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Development of the Fast Exchange Refrigerator for Neutron Science (FERNS)

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Abstract

The Fast Exchange Refrigerator for Neutron Science (FERNS) is being developed to enable efficient use of beamline resources by achieving a high throughput of samples in a cryogenic environment. The objective of the instrument development is to provide fully automated sample exchange and monitoring in a cryogenic environment by using computer-controlled sample handling and a cryogen-free cooling system with programmable temperature control. The inital system is targeted at high throughput beamline such as those being developed at SNS. A retrofit system suitiable for use with existing cryostats is also being developed. The research is evaluating techniques to improve thermal responsein the temperature range 3K - 500K; developing a benchtop sample can sealing system; and enabling automated sample tracking using optical character recognition of serial numbers. A prototype sample changer module is being tested at SNS using a liquid helium cryostat and a closed cycle refrigerator. The test system is being used to benchmark cooling rates for a variety of sample types and to test and perfect mechanical and software designs. A prototype instrument will be delivered to the POWGEN 3 beamline in 2006. This paper describes the FERNS instrument, presents results of tests and discusses plans for the ongoing development.

1. Introduction.

Neutrons are an increasingly important tool for investigating physical, chemical and biological phenomena [1-8]. There are 32 neutron facilities in operation worldwide and seven new sources are under construction. The Spallation Neutron Source (SNS), now under construction at Oak Ridge National Laboratory, expects up to 2000 users per year, many of them newcomers to neutron scattering [1,2,8]. The expectations of a growing user community, faster experiments, and higher throughput of samples create a need for advances in experimental support facilities. This need is especially strong in areas such as sample environment where there already exists a demand for improvements [6-9].

Neutron science relies on the use of controlled sample environments to serve the crucial role of holding samples in the neutron beam and controlling parameters such as temperature, pressure, and magnetic field. Despite the availability of commercial components, many existing sample environment systems are limited by poor reliability and complex operating procedures that lead to reduced utilization of beam time. This situation needs to be remedied in order to exploit the next-generation neutron sources that offer fast data collection rates and potentially much higher throughput of samples. Many of the problems with sample environment control can be addressed by integrating commercially available automation components with environmental control equipment and custom neutron-specific components [7-11].

The Fast Exchange Refrigerator for Neutron Scattering (FERNS) is being developed to enable automated exchange and monitoring of samples at cryogenic temperatures. Analysis of instrument requirements included a literature review [1-11] and one-on-one discussions with instrument scientists [8,12]. The main capabilities that are being designed into the FERNS instrument are:

- Reliable, high throughput of samples with remote computer monitoring, user-selected sequencing and recording of thermal history.
- Automation components that do not compromise cryostat performance.
- Extended operation without user intervention.
- Ability to sequence up to 24 samples in an unattended operation cycle with potential for expansion by addition of extra sample "carousels".
- Ability to probe samples over a range of temperatures from a few Kelvin to ~500 K.
- Compact, crane-compatible package including insert, cooling head, and sample exchange device.
- Simple load-and-seal sample encapsulation system.
- Computer logging of sample identification.

These requirements are guiding the instrument design. The new instrument is being designed to integrate with a closed cycle refrigerator for investigation of multiple samples at cryogenic temperatures. A prototype instrument will be tested at the POWGEN 3 beamline [13] at SNS in early 2006, shortly before beamline commissioning.

In our early work, [14] that is summarized in the results section, experiments were performed using a Janis model SVT-XG-400T top loading helium cryostat located at the HFIR facility at Oak Ridge National Laboratory. Cooling rate data were obtained under different conditions to determine the relative temperatures of the cryostat, sample holder and sample can. Preliminary designs for a sample "landing pad" on which the can was placed with an actuator were investigated. The novel feature that was developed using the helium cryostat test facility was to release of the sample from the transport mechanism onto a thermally-controlled "landing pad". The landing pad reduced the effective thermal mass of the sample. After the sample was placed on the landing pad, the actuator was disengaged and retracted by about 5 cm. The sample was retrieved after cooling and moved out of the cryostat.

2. Experimental

Advanced development work is in progress using a cryogen-free cooling system that can potentially allow unattended operation and sample exchange/monitoring from a remote location. The experiments are being performed using the SMASH (SNS Multiple Adaptive Sample Housing) test facility at ORNL/SNS. This test facility was constructed to SNS design specifications by Advanced Research Systems and it uses a DE-210 cooling head. A diagram and photograph of the SMASH test facility are shown in Fig.1.

