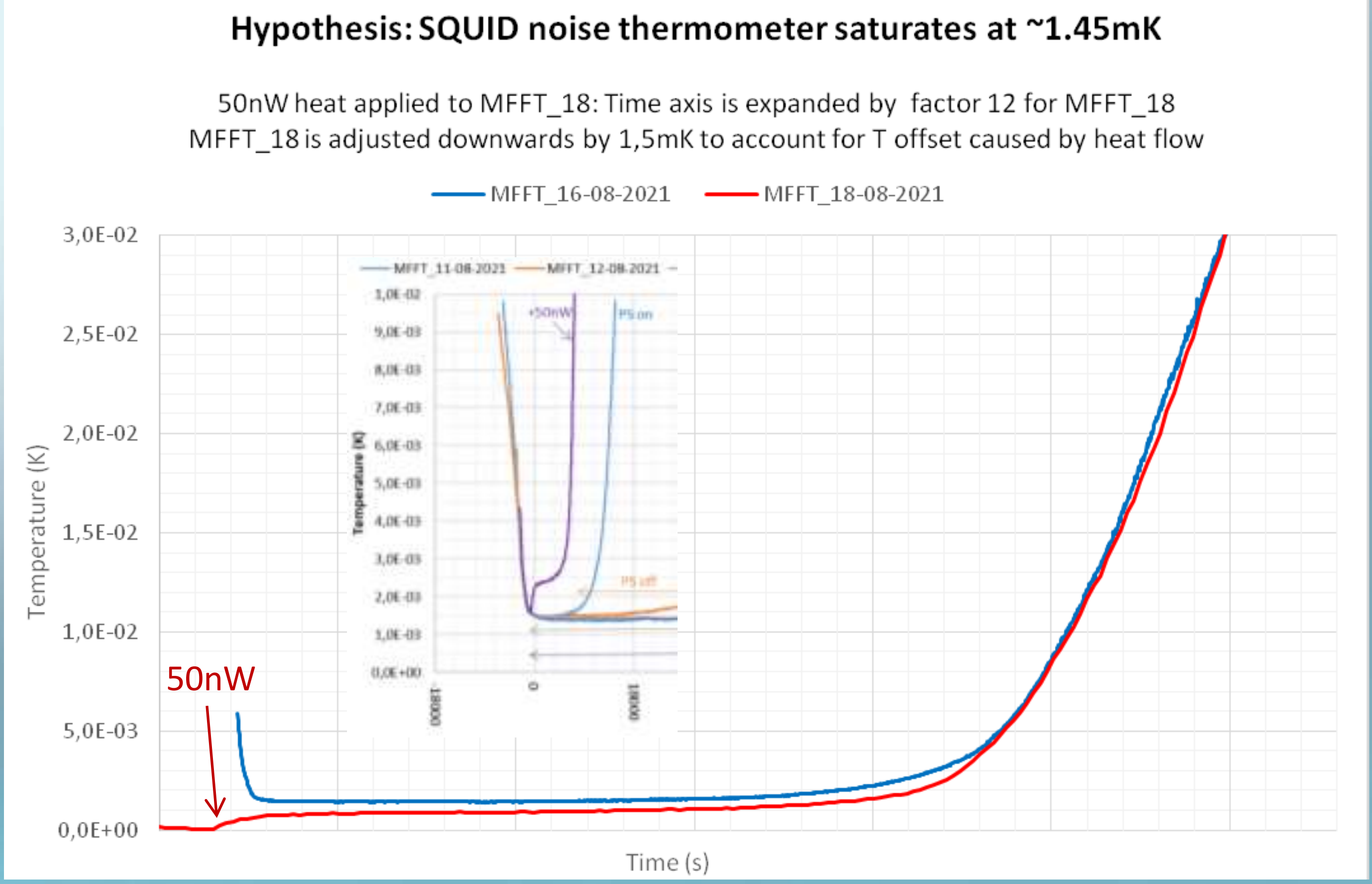
TEST OF HS + NDS

Results:



