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A "HOT STAGE" DISPLEX®: 20 - 800K IN A NEUTRON SCATTERING ENVIRONMENT

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ABSTRACT

A "Hot Stage" Displex® has been implemented at the Intense Pulsed Neutron Source (IPNS) for performing neutron diffraction experiments in the temperature range 20K to 800K. The new piece of ancillary equipment allows for extended temperature range studies through room temperature without having to change sample containers or environments. The hot stage was designed for and installed on a refurbished closed-cycle helium refrigerator cold head. The unit employs four (4) single crystal sapphire stand off rods to provide a thermal conduction path to the sample at low temperatures, where sapphire is a conductor, and to isolate the sample and heater at high temperatures, where sapphire is an insulator. As with a standard closed cycle refrigerator, the first stage heat shield is held at about 20K throughout the temperature range. The second stage heat shield, along with the cold finger holding the sample container, is heated to the desired temperature with a resistance heater. Temperature is monitored by a silicon diode located in the second stage and a platinum resistor mounted in the hot stage. The temperature is controlled by a microprocessor-based controller with an RS-232 communications link to the neutron scattering instrument's data acquisition computer, allowing for run file temperature control. To ensure thermal equilibrium throughout the sample, the sample container is typically filled with one atm. of helium. A new flange design for the standard closed-one-end vanadium sample cans has been incorporated to contain the helium atmosphere throughout the extended temperature range. Preliminary tests on a standard sample have shown a thermal gradient of <1 K/cm at 10K and at 800K over the length of the sample container. Cool-down times to 10K are on the order of 90 minutes, slightly longer than a standard Displex®. The neutron scattering background compares favorably with a standard closed-cycle refrigerator or mid-range furnace currently in use at the IPNS.

1. Introduction

Since the inception of the IPNS there has been a need to measure the structural characteristics of materials at elevated temperatures. For many years, a resistance heater furnace, the Coffee Can Furnace (so named for it's visible appearance rather than it's actual construction material,) has served the neutron diffraction instruments well. A main fault of the Coffee Can Furnace was the lack of a cooling load that the resistive heaters could work against. This lead to a large thermal time constant in the system with an accompanying slow response to set point changes. Once heated above ambient, the return to room temperature followed an exponential tail. With the increased usage of furnace based experiments at the IPNS in recent years a real need was identified for a room temperature to 500°C furnace that was more responsive to set point changes. With the increased interest in polymers, a below ambient, i.e. -25°C, capability was also thought advantageous.

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2. Design and Construction

The design of the Hot Stage[1] is that of APD Cryogenics, Inc. in response to an inquiry from the IPNS about the possibility of operating a Displex®[2] at above room temperature. Users at IPNS have repeatedly asked how high in temperature could they run the closed-cycle helium refrigerators. A limit of 350K was typically given, this figure based on a number of factors concerned with the construction materials of the expander module cold head. APD Cryogenics engineers came back with an 800K Interface that could be mounted on their Model DE108 Expander Module. Figure 1 is a photo showing the Hot Stage Interface mounted on a refrigerator cold head. The temperature range of 10K to 800K is achieved by utilizing the unique thermal properties of single crystal sapphire. Figure 2 shows the thermal conductivity of sapphire plotted along with that of several materials used in the construction of furnaces for neutron scattering. At low temperatures, <100K, sapphire is a thermal conductor on the order of copper, aluminum, or even silver. The sapphire provides a thermally conductive path for sample cooling by the expander module cold head. At higher temperatures, sapphire becomes a thermal insulator on the order of stainless steel or vanadium. This thermally isolates the Hot Stage and sample from the cold head and allows the resistance heater to heat the sample above ambient. Figure 3 shows the temperature of the cold head and Hot Stage as a function of the Hot Stage set point. Heating from a base temperature of 20K, the temperature of the cold head and Hot Stage coincide until about 100K where the thermal properties of the sapphire begin to isolate the Hot Stage from the cold head. This transition is seen clearly in the plot of the percent power output to the heater. The cold head never heats up above 200K while the Hot Stage is heated to 800K.