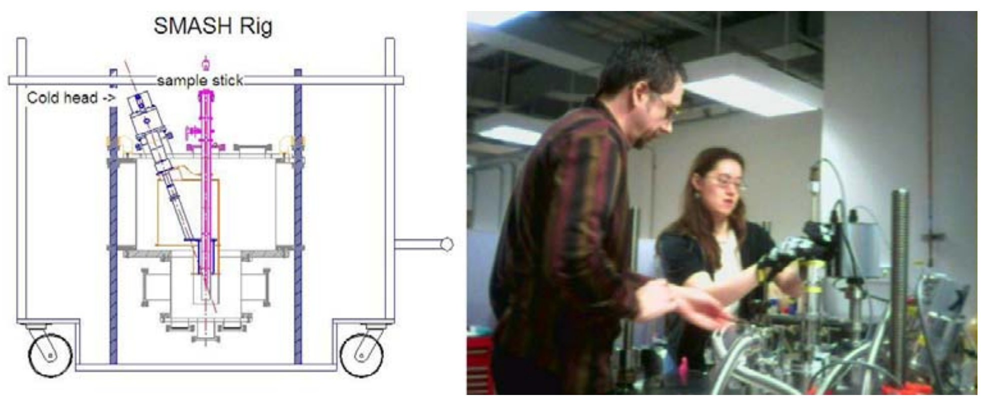

Figure 1. Left: diagram of the SMASH rig showing the location of the closed cycle refrigerator (ARS model DE-210) and sample "stick" in the re-entrant well. Right: photograph of the top plate area of the SMASH showing ports and instrumentation. The pumping system and compressor for the refrigerator are located on the floor near to the rig. The stick was instrumented with silicon diode sensors and a heater that were operated using a CryoCon Model 34 controller.

The objective of the tests was to measure the cooling rates of selected, representative samples using a closed cycle refrigerator. The sample information is given in Table I.

Table I: Sample materials. The mass is the actual weight of material required to fill a 0.8 cm diameter vanadium can to a depth of 2 cm.

Material	Density, (g/cm^3)	Heat cap. (J/g·K)	Mass (g)	Conductivity (W/cm·K)*	Energy (J)#
Alumina powder	4.0	0.775	1.48	0.35	332
Alumina shot	4.0	0.775	1.92	0.35	432
Copper powder	8.9	0.385	2.15	4.01	240
Copper shot	8.9	0.385	4.32	4.01	482 (322)
Nylon pellets	1.2	1.7	0.54	0.003	266
Steel shot	7.9	0.45	4.09	0.52	534

* Conductivity values are for the solid. #Energy for a temperature change of 290 K assuming constant heat capacity. The values given in the table are upper limits since the heat capacity decreases considerable at cryogenic temperatures and in many cases, the thermal conductivity increases at low temperature. For example, the "corrected" heat content of the copper shot sample would decrease from 482 to 322 J when it is corrected for changes in heat capacity.

The sample materials were chosen to bracket a range of thermal properties (heat capacity and thermal conductivity), density and form (fine powder to shot) that would represent a variety of sample types that might be used in the facility. Sample material was loaded into a 0.8 cm diameter vanadium can that was instrumented with a silicon diode temperature sensor (Lakeshore model DT-670B-SD) that was covered to a depth of ~ 1cm by the sample material. The mass of the can, sensor and aluminum cap was about 12 grams. The mass of sample ranged from ~0.5 to 4 g and it is given in the fourth column of Table I. The can was backfilled with helium and sealed with a metal gasket. The lid of the can was equipped with a special cap assembly that latched onto the FERNS actuator. The actuator could be operated by rotating the stick to disengage a “bayonet” type locking device to release the can. Several images of the sample can are shown in Fig. 2.

Sample cans were delivered to the landing pad by three different means: (i) using a vendor-supplied stick equipped with FERNS bayonet; the can was engaged to stick, delivered and locked into landing pad, disengaged from the stick, and the stick was retracted by about 5 cm; (ii) using a lightweight stainless steel stick following the same procedure as (ii); and (iii) using a can suspended by sensor leads and lowered into the cold zone of the sample tube such that the can was not mechanically engaged into the landing pad.

