Phase II Final Technical Report
11/17/2012

Covering the period of 8/14/2009 to 8/13/2012

DOE STTR Grant# DE-FG02-08ER86344; DOE Office of Science

Development and Neutronic Validation of Pelletized Cold and Very Cold Moderators for Pulsed Neutron Sources

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Executive Summary

Intense beams of cold neutrons are produced at several DOE facilities and are used by researchers to study the microscopic structure of materials. Energetic neutrons are produced by a high energy proton beam impacting a target. The fast neutrons are converted to the desired cold neutrons passing through a cryogenic moderator vessel, presently filled with dense cold hydrogen gas. Moderators made from solid methane have demonstrated superior performance to the hydrogen moderators but cannot be implemented on high power sources such as the SNS due to the difficulty of removing heat from the solid blocks of methane.

Cryogenic Applications F, Inc has developed the methane pellet formation and transport technologies needed to produce a hydrogen cooled solid methane pellet moderator, potentially capable of being used in a high power spallation neutron facility. Such a methane pellet moderator could double the brightness of the neutron beam. Prior to this work a methane pellet moderator had not been produced or studied.

The Indiana University LENS facility is a small pulsed neutron source used in part to study and develop cold neutron moderators. In this project cold neutrons were produced in a solid methane pellet moderator and analyzed with the LENS facility diagnostics. The results indicated that the neutron beam formed by the pellet moderator was similar to that of a solid methane block moderator.

Work carried out.

A cryogenic apparatus which produces pellets of frozen methane was constructed and installed on the IU LENS neutron facility. The device was shown to be capable of filling the LENS cold moderator cell with methane pellets. Measurements of the neutron spectra from the pelletized moderator were made which were similar to the standard solid methane moderator. This report is a summary of the tasks and accomplishments made during the project at both Cryogenic Applications F and at the Indiana University LENS facility. The following describes each of the tasks to be performed during the period and the progress achieved.

Phase II Work Plan. As proposed with progress.

Task 1: Finalize the pelletizer LENS interface geometry and safety requirements.

As proposed:

The phase I project included a preliminary pelletizer design and safety analysis. The general design interface has been established with the exceptions of some minor details which will be finalized in the beginning of the Phase II project. A preliminary safety analysis has been performed. One of the findings of the safety analysis was the need to establish an allowable working pressure of 8 atmospheres for the pelletizer chamber and cryopump. This will be established by a pneumatic test at 10% over the 8 bar, established as the maximum air-methane deflagration pressure.

Progress:

The interface configuration of the pelletizer was determined to consist of a pneumatically operated 2 3/4" conflat gate valve and a bellows assembly with a set of internal funnels to attach to the fill tube of the IU cryogenic moderator. This was constructed. In addition a test stand and a mounting stand for the pelletizer on the Lens facility was designed and constructed at IU. The initial requirement to pressure test the pelletizer chamber was removed in lieu of operating the pelletizer within a concrete block enclosure when tested outside the LENS vault. A description of the pelletizer and the IU Safety analysis is covered in task 9N.

Task 2: Complete assembly of the Mobile Pelletizer.

As proposed.

In the phase I project, the pelletizer apparatus design and the installation interface to the LENS cryogenic moderator system was developed. Work was also begun on the assembly of the pelletizer into a mobile unit that could be rapidly installed into the LENS facility. This task will complete this assembly.

The initial configuration of the mobile pelletizer shown in Fig 2.1a, included a dump chamber under the pelletizer and the cryopump above. In order to satisfy the physical constraints of installing the unit in the LENS TMR it was determined that a better configuration would be to shorten the overall height of the apparatus so that it would fit below the ceiling beams. This was accomplished by removing the dump chamber and installing the cryopump alongside rather than above the pelletizer as shown in Fig 2.1b.

Figure 22. Pelletizer Apparatus A

Figure 21B Pelletizer Apparatus.

Progress:

The pelletizer and controls were first completed in the initial configuration as shown in Fig. 2.1. This work consisted of completing the control system and wiring, the gas feed lines , liquid nitrogen feed valves and associated insulated tubing lines.

A temporary moderator chamber was connected to the pelletizer. This contained a “moderator” box made of 1/8" Lexan, 4"wide x2"deep x 5" tall OD , which is similar to the size moderator which will be used on LENS. The pellets were transported to the moderator cell through a 5/8" x 035 wall SS tube which is similar to the feed tube to be used on LENS and considerably smaller than was previously used. The “moderator” cell also contained a number of 3mm Stycast beads with internal silicon diode thermometers and a chip heaters. By pulsing the heaters, a measurement could be made of the effective cooling time constant of the pellets in the moderator. The moderator chamber was also fitted with a metal mirror which allowed the observation using an external camera of the pellets filling the cell. In addition the moderator cell was attached to an external electromagnetic vibrator with a coupling rod passing through a bellows feedthrough.

After completing the apparatus in the original configuration three test runs were made.

In the first run the supply of ethane gas ran out so the pellets were made of pure methane. The sticky pure methane pellets rapidly plugged the collection funnel in the pelletizer and prevented the filling of the moderator cell.

With 1% ethane added to the methane the following runs did not plug the transfer tube into the moderator. The methane pellets are shown filling the moderator chamber during the 20 minute defrost cycle of the pelletizer in Figure 2.3.

Figure 2.3 shows a sequence of photos of the Lexan moderator cell being filled with methane pellets. The cell has a cm scale attached to the left and an inch scale on the right. The cell is 4.75" tallx 3.75" wideX1.75" deep. The small black beads hanging in the middle of the empty cell are the silicon diode thermometers with attached heaters.

Task 3: Perform trial tests of the unit prior to delivery and installation on LENS.

As proposed:

Before delivering the pelletizer to LENS it will be tested in a configuration as close to the LENS facility installation as possible so as to identify and remedy any problems. The unit will be installed in the CAFI test facility. A test stand will be fabricated to support the pelletizer above a replica of the The LENS cryogenic moderator and the associated pellet delivery tubes which will be installed in the existing mock-moderator chamber used in the previous 03SBIR. The test stand will be delivered with the pelletizer apparatus to be used as an off-line test assembly of the apparatus before mounting it in the LENS facility. The CAFI test moderator will be conduction cooled with a high purity aluminum bar system similar to the LENS design. Refrigeration will be accomplished using a metered liquid helium system.