3. Operation

The Hot Stage Displex® is operated similar to a standard closed-cycle refrigerator. The sample is loaded under an atmosphere of helium into an aluminum gasket sealed vanadium. The sample can is then screwed onto the Hot Stage. Care must be taken in attaching the sample to the Hot Stage due to the fragility of the four sapphire rods. The second stage and first stage heat shields are then attached and the unit installed into the sample well of the neutron instrument. A high vacuum of better than 10-5 vacuum is desired to avoid cryopumping air and moisture onto the heat shields. Initial cool-down times to 20K are on the order of 90 minutes, slightly longer than a standard Displex®. The temperature is controlled by a microprocessor-based controller[3] with an RS-232 communications link to the neutron scattering instrument's data acquisition computer[4], allowing for run file temperature control[5]. Temperature stability is ±0.1K throughout the temperature range. Preliminary tests on a standard sample[6] have shown a thermal gradient of <1

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[1] 800K Interface, APD Cryogenics, Inc, Allentown, PA.

[2] Displex® is a registered trademark of APD Cryogenics, Inc, Allentown, PA.

[3] Lake Shore Cryotronics, Inc, Model 330.

[4] Typically a DEC VAXStation.

[5] TEMtur temperature control program, R. K. Crawford, IPNS, ANL, 1996.

[6] Helium filled 7/16"(1.1 cm) diameter x 5 cm vanadium sample can.

K/cm at 10K and at 800K over the length of the sample container. Control parameters, proportional band (gain), integral (rate), and derivative (reset), can be autotuned by the controller at each set point change, although in practice this is not usually deemed necessary.

4. Results

Beyond the thermal characteristics of the furnace, the crucial test of any piece of ancillary equipment is the neutron scattering data obtained. Already the Hot Stage Displex® has been used for experiments on the QENS spectrometer and on the SEPD and GLAD powder diffractometers. Figure 4 shows a comparison of raw TOF data taken on QENS with a sample in the Hot Stage Displex® at 350K and with a different sample in a standard Displex® at 50K. though the samples are different, the backgrounds of the two plots in the 1500 – 15000 μsec range are comparable if not identical and quite good for the measurement.

5. Future Improvements

With the first experiments completed, we have already identified several desired improvements to the Hot Stage Displex®. The list is not all inclusive but will include the following:

- Additional thermometry to monitor the sample temperature directly.
- Cadmium or gadolinium oxide shielding on the second stage cold finger to reduce the multiple scattering background.
- The addition of a high temperature alarm and other protective measures in the control circuitry.
- Instrument specific heat shields incorporating cadmium or gadolinium oxide masks tailored to the instruments neutron beam dimensions to reduce the stray scattering background.
- Improvements to the standard sample container to maintain a helium leak tight seal over the wide temperature range of the Hot Stage.

And if the scheduling of the Hot Stage Displex®, which has been a heavily requested item, continues as it has in its first several run cycles, we will certainly consider adding a second unit to our suite of ancillary equipment.

6. Summary

A "Hot Stage" Displex®[7] has been implemented at the Intense Pulsed Neutron Source (IPNS) for performing neutron diffraction experiments in the temperature range 20K to 800K. This new piece of ancillary equipment allows for extended temperature range studies through room temperature without having to change sample containers or environments. Preliminary tests on a standard sample[8] have shown a thermal gradient of <1 K/cm at 10K and at 800K over the length

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[8] Helium filled 7/16"(1.1 cm) diameter x 5 cm vanadium sample can.

of the sample container. Cool-down times to 10K are on the order of 90 minutes, slightly longer than a standard Displex®. The neutron scattering background compares favorably with a standard closed cycle refrigerator[9] or mid-range furnace[10] currently in use at the IPNS.

More information on the new "Hot Stage" Displex® and other ancillary equipment available at the IPNS is accessible at the IPNS Web site: http://www.pns.anl.gov.

7. Acknowledgements

In addition to the help and guidance of the design engineers at APD Cryogenics, we would like to acknowledge the assistance of J. Kowalski[11], D. Leach, J. Toeller, and C. Piatak, of the IPNS NGS Operations staff, in the design, construction, and assembly of the Hot Stage Displex®. We would also like to acknowledge the assistance of S. Short and J. Jorgensen, of the Materials Science Division, in the testing of the Hot Stage Displex® and R. Connatser of IPNS for providing the raw data plots of Figure 4. The work reported in this paper was supported by the U.S. Department of Energy, BES-Materials Sciences, under contract W-31-109-ENG-38.