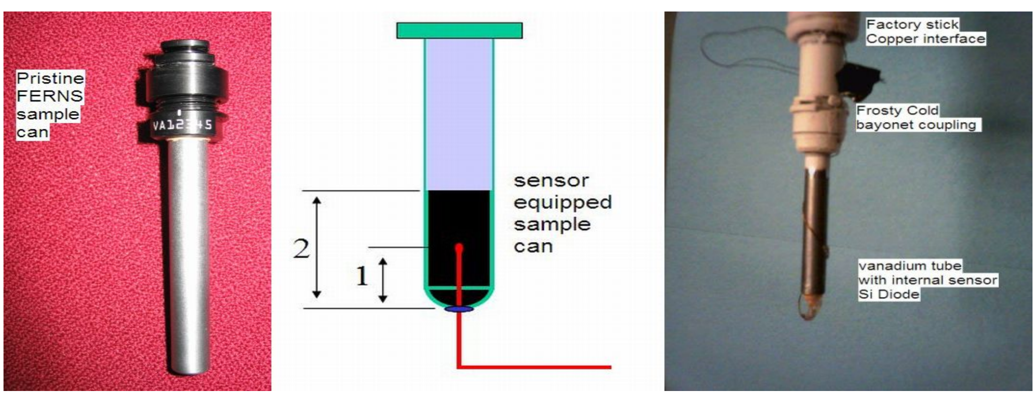

Figure 2. Left: photograph of a vanadium sample can with lid equipped with a device for latching to the FERNS actuator. Note the index number on the can flange. Center: diagram showing location of the temperature sensor, the numbers are the depth of the sensor and sample in cm. Right: photograph of can attached to the stick shortly after removal from the cryostat.

Temperature data were acquired using a CryoCon Model 34 controller. Three measurements points were used: (i) inside the sample can, (ii) the stick approximately 10 cm above the bayonet mount, and (iii) the landing pad/variable temperature insert. The three temperature channels were acquired on a common time base. Cooling experiments were performed with helium "exchange gas" backfilled to a pressure of 1 atmosphere in the cryostat before sealing or with the system pressurized with helium to about 20 kPa (3 PSI) throughout the experiment. A few runs were made with the system evacuated to a pressure of a few Pa. The cooling rate under vacuum was much smaller, after 2 hours the sample was at 50 K and the stick temperature was at 170 K.

2.1. Sample tracking and sample can sealing system.

In anticipation of the required high sample throughput, the FERNS development includes investigation of automated sample tracking and rapid sample can sealing capabilities. Sample tracking is enabled with a unique seven character identifier that is laser engraved onto the sample can lid. The characters can be read using a video camera equipped with optical character recognition and storage capabilities.

Sample cans are sealed under dry helium that provides an inert environment and acts as a heat exchange medium to increase heat transfer from the sample to the can walls where it is extracted by the cooling system. In prior work, canning was usually performed in a dry helium-filled glove box or glove bag. In this work, a simple caning system is being developed. The preliminary design of the canning system is illustrated in Fig. 3.

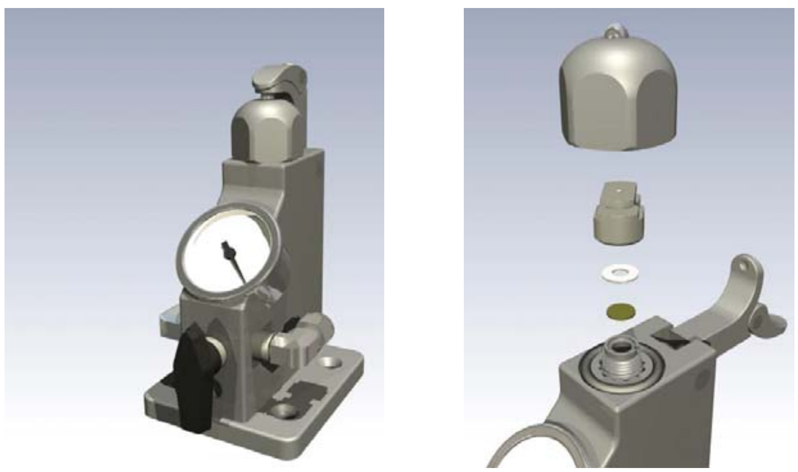

Figure 3. Illustration of the prototype bench top sample canning system. Left: overall system. Right: section showing a can in place. The operation procedure is simple to enable rapid canning of multiple samples. A can with sample is placed into the cell and the lid is loosely installed. Gas is evacuated from the can and it is backfilled with dry helium by adjusting the position of the three-way valve. The procedure is performed two or three times if necessary to remove residual air from a powdered sample. The can is then sealed under helium by tightening the hexagon cap nut on the device. The can is then removed and ready for use.