The phase I experiments with the polyethylene pellets also established the advantages of fluidizing the pellets with a vibrator during the fill cycle. Various vibrators and coupling methods will be investigated to establish one compatible with the LENS apparatus.

The mockup will allow verification of the loading of the pellets through the transfer tubing and allow direct viewing of the pellets filling the chamber. The tests will also establish the best “recipe” for making the appropriate quantity of pellets and delivering them into the moderator.

Progress:

During the Phase I study it was determined that the apparatus would have to be considerably shorter to fit it above the LENS experiment. This was accomplished by removing the temporary moderator chamber from the cart, lowering the pelletizer 20" into the cart, and removing the cryopump from above the pelletizer to a location adjacent to the pelletizer. The new arrangement also included the addition of a valve between the cryopump and the pelletizer. The valve will allow the cryopump to be subcooled prior to the pellet release cycle. In addition a gate valve which will interface the pelletizer and the LENS experiment was added. The pellets now are passed through the gate valve with a series of funnels followed by a funnel/bellows assembly which will attach to the top of the LENS moderator apparatus. Below the bellows a transfer tube with a dogleg offset similar to the fill tube on LENS moderator connects the pelletizer to the CAFI moderator chamber which is now mounted under the pelletizer cart in a position similar to the LENS moderator.

The pelletizer and mock moderator chamber after modifications to enable operation on the LENS facility are shown in figures 3.1 to 3.5

Fig 3.1 Front of pelletizer cart
with control panel

Fig 3.2 rear of pelletizer
showing pelletizer behind the
cryopump

Fig 3.3 Moderator chamber
under the pelletizer

Fig 3.4 Interface gate valve mounted below the pelletizer.

Fig 3.5 Interface bellows with movable funnel.

The initial testing of the apparatus is discussed in Task 2. The lowered configuration was accomplished.

This was accomplished by:

- Removing the mock moderator chamber from the cart.

- Lowering the pelletizer chamber 22" and fastening it to the cart.

- Removing the Cryopump and remounting it at the back of the cart.

- Adding a 2" angle valve to the cryopump

- Fabricating a crossover pipe from the pelletizer to the cryopump.

- Fabricating a 2 " tee section with an internal funnel to the pelletizer output to the cryopump valve.

- Remounting the gate valve to the pellet exhaust

- Fabricating and installing a bellows/funnel moderator adapter below the gate valve.

- Fabricating and installing a transfer line with a dogleg bend similar to the LENs moderator pipe.

- Adding a relief valve to the cryopump chamber.

- Building a stand to mount the pelletizer over the "pi" where the "mock" moderator would be installed.

- Relocating the moderator into the pit.

- Rebuilding the LN2 solenoid valve manifolds to accommodate the new pelletizer position.

- Relocating the vacuum pumps on the stand.

- Adding control switches for the new valves.

The modified apparatus was tested several times, the first few runs uncovered some minor changes needed, followed by several runs which successfully filled the test moderator with pellets.

Task 4: Develop and test diagnostics to monitor and measure the pellet packing density.

As proposed:

In an operating neutron facility using a pellet moderator it would be desirable to be able to verify and quantify the uniform filling of the moderator. A remote camera system coupled with a fiber optic image transfer system will be installed and tested.

To measure the packing density the moderator will be back filled with liquid hydrogen with a mass flow controller.

Various liquid and solid level detection diagnostics are available which may be compatible with high radiation environments. Microwave systems might be an interesting area to investigate. We have previously designed and developed microwave pellet mass detectors for hydrogen and deuterium pellets. These work by measuring the frequency shift of a resonant cavity due to the change caused by the dielectric mass of a single pellet as it passes through the cavity. This measurement gives a rather small shift since we were dealing with one pellet of hydrogen which has a low dielectric constant. In the moderator case there would be tens of thousand pellets with a large dielectric constant. The moderator chamber would create a rectangular resonant cavity which could be coupled to external oscillators with a ceramic co-ax cable terminated by a small

antenna loop mounted inside the moderator chamber near the top. The fundamental frequency would be shifted by the change in dielectric constant weighted by the square of the electric field which will be peaked in the center and fall to zero at the edges. The first harmonic, which has a node in the center would be useful as a measure of the progress of the filling of the moderator. By measuring the higher harmonic resonances the mass distribution of the pellets could be further determined by a Fourier analysis.

Progress:

A test of the microwave measurement of the dielectric mass was performed. A 2"x4.25"x4" copper box was fabricated to mimic a moderator cell. This cavity has a theoretical fundamental TE(011) frequency of 2.03 Ghz. [note. The web site <http://www.falstad.com/embox/> Has an interactive applet which shows the various modes of the cavity.] The TE(011) mode has the electric field perpendicular to the faces of the moderator cell, the field is high in the center and falls off at the sides, top and bottom of the cell. Coupling to this mode can be achieved with a loop antenna with the magnetic fields passing through the loops. TE(101) mode has the electric fields in the vertical direction, which would change linearly with the pellet fill depth, but several redundant modes complicate things. A simple test setup, shown in Fig 4.1 and 4.2, was assembled with voltage controlled oscillators, splitters, directional coupler, detector, frequency counter and a digital voltmeter. We first tried using a single antenna loop with the detector looking at the reflected wave. The advantage is that this would only require one coax to the moderator, but this was also more difficult. Using a separate antenna loop for the detector gave a very strong signal shift at the resonance. Using some 3mm polypropylene pellets, which are manufactured for use in plastic injection molding equipment, the frequency shift was measured with various fill depths as shown in Fig.4.3.

The microwave system appears to be a very robust and precise quantitative measurement of the dielectric mass and packing density of the moderator. Very small (.05mm) stainless steel coaxes are available which should allow the signals to be passed down through the moderator fill tubes.

Fig 4.1 Microwave pellet level detector , test setup.
From copper moderator cell, clockwise. DVM,
frequency counter, pellets,microwave
components(coupler, VCOs, splitters, mixer, detector).

Fig 4.2. Antenna loops.

Figure 4.3 shows the shift in frequency of the moderator cell as plastic polypropylene pellets are added.

Fig 4.3 Shows frequency shift as pellets are added to the moderator cell

Task 5: Deliver and assist in the installation of the pelletizer on the LENS apparatus.

