Development of a Broadband LN2 cooling Cryogenic Preamplifier for FTICR MS

Myoung Choul Choi[1], Stefan Stahl[2], Seung Yong Kim[1], Hyun Sik Kim[1]

[1]Korea Basic Science Institute, Mass Spectrometer Development Team

[2] Stahl-Electronics, Germany

Contact KBSI : cmc@kbsi.re.kr, contact Stahl-Electronics: s.stahl@stahl-electronics.com

News

1. A novel broadband cryogenic-detection system has been developed to improve the signal-to-noise ratio (S/N) of FT-ICR mass spectrometry at liquid nitrogen (LN2) temperature.

2. Recent results show significant improvement of the FT-ICR signal originating from liquid nitrogen cooling.

Goal

Operating a preamplifier of a FT-ICR MS setup at cryogenic temperatures has potentially two main advantages: First, better vacuum conditions are achieved. Second, a larger signal may be obtained since the preamplifier is installed very close to the ICR trap and at the same time is cooled, thus reducing noise. The net resulting higher electrical signal to noise (S/N) ratio promises greatly improved sensitivity. The development of cryogenically operating amplifiers in conjunction with a cooling system allows to check the advantages of this novel approach and to explore its limits.

1. Implementation

The novel aspects of the developed FT-ICR MS detection setup are a partially cryogenic vacuum setup in conjunction with cryogenic preamplifiers. The system was developed by the KBSI Korea Basic Science Institute in close collaboration to Stahl-Electronics, Germany, providing cryogenic amplifier technology. The broadband preamplifier can be operated over a very wide temperature range from room temperature to low temperature environments (T = 4.2 K, 77 K).

A cooling system inside the FT-ICR MS setup has been designed, which uses circulation of LN2 (liquid nitrogen, 77K) or LHe (liquid helium, 4.2K) to cool down a copper cold finger including cryo preamplifier and FT-ICR cell to 80 K or to 16 K. In contrast to other ways to improve the sensitivity of FT-ICR MS, such as higher transmission efficiency, linear excitation trap and various ion optic devices, the cooling of crucial parts (trap and electronics) represents a more fundamental approach. A conventional trap (Open Cylinder trap) has been modified and its signals are processed by a low noise cryogenic amplifier, being located in close proximity to the trap inside vacuum. The cryogenic amplifier is followed by a new 2nd amplifier (Stahl-Electronics) which amplifies the signal subsequently after the cryo-preamplifier.

2. Diagram of cryogenic preamplifier setup

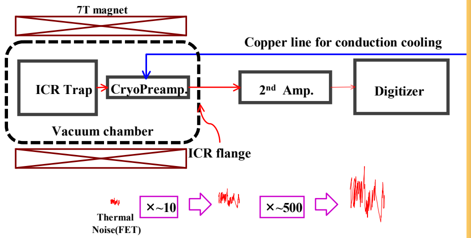

Fig. 1:
Schematic diagram of the cryo-preamplifier system:
A preamplifier is located inside the vacuum in close proximity to the ICR trap, thus improving signal strength. Liquid Nitrogen or Helium cooling additionally lowers amplifier noise.

3. COOLING SYSTEM FOR PREAMPLIFIER AND TRAP

▪ Liquid (LN2, LHe) Circulation System

- Liquid circulates in the tube-in-tube setup in order to reduce heat leakage from the flange
- Portable and compact cooling unit

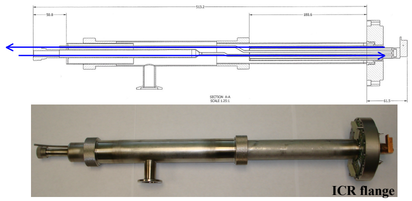

Fig. 2 Liquid Circulation Unit

▪ Cryo-Preamplifier & Cooling Insert (Liquid Circulation)

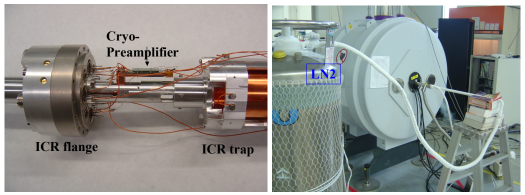

Fig. 3: Cryopreamplifier and LN2 circulation cooling

▪ Temperature at Tip of Cryo-preamplifier (Liquid Circulation)

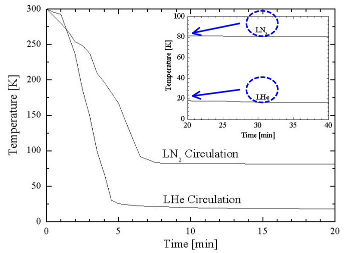

Fig. 4: Time vs. Cooling temperature by LN2 or LHe circulation cooling

- Temperature history during initial cooling down
- LN2 circulation: T = 82 K @ 10 min, maintaining T ≈ 80 K during 30 min
- LHe circulation: T = 20 K @ 10 min, maintaining T ≈ 16 K during 30 min
- Thermal resistance: Thermal radiation, conduction, and contact resistance

Challenges for Cryogenic FT-ICR amplifiers

- Preferably operate at room temperature as well as at 77K, 4.2K
- Operate in ultra-high vacuum
- Operate in high magnetic fields
- Withstand mechanical stress during repeated thermal cycling
- Small geometrical required size in order to fit into cryogenic setups

4. Cryo-PREAMPLIFIER for FTICR MS

Semiconductors at low temperatures

Problem:
Standard silicon-based components fail to work at low temperatures (<60 K) and have difficulties to work at high magnetic fields (Hall effect).
Solution: GaAs-based components can operate at low-T and high-B but show increased noise at low frequencies (1/f-noise)

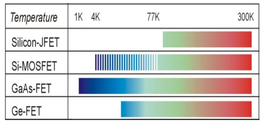

Fig. 5 Usable temperature range of different semiconductors. The readily available and well-developed Silicon based components fail to work at low temperatures.