3. Results

3.1. Measurements with Janis cryostat.

A prototype sample exchange system, sample holder, and several sample tubes were constructed and tested using a Janis SVT-XG-400T helium cryostat. The system employed a Parker model 6K4 stepper motor controller that was integrated using a combination of modified purchased and custom-made parts. Photographs of the system and components are shown in Figs. 4and 5. The test instrument was controlled from a laptop computer *via* a LabView control program. The temperature of the sample and variable temperature insert were measured with silicon sensors bolted to the parts. Data were acquired with a Lake Shore model 340 controller. Sample insertion, "parking", retrieval, and re-insertion of a new sample were demonstrated under remote computer control. The cool down rate was measured under a variety of operating conditions and over a range of ambient gas pressures. The effect of introducing a sample at ~300 K and ~50 K was investigated to determine the effect of pre-cooling of the sample on the overall cool down rate. Temperature time data for representative cycles are presented in Figs. 6 and 7.

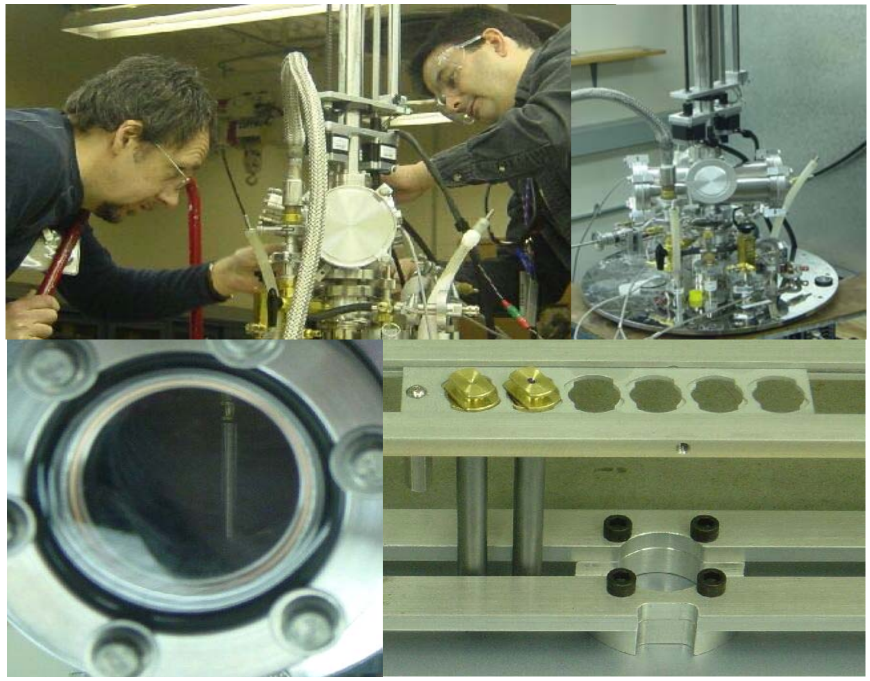

Figure 4. Images of the early test facility installed at Oak Ridge National Laboratory for testing in March, 2004. Top left: adjusting the FERNS instrument. Top right: the sealed linear actuator used to translate the sample into the variable temperature insert and onto the cryogenic "landing pad", the horizontal sample storage chamber and the drive motors are visible near the center of the image. The translation device was mounted onto the top plate of a Janis model SVT-XG-400T cryostat for testing. Bottom left: a sample held by the bayonet mechanism located inside the controlled environment. Bottom right: multiple sample holder with two samples showing the developmental version of the latching point on the top of the sample tube.