As proposed:

The pelletizer will be prepared and delivered to Indiana. This should be possible with the unit assembled in the mobile frame. IU will test the ability of the CAPI pelletizer to interface with the LENS moderator test bed and explore the efficiency with which methane pellets may be loaded into the test bed. This task will include the installation of the LENS cryogenic moderator to the CAPI pelletizer and support structure and the purchase of a compressor to allow the test bed to run outside of the LENS Target Moderator Reflector assembly. In-situ diagnostics such as a borescope camera and ultrasonic transmission experiments will be utilized to study the filling of the moderator and confirm that the pellets may be transported through tubing qualitatively similar to that likely to be encountered in real applications at a spallation neutron source.

Progress:

The pelletizer apparatus (without the mock moderator chamber) was delivered (fully assembled) to The Lens facility on September 7, 2010.

After completion of the Safety analysis report and constructing a test stand the pelletizer was mounted to one of the LENS cold moderator chambers (outside the TMR vault). The equipment was checked out and a few minor repairs of broken wires were made. The apparatus was enclosed by concrete block barriers and inspected and tested for leaks by IU safety personnel.

The initial attempts at filling the moderator were unsuccessful, as a blockage occurred in the pellet transfer line. Inspecting the transferline in the moderator apparatus with a boroscope found a blockage caused by a weld where the transfer line went through a pair of bellows. This was corrected by fitting a smooth plastic sleeve liner into the transfer line past the obstructions. With the liner in place the moderator was successfully filled and significant frequency shifts were observed on the microwave apparatus.

Task 6: Install and test the pelletizer on the LENS beam line.

As proposed:

Once adequate experience with transfer of methane pellets from the pelletizer to the moderator vessel has been demonstrated, the apparatus will be installed on the LENS facility. Neutronics testing of pelletized moderators with various fill liquids will be conducted. This task will also include confirmation of the transferability of techniques developed at Cryogenic Applications for extracting pellets from a moderator to the LENS moderator design.

Progress:

The tests of the moderator outside the vault was not done, in favor of testing the pelletizer as a moderator.

Task 7: Install and test the cryogenic pellet moderator system installed as the LENS moderator.

As proposed:

Up to three experiments will be conducted in which the pelletizer is mounted to load pellets into the LENS moderator while located within the LENS TMR. In these experiments, neutron spectra and emission time distributions will be measured in order to test our ability to adequately model such inhomogeneous moderator materials using existing Monte Carlo computer simulation codes. We will be particularly interested in studying the neutronic reproducibility for various fill conditions, the impact of different fluid media (helium, hydrogen, para-hydrogen, ortho-hydrogen) on the neutronic performance of the moderator.

Progress: These experiments are covered in the IU progress report.

Task 8: Prepare the Phase II renewal progress report .

The renewal report was submitted.

Phase II Work plan for the second year: As proposed

The second year program will continue the experiments on the LENS facility. In addition a second larger pelletizer will be prepared and tested.

The 600cc pelletizer used for the first years program was chosen because it was small enough to fit in the LENS TMR. A larger 1100cc methane pelletizer was constructed and tested in the 03 SBIR. The pelletizer is shown in figures 6,7 and 8. This unit also has the advanced capability of being able to accumulate the pellets and rapidly filling the moderator cell as shown in figures 11,12 and 13 which were from a test which demonstrated the filling of the moderator cell in 10 seconds. The rapid fill technology, is not necessary for the LENS experiments, but would be important for use on high power pulsed neutron sources such as the SNS, since the pellets in the moderator would need to be replaced frequently.

Progress.

Tasks 9 through 12 all related to replacing the 600cc pelletizer on LENS with the 1100cc pelletizer were not done due to a change in the scope and time schedule of the project. Two new tasks were added to better support the IU experiments. Tasks 9N (The completion of a detailed description and operations procedure for the pelletizer in support of the IU Safety documentation) and 10N (the design and construction of a computer controlled operating system for the 600cc pelletizer.) (see below)

Task 9: As proposed

The 1100cc pelletizer in Fig 7 and 8 is shown filling a large moderator cell with methane pellets in Fig 13 and 14.

Figure 7: Shows the 03 SBIR methane pelletizer with the 24" OD SS chamber installed. The apparatus is cooled with LN₂.

Figure 8: Shows a batch of methane pellets on one of the lowered shutters, just prior to releasing them into the moderator vessel. The empty honeycomb panel is seen at the top.

Figure 13: Shows the moderator being filled with methane pellets, $t=4$ sec.

Figure 14: Shows the moderator filled with 1.1 liters of pellets, $t= 10$ sec.

The 110cc pelletizer is too tall to fit in the LENS TMR. The unit is taller than the 600cc pelletizer because the smaller pelletizer panels are contained in a Dewar configuration, whereas the 1100 cc pelletizer utilizes foam insulation which has a demountable flange, allowing for easier access to the panels. The large flange which has an elastomer o-ring seal and top dome are kept at room temperature. The pelletizer panels are situated in the lower half of a deep chamber. To accommodate the low ceiling in the LENS facility, a new design, would allow the flange to drop to -40C , which will allow for the chamber to be shortened considerably. A low temperature o-ring seal rated to -54C will be used. The flange and upper dome will then be foam insulated and an internal multilayer superinsulation Mylar blanket will be installed inside the flange and dome. A band heater and temperature controller will maintain the temperature of the flange to above -40C. The lower chamber will then be shortened.

Progress:---

Task 10: As proposed.

The moderator cell used in the first years operation and experiments will be replaced with a larger 1100cc cell which will be within the maximum 1200cc moderator size of the LENS facility. The new design will provide a single 20mm ID fill tube feeding the center top of the moderator with two smaller tubes for diagnostics, gas feed and liquid withdrawal mounted on either side. The larger fill tube will allow for the rapid pellet fill.

Progress: ---

Task 11: Perform trial tests of the unit prior to delivery and installation on LENS.

As proposed.

As in the first year testing of the 600cc apparatus, the 1100cc pelletizer will be tested with the larger size moderator cell at CAFI.

Progress:---

Task 12: Deliver and assist in the installation of the 1100cc pelletizer on the LENS apparatus.

As proposed.