[9] i.e. Standard DE-202 Displex®, 10K - RT.
[10] i.e. Coffee Can Furnace, RT - 350°C.
[11] Currently with ComEd, LaSalle, IL.

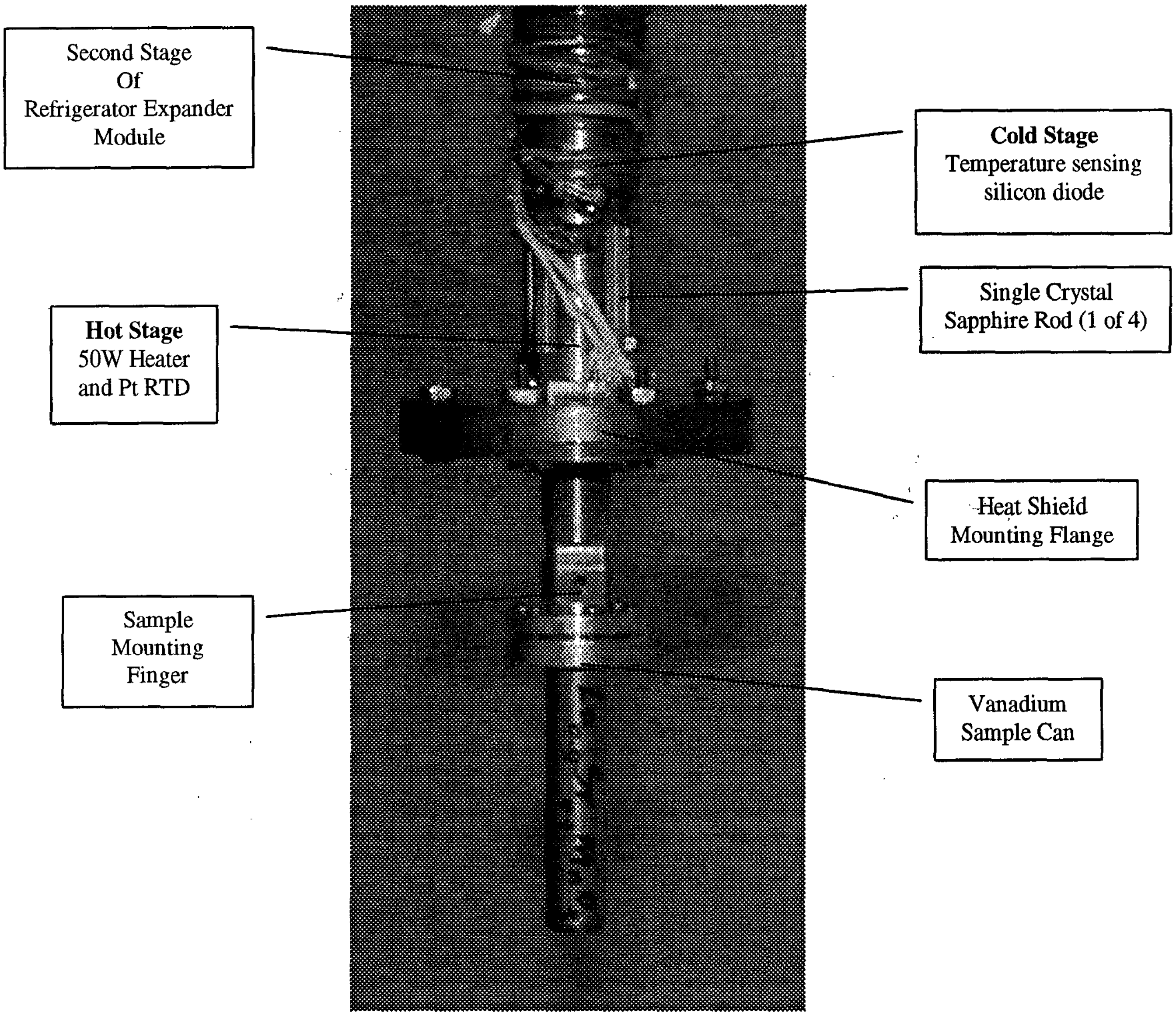

Figure 1. The Hot Stage Displex®. The sample can show is a low temperature indium o-ring sealed vanadium sample can of the type that is usually used in low temperature measurements on the powder diffractometers. A similar sample container with a flange capable retaining a helium leak tight seal throughout the temperature range of the Hot Stage is being developed.