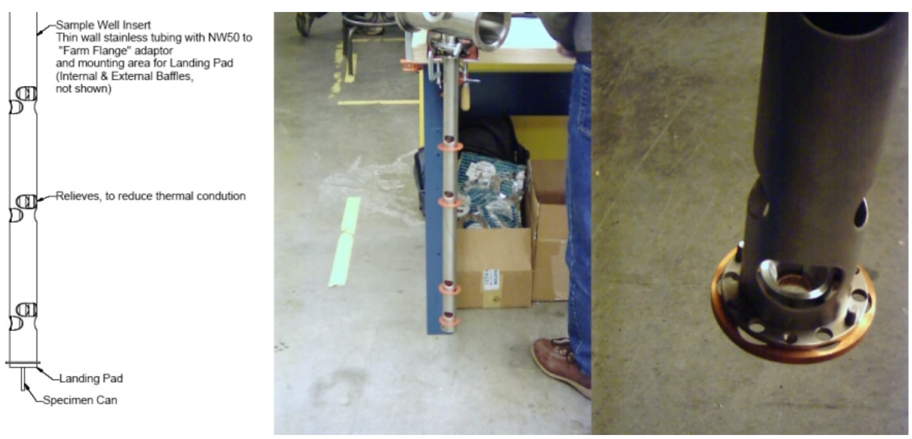

Figure 5. Left: assembly drawing of the variable temperature insert, landing pad, and sample. Center: photograph of the insert being outfitted with baffles and prepared for integration and testing. Right: close-up view of the landing pad, designed to mate with the custom sample holder which uses conventional vanadium sample cells. Note that the landing pad insert is installed into the permanent well of the cryostat, and does not move during FERNS operation

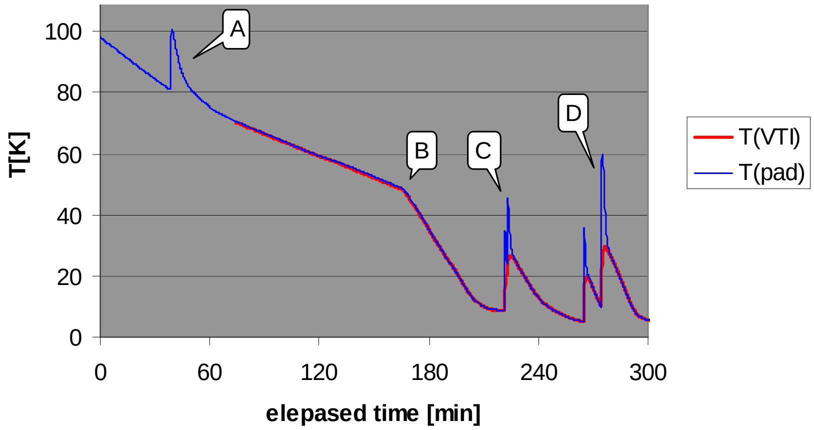

Figure 6. FERNS early prototype test data. Two separate temperatures were monitored during the above tests: "T(pad)" is read from a sensor attached to the landing pad, and "T(VTI)" from the vendor-supplied sensor attached to the variable temperature insert, an integral part of the cryostat. The plot corresponds to a sequence of events starting with the initial cryostat cool down (slow helium flow/modest cooling rate), insertion of a sample to the landing pad (A), increase of helium flow/cooling rate (B), sample retrieval and replacement of pre-cooled sample (C), and finally retrieval and insertion of a sample at ~300 K (D).

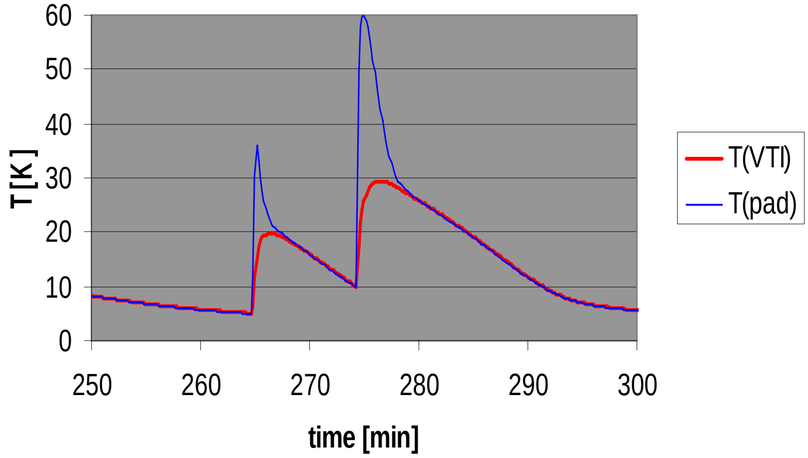

Figure 7. Expanded view of the temperature-time trace showing first a retrieval and then insertion of a sample at ca. 300 K. Note the lag in response between the temperature of the sample landing pad and the variable temperature.