The 1100cc pelletizer will be delivered and installed on the LENS pelletizer test bed . Testing of the larger pelletizer can be done in parallel to the tests on the 600cc pelletizer in the TMR by using the second refrigeration system.

Progress:---

Task 9N: Description and operations procedure for the pelletizer in support of the IU Safety documentation.

Progress:

1. Description of the CAFI Pelletizer

The pellet forming process developed by Cryogenic Applications F, Inc. (US Patent 6,003,332) produces high density, clear cryogenic ice by freezing gas directly to solid by reverse sublimation. The feedstock gaseous material (98% methane + 2% ethane) is introduced to the freezing chamber at a pressure below the triple point. The gas then freezes out onto a refrigerated surface maintained below the equilibrium vapor pressure/temperature of the gas in the chamber, causing the gas to condense directly to solid on the cold surface. The temperature of the cold surface is maintained close to the equilibrium temperature so that the ice forms as a clear dense solid. The rate of ice formation in this process is dominated by the thermal conductivity of the ice being formed, since the heat of condensation of the freezing gas is carried from the surface to the refrigerated cold surface through the ice layer.

Fig. 9.1 shows a schematic drawing of the liquid nitrogen bath methane pelletizer, Fig. 9.2 shows a photograph of the apparatus. The pelletizer produces app. 45,000 3 mm methane pellets per batch by freezing methane gas onto two honeycomb pelletizer cryopanel (see Fig. 9.3) cooled by LN_2 . The pelletizer can be operated by using 1 cryopanel (producing 22,500 pellets) or 2 panels (producing 45,000 pellets). The honeycomb panels are mounted inside a vacuum insulated Dewar, which has observation windows for viewing the honeycomb panels.

Total amount of methane for the pelletizer operation with two cryopanel is app. 450 standard (STP) liters (= 30 STP liters/min*15 min), when both pelletizer cryopanel are filled (see 2.9 and 2.10 in this document). In operation at LENS we will run with only 1 cryopanel in operation, so our total gas charge will be roughly 225 STP liters. We note that this is only about 50% more gas than is used in standard LENS moderator operation and it is less than 1/3 of the safety envelope of 743 STP liters established by the LENS moderator SAD (table 2 of that document, associated with a moderator volume of up to 1100 ml).

The upper dome has the following connections (Fig.9.1):

- 1.1. An access port to the growing chamber located in the center, through which the electrical connections for the heaters and thermocouples pass
- 1.2. Two methane gas feed tubes
- 1.3. Liquid nitrogen feed tubes for the upper and lower honeycomb pelletizer cryopanel
- 1.4. The cold nitrogen exhaust tubes for the two cryopanel
- 1.5. Access ports for the liquid nitrogen level detectors and Si diode thermometers

The lower dome has:

- 1.6. Pellet exhaust port, consisting of a 1½" bellows tube, connecting the lower funnel to the pellet exhaust gate valve and a port for pumping the Dewar jacket. The exhaust gate valve is connected to the moderator cell assembly.

Figure 9.1 Cryogenic Applications 600cc methane pelletizer

Fig. 2: Photograph of the liquid nitrogen bath methane pelletizer apparatus.

Figure 9.2 : 600cc pelletizer as delivered to IU LENS

Fig. 9.3: Photograph of one of the honeycomb pelletizer cryopanel, showing the 3 mm SS honeycomb.

To make pellets of the desired shape and size, the freezing surface is formed into an array of freezing cells similar to an ice cube tray so that the pellets are molded to shape. To prevent the ice in individual cells from growing together, the freezing cell array has non conducting barriers between the cells (see Fig.9. 4a, b). The freezing cells are positioned such that they are facing downward, so that the pellets grow upside down.

The pellets are released from the honeycomb cryopanel by performing the pellet defrost cycle. This is accomplished by raising the pellet cryopanel temperature, causing the ice to sublime (vaporize the ice surface). In practice, this is best accomplished by leaving the cryopanel at LN_2 temperature and pumping on the growing chamber to reduce the pressure. To do this, a fairly large pumping throughput is required. This can be achieved either with a 500 cubic meter per hour roots blower/roughing pump system, which has been used for this before, or with a specialized sub-cooled LN_2 methane cryo-condensation pump (referred to as cryopump throughout this document), which has been constructed exactly for this purpose.

Fig.9.4: Schematic of honeycomb panel: (a) shows an array of the freezing cells, and (b) shows the honeycomb cryopanel.

The cryopump (see Fig.9.5 and 9.6) consists of a vacuum insulated SS chamber which is divided into an upper and lower chamber by an internal header dome. Welded into the header dome and protruding into the upper chamber is an array of 55 1" tubes which are pinched and sealed off at the top end. The inside surfaces of the tube array and the header dome provide cryo-condensation surfaces for the methane gas. The upper chamber is filled with LN_2 through the LN_2 inlet and sub-cooled by pumping on the nitrogen bath through the sub-cool pump port. This port is connected to a rotary vane pumping station (referred to as sub-cool pump throughout this document) for continuous high-throughput pumping (Torr range pressures) so that cold N_2 gas will flow through a heat exchanger coupled to a warm ethylene-glycol circulator. The cryopump is designed to operate in a batch operation.

Fig. 9.5: Schematic of the cryopump.

Fig. 9.6: Photograph of the cryopump.

Liquid nitrogen is supplied to the apparatus from a storage Dewar with manual valve MV2. A Liquid Nitrogen (LN_2) flowchart diagram is shown in Fig. 9.7. The supply line feeds a manifold of three LN_2 service solenoid valves (SV1, SV2, SV3) which feed the two honeycomb cryopanel and the cryopump. A fourth solenoid valve (SV4) connects the inlet manifold to one of the exhaust lines which can be used to discharge the LN_2 from the cryopump. The cryopanel and cryopump each exhaust through a solenoid valve (SV8, SV9, SV10), each having a pressure gauge (PG) ahead of them, and then a check valve (CV1, CV2, CV3) to the LN_2 exhaust. The three LN_2 chambers each have relief valves (PRV1, PRV2, PRV3 set at 20 PSIG) and a provision to inject nitrogen gas from the LN_2 Dewar through a solenoid valve (SV5, SV6, SV7). There are a total of 10 cryogenic service solenoid valves (SV1-10).

Liquid Nitrogen Flowchart

Fig. 9.7: Liquid Nitrogen (LN₂) Flowchart Diagram.