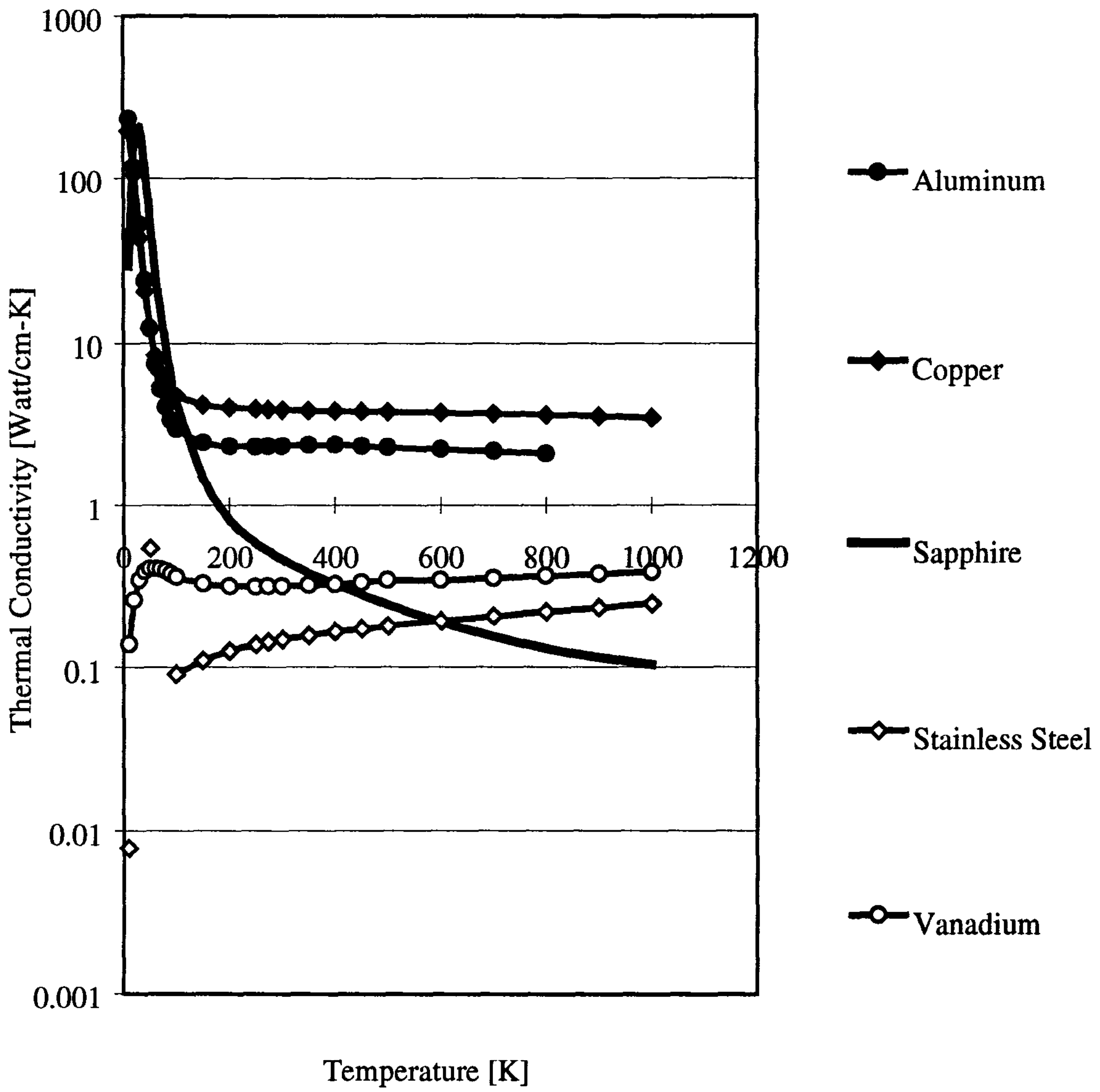

Figure 2. Thermal conductivity[12] of single crystal sapphire plotted along with that of aluminum, copper, 304 stainless steel, and vanadium.