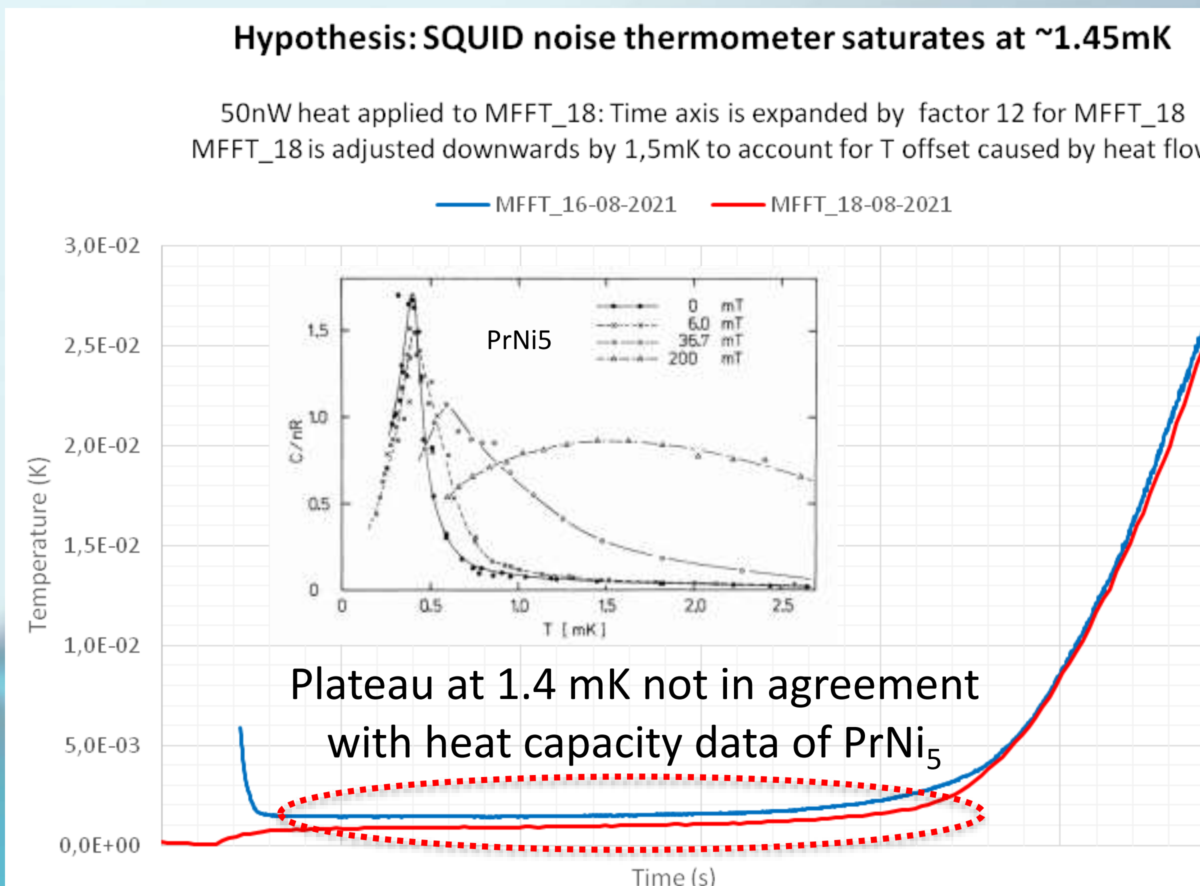
- Power Supply (PS) has hudge parasitics at 0 current!
- Best case : ~23 hours with the SQUID therm. displaying T < 2 mK

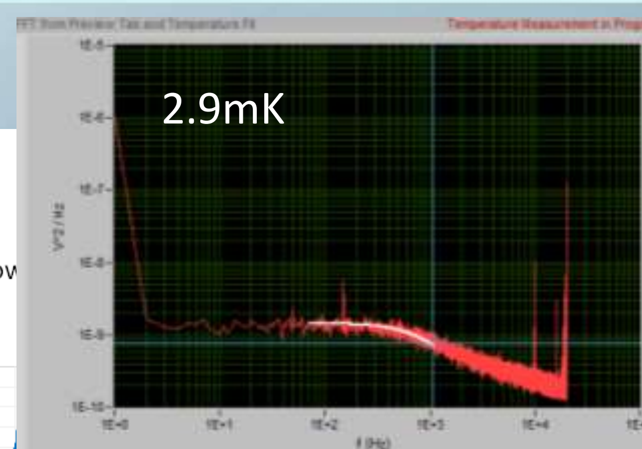
Results:



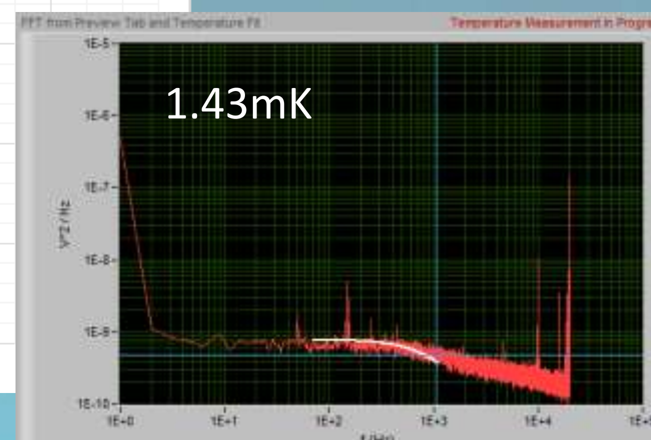
- Increase by a factor ~12 of total heat load when 50 nW heat is applied. This would imply parasitics of ~4.5 nw (OK with previous publications)
- Best case (23 hours) may suggest variable parasitics & as low as ~2 nW

Results:





SQUID Therm. behavior is under Analysis



CONCLUSION

- Most of the thermal resistance issues are solved
- The heat switch has demonstrated very good performance, including with an NDS
- We believe that we have been able to reach below 800 µK on a $PrNi_5$ stage with 2-4 nW parasitics
- Still some optimizations needed: power supply, thermometry

CONCLUSION

We plan to focus our efforts on the design and manufacture of our own NDS

- Manufacturing issue for $PrNi_5$
- Thermal contact issue (Cd not very convenient)

=> Overall $PrNi_5$ to Cu thermal contact might become the bottleneck

- Al less efficient
- Al – Cu contact should not be an issue
- Al not adapted to serial configuration