Fig. 9. 8 shows the methane flowchart diagram. The auxiliary pump is a two stage rotary vane pump which is used to pre-evacuate the gas lines, the cryopump and the pelletizer chamber prior to operation. It is also used to pump out the methane from the cryopump and the pelletizer during regeneration. It connects to the cryopump through a pneumatically operated vacuum valve (PV6). The auxiliary pump can also evacuate the pelletizer chamber through the cryopump by opening pneumatic valve PV5 and allowing the cryopump to function as an LN₂ trap.

Fig. 9.8: Methane Gas Flowchart Diagram.

The auxiliary pump vacuum is monitored by Convection gauge (VI1). The pelletizer chamber has a precision mechanical vacuum gauge (VG1) and a 100 Torr Baratron gauge (VT) which can be zeroed with the Convection gauge during the pelletizer chamber evacuation. The Baratron gauge is connected to a process controller (VC) which displays the pressure and will stop the methane gas flow by closing pneumatic valve PV4 if the chamber pressure rises above the set point value. In case of over-pressurization the cryopump, the pelletizer, and the moderator cell are protected from by Viton-sealed Nupro relief valves (RV1, RV2, RV3 set at 1PSIG). These three relief valves and the auxiliary pump exhaust all contain methane and ethane gases and are to be routed to the flammable gas exhaust of TMR2. Leak testing of the methane/ethane gas system can be performed by connecting a leak detector to the Port for leak testing and opening the manual valve (MV5).

The pelletizer and the cryopump are constructed as super-insulated Dewars. A small turbomolecular pumping system is used to evacuate the vacuum jackets. The Dewar vacuum system flowchart diagram is shown in Fig. 9.9. The Dewar vacuum is monitored by Convection gauges (VI2) and (VI3).

The electrical supply for the apparatus is provided through two 208V/30amp single phase four wire cable plugged into the power box. The inlet power is connected to a set of 110V

breaker switches and a small fuse for the initial control power. The power box contains a DC power supply, a set of four power relays which control the large vacuum pumps and the auxiliary power strips, a small relay/transformer board which controls the power relays via the key switch, and a control board which contains a set of solid state relays with snubbers to drive the LN₂ solenoid valves (SV1-10).

The pneumatic gas valves (PV1-5), vacuum valves (PV6-7), and the LN₂ solenoid valves (SV1-10) are all operated by manual switches located on the Process Control Panel shown in Fig. 9.10. The Control Panel also contains readouts for the three Si diode thermometers, the capacitance level detectors and process controllers for the Baratron gauge (VC) and the methane and ethane flow controllers (FC's). Three process controllers monitor the cryopump temperature (Si diode in Kelvin), the upper cryopanel funnel temperature (type T thermocouple in Centigrade), and the lower panel funnel temperature (type T thermocouple in Centigrade). The process controllers also control the heaters which can be used during regeneration. Switches are provided to manually turn off the heaters.

Fig. 9.9: Dewar Vacuum System Flowchart Diagram

Legend of vacuum components:

CH: Circulator/Heater
 CV1-3: Check Valves
 FC: Flow Controller
 LD: LN2 Level Display
 LS: LN2 Level Sensor
 MV1-5: Manual Valves
 PG: Pressure Gauge
 PRV1-3: Pressure Relief Valves(set at 20PSIG)
 PV1-7: Pneumatic Valves
 PV1: Methane valve
 PV2: Ethane valve
 PV3: Gas manifold vacuum valve
 PV4: Gas inlet valve
 PV5: Cryopump valve
 PV6: Auxiliary pump valve
 PV7: Pellet exhaust gate valve
 RV1-3: Nupro Relief Valves (set at 1 PSIG)
 SV1-10: Solenoid Valves
 SV1: LN2 inlet cryopump
 SV2: LN2 inlet upper cryopanel
 SV3: LN2 inlet lower cryopanel
 SV4: LN2 manifold exhaust

SV5: N2 gas inlet cryopump
 SV6: N2 gas inlet upper cryopanel
 SV7: N2 gas inlet lower cryopanel
 SV8: LN2 ex valve cryopump
 SV9: LN2 ex valve upper cryopanel
 SV10: LN2 ex valve lower cryopanel
 TC1: Temperature Controller (TC 230K)
 for cryopump
 TC2: Temperature Controller (TC -40K)
 for upper cryopanel (funnel)
 TC3: Temperature Controller (TC -40K)
 for lower cryopanel (funnel)
 TD: Temperature Display
 TS: Temperature Sensor
 VC: Vacuum Controller
 VG1: Vacuum Gauge (precision mechanical),
 absolute 0 – 800 mmHg
 VG2-3: Vacuum Gauges (mechanical),
 0 to -30 inHg
 VI1-3: Vacuum Indicators (Convectron
 Gauges)
 VT: 100 Torr Baratron Gauge

Fig.9.10: Process Control Panel with labels for the various control functions and displays.