[12] In Watts/cm-K from Thermophysical Properties of Materials, The TPRC Data Series Vol 1, 2, & 3, Y. S. Touloukian and C. Y. Ho, Eds., IFI/Plenum Data Corp, NY (1970).

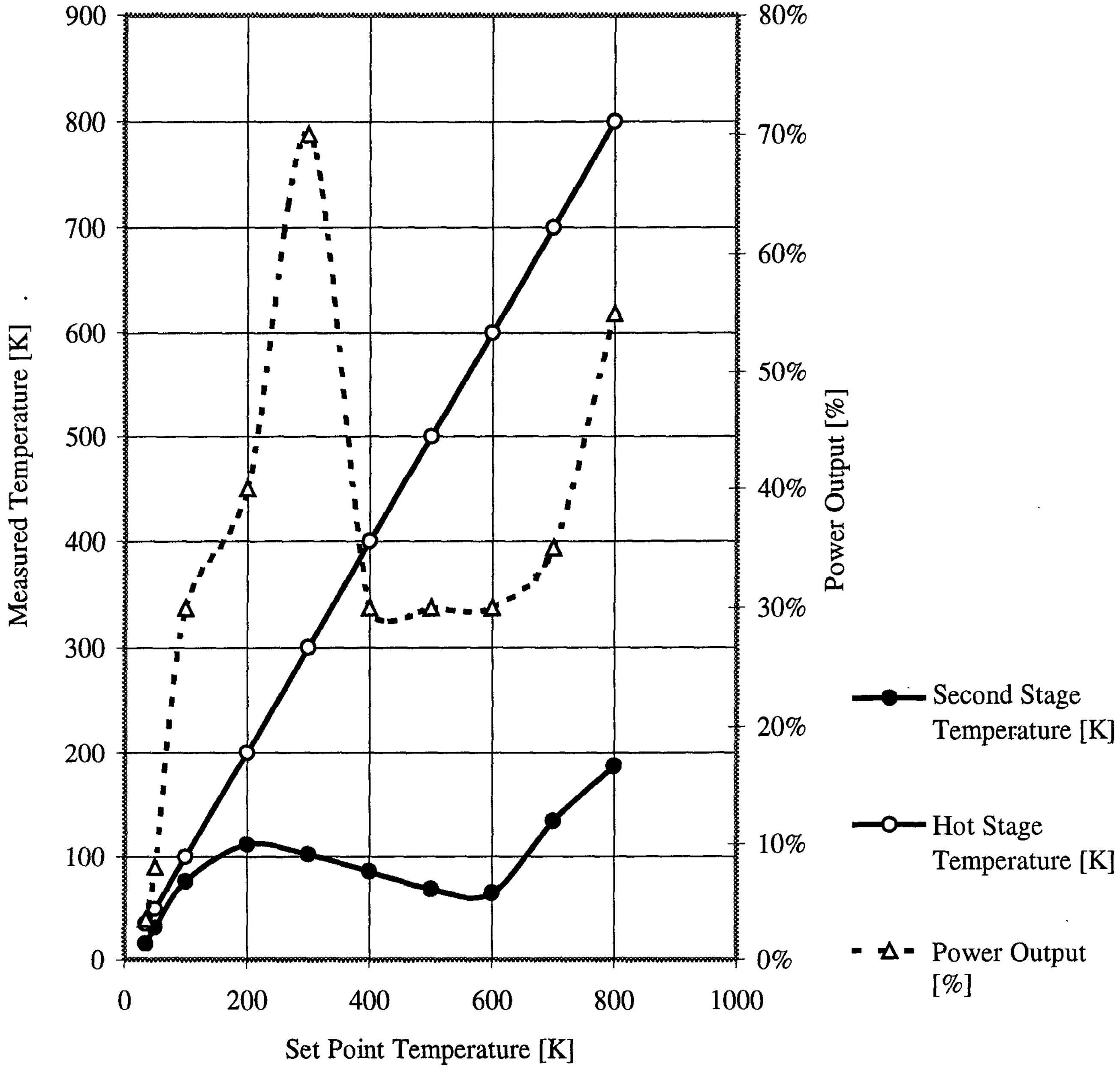

Figure 3. Heating plot of second stage temperature, Hot Stage temperature, and percent power output to heater as a function of Hot Stage set point.