In summary, the sample transfer system was demonstrated with an exchange time of less that one minute. Insertion or removal of a sample resulted in a temperature "spike" as heat transferred from the warm sample to the cold "landing pad" with embedded sensor. The achievable cool down rate in this system was not significantly affected by the initial temperature of the sample.

3.2. Measurements with SMASH rig.

The introduction of a sample into the SMASH rig sample tube at ~300 K results in a significant temperature "bounce" within the tube. Data for the two stick designs is presented in Fig. 8.

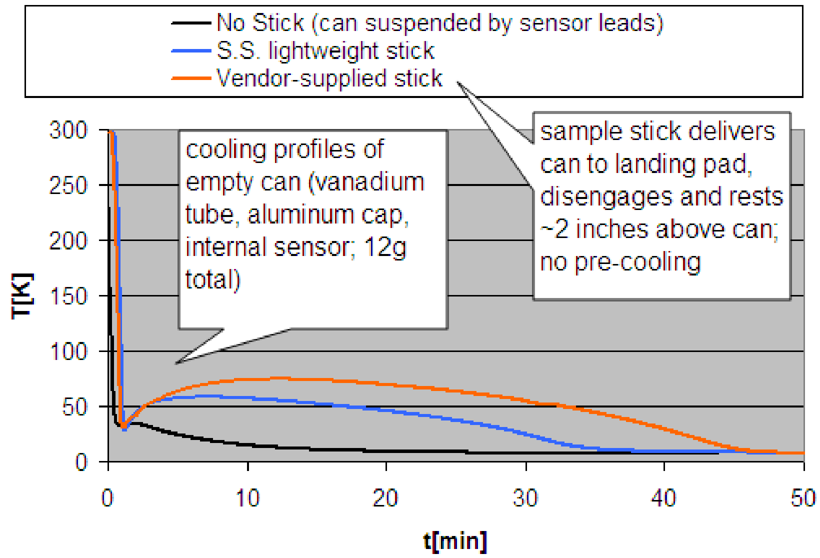

Figure 8. Temperature-time data for an empty sample can inserted into a pre-cooled cryostat using a vendor-supplied and a lightweight stainless steel sample handling stick.

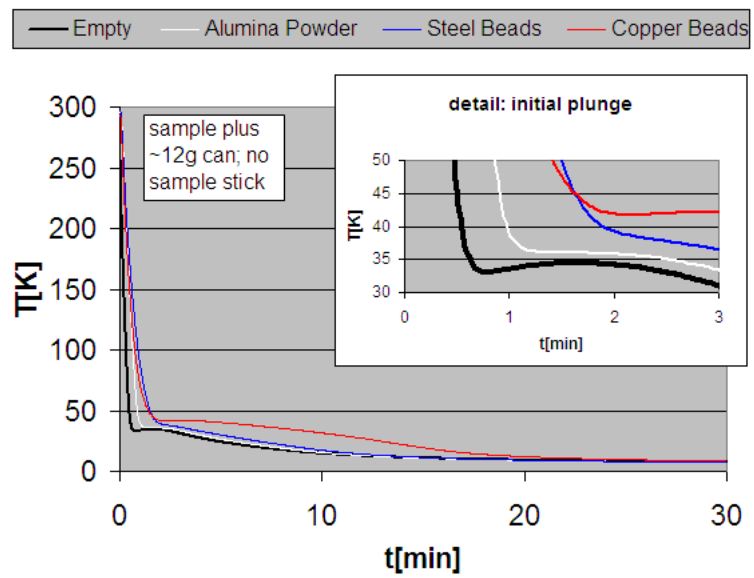

Figure 9. Comparison of cooling rates for different samples. These tests were performed by suspending the sample can on a thin wire that also served as the electrical connection to the temperature sensor embedded in the sample can.