2 Procedure for making a batch of methane pellets:

- 2.1. The Control Panel power switch is turned on, the control power button is turned on, and the circulator heater is turned on at the power strip.
- 2.2. The Dewar vacuum system is activated by turning on the backing pump on the power strip, waiting for the pressure to drop below 1.0 Torr and then activating the turbopump with the switch on the turbo controller. Note that the time required can be fairly long due to the large area of superinsulation. The Convectron gauge on the backing pump (VI3) should be below 50 mTorr and the turbo Convectron gauge (VI2) should zero out.
- 2.3. The auxiliary pump is turned on at the Process Control Panel.
- 2.4. The methane/ethane gas manifold is pre-evacuated by opening the gas manifold vacuum valve (PV3), ethane valve (PV2) and methane valve (PV1) on the Control Panel. When installing new cylinders, the lines should be evacuated to the cylinder with the regulators open, before cracking the cylinder valves to avoid air contamination.
- 2.5. The cryopump and pelletizer chamber are evacuated with the auxiliary pump system by opening the cryopump/pelletizer valve (PV5) and then the auxiliary pump valve (PV6); both switches are on the Control Panel. The system can be flushed with a short injection of methane/ethane gas by momentarily opening the gas inlet valve (PV4) on the Control Panel and re-evacuating. The methane and ethane process control meters should indicate the correct flow during the pulse of gas.
- 2.6. The cryopump and pelletizer cryopanel are then filled with LN₂. Assuming the LN₂ Dewar is installed, the manual valve (MV2) at the Dewar is opened, the three LN₂ inlet valves (SV1, SV2, SV3) are opened, the three LN₂ exhaust valves (SV8, SV9, SV10) are opened, the three N₂ gas valves (SV5, SV6, SV7) are closed, and the autofill switches are off.
- 2.7. **In case only 1 cryopanel (e.g. the lower honeycomb cryopanel) to be filled**, only two LN₂ inlet valves (SV1, SV3) and two LN₂ exhaust valves (SV8, SV10) are opened, the other valves are closed (SV2, SV5, SV6, SV7), and the autofill switches are off.
- 2.8. LN₂ is allowed to flow until liquid is observed coming out of the exhaust lines (at the three check valves (CV1, CV2, CV3)). As this occurs, the LN₂ inlet valves (SV1, SV2, SV3) can each be closed. Following the initial fill, the cryopanel is allowed to chill for ~5 minutes, and then the honeycomb cryopanel and the cryopump are topped off. At this point the temperature gauges should be near 77K and stable and the LN₂ level gauges should indicate full.
- 2.9. The pelletizer chamber/cryopump Vacuum Indicator (VI1), i.e. a Convectron Gauge, should read below 20 microns (1 micron = 1 mTorr) and the Baratron gauge (VT) should read zero (if not, it can be manually zeroed). The auxiliary pump valve (PV6) and the cryopump/pelletizer valve (PV5) are then closed.
- 2.10. The methane gas is then injected into the chamber through mass flow controllers at a rate of 30 SLM (Standard Liter per Minute) of methane and 0.3 SLM of ethane by closing the gas manifold vacuum valve (PV3) and opening the methane valve (PV1), ethane valve (PV2) and gas inlet valve (PV4). The addition of the 1% ethane impurity into the methane ice has been found to reduce the stickiness of the pellets.

- 2.11. The flow is administered for 15 minutes (the total dose determines the size of the pellets) and will fill both honeycomb cryopanel. With a flow rate of 30 SLM and a duration of 15 minutes, the total amount of methane in the apparatus is app. 450 liters at atmospheric pressure, when both pelletizer cryopanel are filled.
- 2.12. During this time the pressure in the growing chamber will rise, beginning at about 13 Torr (the equilibrium pressure at 77K) and rising to about 23 Torr as the pellet ice thickness increases. The pressure rise is proportional to the flow rate, which increases the condensation rate and heat load on the surface of the ice. The heat load produces a temperature differential across the ice thickness, increasing the surface vapor pressure. After the desired growing time, the gas is turned off (closing PV1, PV2, PV4) and the ice is allowed to cool, lowering the chamber pressure back to ~13 Torr at the 77K equilibrium vapor pressure.

3 Pellet release:

- 3.1. Midway through the pellet growing cycle, the cryopump temperature is lowered by starting the sub-cool pump (putting the ON/OFF switch of the sub-cool pump to On on the Process Control Panel) with the LN₂ exhaust valve (SV8) left open. This will reduce the LN₂ chamber pressure and temperature. The cryopump LN₂ bath pressure should drop below ½ atmosphere before starting the defrost cycle.
- 3.1. At the start of the pellet defrost cycle the cryopump/pelletizer valve (PV5) is opened, allowing the methane to transfer from the honeycomb cryopanel to the sub-cooled cryopump. The chamber pressure will drop rapidly to about 5 Torr and continue to drop to about 1 Torr.
- 3.1. Soon after the defrost cycle starts, pellets will start to release from the honeycomb and drop through the funnels. The pelletizer/moderator gate valve (PV7) should be opened (with the switch on the Control Panel) at the start of the release cycle to prevent the pellets from plugging up above the gate valve.
- 3.1. The pellet release cycle takes about 20 minutes.

4 Pelletizer and cryopump regeneration:

After the release cycle is completed and the Pellet exhaust valve (gate valve PV7) is closed, the pelletizer cryopanel and the cryopump can be regenerated, using the following procedure:

- 4.1. The sub-cool pump is turned off.
- 4.1. The auxiliary pump should be on with the auxiliary pump valve (PV6) and the cryopump/pelletizer valve (PV5) open.
- 4.1. The pelletizer can then be warmed up by turning on the funnel calrod heaters (switch on the heater Control Panel), which will accelerate the evaporation of the excess LN₂ remaining in the pelletizer cryopanel.

- 4.1. The cryopump can then be regenerated by dumping the remaining LN₂. This is accomplished by turning off the liquid nitrogen supply from the LN₂ manifold by closing the manual valve (MV2) at the LN₂ supply Dewar, closing the cryopump LN₂ exhaust valve (SV8), turning off the sub-cool pump, opening the cryopump LN₂ fill valve (SV1), opening the LN₂ manifold vent valve (SV4), opening the warm N₂ gas inlet valve (SV5) to the cryopump (on the Control Panel), and turning on the cryopump cartridge heaters (on the heater Control Panel). Note that the heater controllers are programmed to put out a short duty cycle pulse of power, so the switch light will blink when on. This will cause the pressure to rise in the LN₂ chamber and force the LN₂ out through the inlet line and into the LN₂ exhaust line.
- 4.1. When the cryopump starts to warm up, the frozen methane will sublime/evaporate and be exhausted through the auxiliary pump. The system is regenerated when the temperature of the cryopump and pelletizer are above 120K and the chamber pressure is below 20 mTorr.
- 4.1. Now, the apparatus is ready for making another batch of methane pellets.

2. Safety Analysis and Mitigation:

Operation of the pelletizer will be carried out within the safety envelope established for the operation of the LENS moderator. The total charge of flammable gas is kept well under the class-0 limits established in the moderator SAD, and the amount of condensed gas will also be kept less than 50% of the limits established in that document. For operation outside the vault, a suitable enclosure of cement blocks will be constructed for the moderator and pelletizer to sit within during operation (since the moderator will not be within the TMR as specified in the moderator SAD).