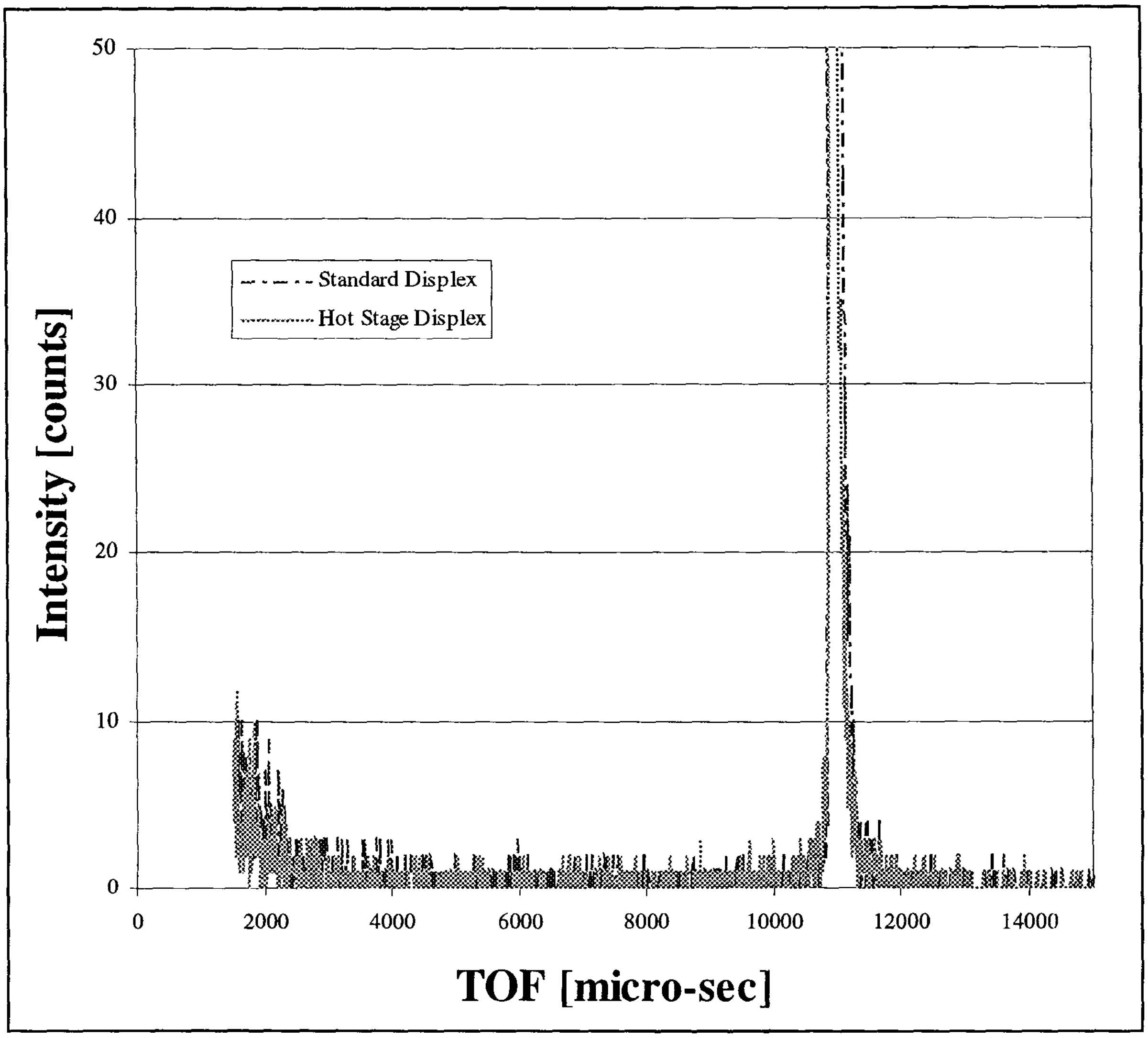

Figure 4. Comparison of raw TOF data taken on QENS with the Hot Stage Displex® and with a standard Displex®. Though the two plots are of different samples, the backgrounds of the two plots in the 1500 – 15000 μsec range are comparable and quite good.