The temperature "bounce" occurs despite mechanically disengaging from the sample can once it is locked into the landing pad. The initial quench of the can is practically the same in all cases, the bounce occurs a few minutes after the can quenches. The results show that there is rapid equilibration between the can and its surroundings, and slower heat exchange between the stick and surroundings, probably due to the greatly different thermal masses of the stick and can. The relatively slow recovery time after the bounce indicates that heat exchange between the sample tube and cold head is also slow.

Effects of sample heat content are illustrated in Fig. 9. All samples rapidly cool to ~40 K, then a slower cool down to base temperature (~ 6K) occurs. The samples with the highest thermal mass (copper and steel) are slowest to cool.

To better understand the temperature distribution in the cryostat, the temperature was monitored at three locations. "*Tvti*" is the reading from a vendor-installed sensor which is indirectly attached to the sample tube, or variable temperature insert (VTI). The VTI sensor is actually mounted on the interface between the cold head and the VTI, and its reading is often considerable lower than *Tcan* (temperature within the sample can) and *Tstick* (temperature measured on the tip of the sample stick). It is planned to add a sensor to the landing pad in future experiments. Measurements of temperature at the three locations in the system are presented in Fig. 10.

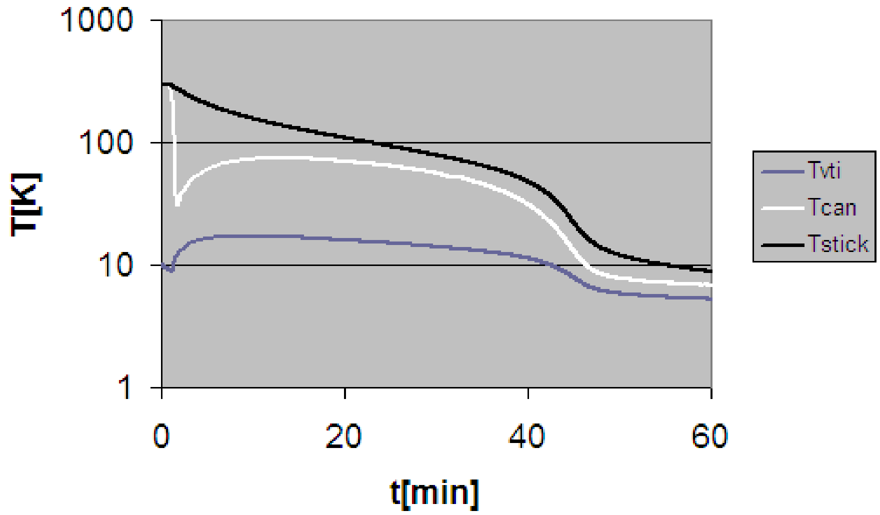

Figure 10. Comparison of temperature data measured at different points in the system

The temperature bounce after insertion of the sample is the result of heat introduced by the sample stick. In order to achieve rapid cool down using a closed cycle refrigerator, pre-cooling of the sample stick and possibly the sample itself is being evaluated. The effect of pre-cooling on the sample temperature was simulated by gradually inserting the sample stick into tube as noted on Fig. 11. With the stick resting about 15 cm above the pad, the sample quenches below 10K while the stick slowly cools. Surprisingly, the sample quench under these conditions is even better than full insertion without a stick. Final delivery to the landing pad was made after the stick cooled below 100K.

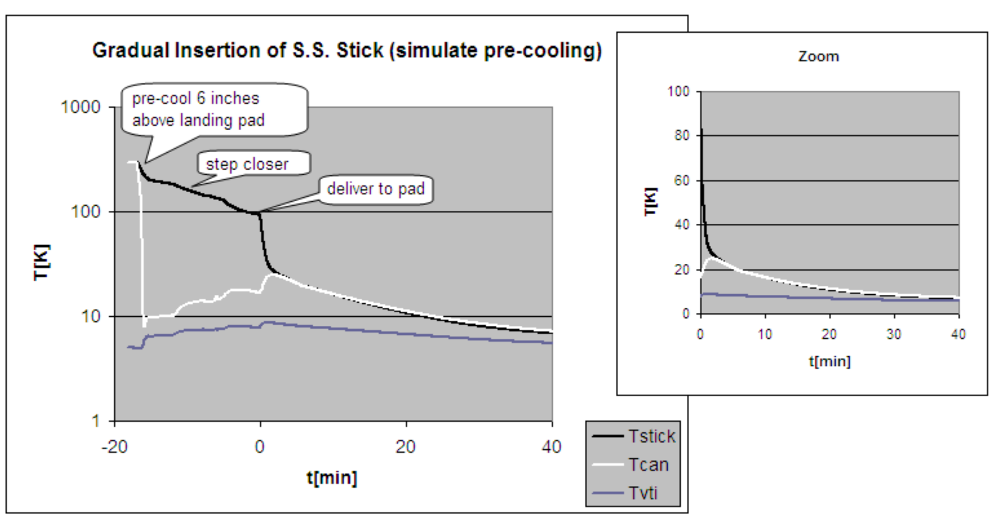

Figure 11. Effects of simulated pre-cooling of the sample.