The total amount of methane/ethane for the pelletizer operation with two cryopanel would be approximately 450 standard (STP) liters (= 30 STP liters/min*15 min; see 2.9 and 2.10 in this document). In operation at LENS we will run with only 1 cryopanel in operation, so our total gas charge will be roughly 225 STP liters. We note that this is only about 50% more gas than is used in standard LENS moderator operation and it less than 1/3 of the safety envelope of 743 STP liters established by the LENS moderator SAD (table 2 of that document, associated with a moderator volume of up to 1100 ml). We will fill a moderator volume of approximately 380 cm³ (again only 35% of the upper volume limit specified in the moderator SAD).

We note that in this case, since the methane is being condensed directly into the solid state in within a vessel of substantial volume (130 l), the possibility that the fill line might be plugged by freezing methane liquid in the line is eliminated in pelletizer operation. Below we identify the most likely accident scenarios associated with pelletizer operation, and explain how none of these pose a substantial safety risk to personnel.

2.1. Loss of electrical power

In the first few seconds (i.e. during initial condensation of methane):

All of the solenoid valves close. The pumps turn off. The pneumatic gas valves close (except for PV7 which opens, assuring that the pelletizer and moderator vessel are in communication). The vacuum valves close.

After a few minutes:

The pressure in the pelletizer chamber will drop to ~13 Torr, since the cryopanel are filled with LN₂ and the gas feed has been stopped. The pressure in the pelletizer LN₂ chambers and the cryopump LN₂ chamber will start to rise in response to the closing of the LN₂ exhaust valves (SV8, SV9, SV10). When the pressures reach 20 PSIG the relief valves (PRV1, PRV2, PRV3) will periodically open to vent the N₂. The rise in LN₂ temperature will be to about 80K which will cause the methane pellet vapor pressure to increase to about 16 Torr.

In the next few hours:

The LN₂ will evaporate, causing the temperatures slowly rise. When the temperature gets to the triple point, the frozen methane will drip out of the honeycomb and come in contact with the inner vessel; this will cause a further increase in pressure. When the pressure reaches 1 PSIG, the relief valve (RV1) will open, allowing the methane to be released into the flammable gas exhaust system of TMR2.

In case the moderator cell contains methane pellets, these will warm up and the pressure in the cell will rise, since the Pellet exhaust valve (gate valve PV7) opens when electrical power is removed; should the pressure in the combined volume (moderator plus pelletizer) reach 1 PSIG, the relief valves (RV1 and RV3) will open and allowing the methane to be released into the flammable gas exhaust system of TMR2.

The risks associated with this hazard are very low and **no additional mitigation** is needed.

2.2. Loss of Dewar vacuum

Small leaks:

Loss of Dewar vacuum would have no major effect. The experiment should be shut down in the normal way and the problem be resolved.

Massive leak:

The processes (in chapters 2.-4.) might be affected but not to a large degree. The experiment should be shut down in the normal way and the problem be resolved.

The risks associated with this hazard are very low since all volumes with condensed gas are protected by relief valves, and the total amount of condensed flammable gas in the moderator and pelletizer would produce a pressure of only about 10 psig even if the relief valves were to fail to open.

The mitigation in both scenarios would be the same: turn off the gas flow, open PV6 and evacuate any gas build up through the flammable exhaust system.

2.3. Development of an air leak into one of the pelletizer chambers, cryopump, or methane/ethane gas handling system

Small leak:

Small leaks will result in a rise in the chamber pressure, since the air would not be cryopumped by the LN₂ cooled honeycomb cryopanel. The pressure would exceed the set point process controller (VC) causing the gas to be shut off.

The risks associated with this hazard occurring are low, since before every experiment the vacuum integrity of the system will be confirmed with a leak check.

The mitigation: a sensor for combustible gas detection (sensor 3) is mounted on the pelletizer apparatus and via a cable connected to the existing gas detection monitor (Beacon 410, RKI Instruments). This detection unit has 4 input channels, two of which are already connected to combustible gas sensors (one inside of the TMR2 vault and the other outside above the cryogenics setup), and will make an audible alarm, when methane is detected. Sensor 3 will be tied in this already existing safety system. When an alarm is activated, the gas flow for the pelletizer system will be turned off by closing PV1 and PV2, and the pelletizer chamber/cryopump need to be evacuated through the auxiliary pump by opening PV5 and PV6.

Massive leak:

Massive leaks of air would cause methane ice to melt, evaporate and possibly exhaust into the room through the same leak. Should such an event occur, the flammable gas sensor would sound an alarm.

The probability of a such a massive leak occurring is extremely low, and the risks are small due to the small charge of methane being used in these tests. The most likely scenario leading to this would be a window on the pelletizer breaking due to human action.

The mitigation: Again, sensor 3 (mounted on the pelletizer apparatus) will immediately set off an alarm, any gas flow into the pelletizer system will need to be turned off by closing PV1 and PV2 and the area evacuated.

2.4. Methane is detected outside of the pelletizer

The most likely causes would be due to leaks in the gas supply cylinder, the gas handling system, or a leak in the flammable gas exhaust system. However, leaking of methane would most likely not be from the pelletizer, since it is well below atmospheric pressure.

The risks of occurrence of this hazard are low.

The mitigation is the same as in 5.3 above. The flammable gas detector (activated by sensor 3) will give an audible alarm and PV1 and PV2 need to be shut off, and PV5 and PV6 need to be opened. The gas cylinder valves would be shut and the area would be evacuated until the alarm ceased.

2.5. Development of a leak in the LN₂ gas handling system

This would not affect the system, since pressure relief valves (PRV1, PRV2, PRV3 set at 20 PSIG) are located downstream of the cryopump and of the pelletizer and check valves (CV1, CV2, CV3) even further downstream.

The risks associated with this hazard are very low.

The mitigation would be to shut off the LN₂ manual valve (MV2) at the Dewar.

2.6. Operator error: opening of a wrong valve

Under the number of anticipated scenarios, the major concern would be, turning the gas feed valves PV1, PV2, and PV4 on and leaving them on; then more methane/ethane would accumulate than normal. However, the filling would automatically stop at 30 Torr, since the pressure would exceed the set point process controller (VC). If this did not function, the LN₂ would run out eventually causing a melting and release of the methane through relief valve RV1 into the flammable exhaust system.

The risks associated with this hazard are low.

The mitigation would be to shut off PV1 and PV2.

Task 10N Design and construction of a computer controlled operating system for the 600cc pelletizer.