Different semiconductors feature different noise properties. The 1/f-noise, also called „flicker noise" represents a problem, limiting the noise performance at low frequencies. Thorough selection of the transistor types is required and good thermal anchoring to achieve a low device temperature and low noise behaviour at low frequencies below 0.5MHz.

Amplifier Realisation

Implemented as printboard-copper stack (sandwich-type) the cryo-preamplifier incorporates GaAs and latest thin film technology, suited to be operated inside a high magnetic field and cryogenic vacuum. A massive copper substrate provides high heat conduction.

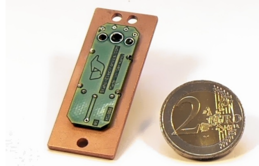

Fig.6: Amplifier and coin for size reference

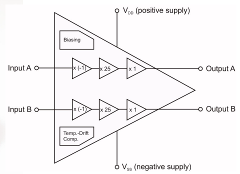

Fig.7: Internal structure

Noise Characterisation

Both current noise (charges streaming towards the trap) as well as voltage noise (virtually appearing at the amplifiers output) are of relevance and can be optimized. Separate measurements help to understand possibilities for parameter optimisations.

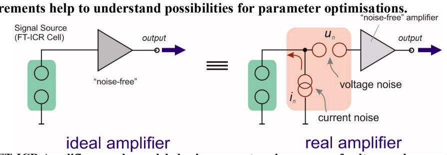

Fig.8: FT-ICR Amplifiers can be modeled using separate noise sources of voltage and current noise

Amplifier Characteristics

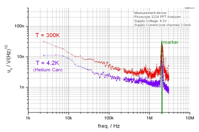

Fig. 9 Input voltage noise density frequency

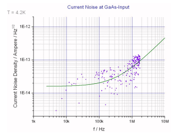

Fig.10 Input current noise density

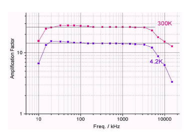

Fig. 11 Amplification vs.

5. Experimental Results

Mass Spectrometer : 7T KBSI FTICR
Sample : Agilent tune mix. (622 Da, 922 Da, 1521.9 Da..)

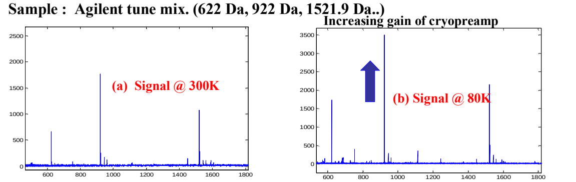

Fig.12: Agilent tune mix. Signal @ 300 K , @80 K

As shown in Fig.12 (b), signal peaks increase with LN2 cooling, which means that the gain (operation point) of the cryo preamplifier circuit changed with temperature. This can be explained by changes of internal circuit components (R, C, FETs).

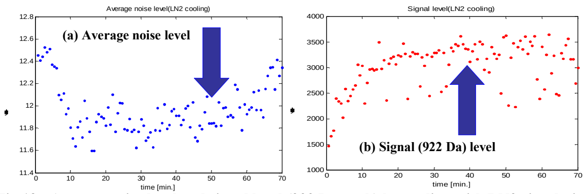

Fig.13: Average noise level and signal level (922 Da peak) by cooling with LN2 circulation

As depicted in Fig.13 (b), peaks at 922 Da increase (1500 => 3400) with LN2 cooling. It is shown that the LN2 cooling improved the gain of the cryopreamp. Absolute noise level slightly decreased (12.4 => 11.7). Which means that the signal-related noise level decreased down to a 41% level.

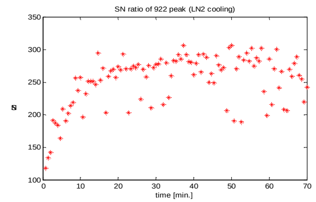

Fig. 14 Signal to noise ratio of the 922 Da peak.

S/N Ratio Improvement @ LN2 cooling (300 K => 80 K)

The increase in signal level and decrease in noise both contribute to improve the signal to noise ratio (S/N) by using the LN2 cooling. The S/N ratio is in total improved by more than 200%.

Conclusion

A combination of a cryo-cooling system and cryogenic amplifiers has been developed in order to improve the ICR signal sensitivity. As shown above, the noise level decreases and the Signal-to-Noise (S/N) ratio improves at 80 K by cooling the trap and detection circuit with a LN2 circulation.

Work in Progress

In order to improve the cryo-detection system, a modified circulation system is being redesigned to control the final cooling temperature. Furthermore, the cryopreamplifier is being modified to maximize its gain at low temperatures (80K, 4K).

Acknowledgement:
This work was supported by a grant from the KBSI (D32803)