4. Discussion.

4.1 Sample exchange

The sample exchange system that uses a carousel to store samples and select them was demonstrated on the bench and operated under computer control. The system is being integrated into the FERNS instrument and being developed as a retro-fit component that can be added to existing cryostats. The samples are mounted into a carousel that can hold 24 can. Additional carousels can potentially be loaded using a pick and place system enabling unlimited numbers of samples to used in a fully automated system.

4.2 Cooldown rates

The main mechanism of heat transfer from the can to the cooling head was via the "exchange gas". It is clear that the sample well must be backfilled with gas, preferably helium, in order to achieve useful cooling rates. With an exchange gas, even with the current system, cool down rates were on the order 30 minutes from 300 k to ~ 6K. With further optimization and reduced heat input from the stick, the goal would be to reduce cooling time to less that 10 minutes. It would be nice to have a some sort of simple model, but we may have to save that for a later date

4.3 Temperature control issues

So far, the work has focused on rapid cooling rates required to achieve fast throughput of samples in a cryogenic environment. Preliminary measurements presented in Fig. 12, indicate that achieving control of cryogenic temperatures in an area that will require further research and development.

In its current configuration, the SMASH rig provides slow and rather unresponsive temperature control in conditions where the heating system is operating with the refrigerator. Work is in progress to evaluate the use of "thermal switch" [15] type links that would allow enhanced control of temperature.

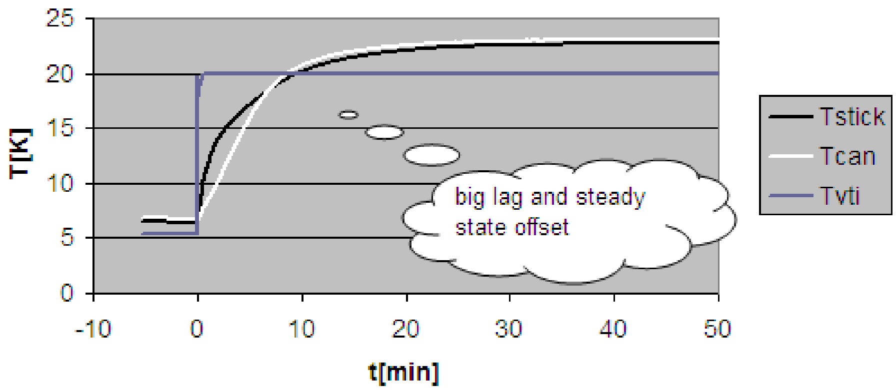

Figure 12. Comparison temperatures at three regions with a set point of 20 K. The can contained ~0.5 g nylon and the stick was disengaged and retracted by 5 cm. Maybe the thought cloud is a little to informal for a paper, I will remake.

5. Conclusions.

The FERNS system can handle samples in a controlled environment. Sample exchange can be achieved in a period of less than one minute, the current limitation on sample turn around time is the cool down rate from insertion temperature to a few K. Measurements on several sample materials indicate that even though the conductivity and heat content of the sample varied significantly, the overall cooling rate not strongly affected by the sample. The stick has a major effect on cooling rate since it has a very large thermal mass compared to the sample and can. Filling the sample well with helium "exchange gas" had a very strong effect on cooling rates. Conduction through contacting metal components is a poor heat transfer method and exchange gas is essential to achieve fast heat transfer. The use of a disengaged stick helps to reduce heat introduction, but a cooled stick would provide a significant advantage. The use of pre-cooling of the stick and potentially the sample would provide a significant advance. Temperature control issues present a significant challenge. Work is in progress to investigate use of a "thermal switch" approach to decouple the second stage of the cold head at temperatures above 40K.

Acknowledgments.

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