Progress:

Computer Control Remote Operating System

The methane pelletizer, as delivered to IU LENS facility and as used in the experiments, had a manually operated control system which required the operator to be present during the production of pellets and the filling of the moderator cell.

While this worked for the experimental testing of the device on LENS a remotely controlled system would be desirable for routine operation of the system. To this end a computer control system was designed and constructed to replace the manual control panel. A programmable logic controller(PLC) with digital and analog output modules formed the core of the new system. The PLC chosen for the task was Siemens S7- 1214C controller which has 10 relay outputs and 12 dc inputs combined with a 16 channel digital output module, an 8 channel analog input module, and a 4channel analog input module with 2 analog outputs. The PLC was combined with a 16 channel Opto22 output rack with 12 fused 120VAC output modules, and four 110VAC 30 amp solid state relays. The PLC has an ethernet connection which can be routed to a PC for programing and a 6"color Touch Panel interface for remotely operating and monitoring the pelletizer. The PLC is mounted in a 20"X20"X8" electronic enclosure.

In addition to the PLC the enclosure has:

Four DC power supplies , supplying +24V, +5V, +15V, -15V.

A receptacle on the bottom left for the main 220VAC 30A input power.

Four 110VAC 15A power output receptacles, also on the bottom.

Four 15A magnetic breaker switches to protect each of the output receptacles on the left side.

Two breaker switches to protect the PLC, the DC power supplies and the Opto 22 output rack.

A set of strain relief feedthroughs for the input and output cabling on the bottom right.

The PLC enclosure is shown in Fig10.1;The output cables are shown in Figure10.2;

The remote touchscreen panel is shown in figure 10.3;Fig 10.4 is a table of all of the PLC input/output connections, the programming tag names and the cabling connections to the outputs.

The pelletizer has three mechanical/vacuum gauges for monitoring the pelletizer pressure. The cryopump pressure and the cryopump LN2 bath pressure. To implement the PLC monitoring and control system three absolute capacitance manometer pressure transducers were installed which are connected to the analog input module of the PLC.

In addition to the Control box, a new liquid nitrogen level detector system was designed for the two pelletizer cryopanels. The liquid level is monitored by an array of surface mount PIN silicon diodes used as cryogenic thermometers. When the diodes are above the liquid level their temperature rises due to the heat leak down sensor wires. The rise in temperature is detected by open collector differential comparators. A constant current source passes through a series of resistors between which the comparators short the resistors to ground causing a linear voltage rise and fall as the LN2 level goes up and down. One of the diodes also acts as a thermometer with a current source and amplifiers converting the diode voltage to temperature.

The sensor and electronic box are shown in figure 10.5.

Figure 10.1 PLC Control Box

Figure 10.2: PLC Box Output Control Cables

Figure 10.3:PLC Remote Ethernet Operator Panel

PLC Siemens Simatic S7-1200								
Channel #	Name	tag name	Wire bundle tape color	wire color	goes to	goes to	wire color twisted pair	goes to
SM 1214C relay outputs								
DQa 0.0	Methane Gas feed	Methane_Vlv	yellow	red	cable yellow 1	Pneu vlv Manifold		Gran Phil Vlv
DQa 0.1	Ethane Gas feed	Ethane_Vlv	yellow	black	cable yellow 1	Pneu vlv Manifold		Gran Phil Vlv
DQa 0.2	Vacuum purge gas manifold	Vac_purge_Vlv	yellow	orange	cable yellow 1	Pneu vlv Manifold		Gran Phil Vlv
DQa 0.3	Gas In pellet chamber	Gas/PelCh_Vlv	yellow	green	cable yellow 1	Pneu vlv Manifold		Gran Phil Vlv
DQa 0.4	Pellet Chamber to Cryopump Valve	CPu/PelCh_Vlv	Blue	red	cable blue 2	Pneu vlv Manifold		Angle Vlv on auxiliary pump
DQb 0.5	Cryo Pump to Auxiliary pump Valve	AuxPu/CPu_Vlv	Blue	black	cable blue 2	Pneu vlv Manifold		Angle Valve Cryopump inlet
DQb 0.6	Pellet Chamber to moderator Valve	Mod/PelCh_Vlv	Blue	orange	cable blue 2	Pneu vlv Manifold		Gate Valve under pelletizer
DQb 0.7	Spare		Blue	green	cable blue 2	Pneu vlv Manifold		
DQb 1.0	Cry Pump Ln2 Heater	CPu_heater	white	red&green	cable white			
DQb 1.1	Spare							
SM 1214C Digital inputs								
Dla 0.0 to Dla 0	spare							
Dlb 1.0	Aux pump convection relay	Aux_Pu_OK	orange	red	orange cable	Granville phillipsconvection controller		
Dlb 1.1	Turbo backing Pump Convection relay	Turbo_Bak_OK	orange	white	orange cable	Granville phillipsconvection controller		
Dlb 1.2	Turbo convection relay	Turbo_OK	orange	green	orange cable	Granville phillipsconvection controller		
Dlb 1.3								
Dlb 1.4								
SM 1222C Digital inputs								
DQ 8.0	Upper panel Nitrogen gas inlet valve	UpPan_N2_gas	yellow	Brown	SSR#8	Sol valve Blk/W		above pellet chamber
DQ8.1	Lower panel Nitrogen gas inlet valve	LoPan_N2_gas	yellow	gray	SSR#9	Sol valve Br/W		above pellet chamber
DQ8.2	Spare		yellow	orange	SSR#10			
DQ 8.3	Spare		yellow	purple	SSR#11			
DQ 8.4	Sub Cool Pump	SbClPu_Pwr	Brown	Brown	Crouzet SSR #1	Outlet 1		pump
DQ 8.5	Auxiliary Vacuum Pump	Aux_Pu_Pwr	Brown	gray	Crouzet SSR #3	Outlet 3		Pump
DQ 8.6	Dewar vacuum Pump System	Dew_Vac_Pwr	Brown	orange	Crouzet SSR #2	Outlet 2		Term Strip#1
DQ 8.7	Circulator/heater pump	Cir_Pu_Pwr	Brown	purple	Crouzet SSR #4	Outlet 4		Term strip#2
DQ 9.0	Upper Panel liquid nitrogen feed	UpPan_LN2_In	Blue	Brown	SSR#0	Sol Valve Blk/W		LN2 manifold
DQ 9.1	Lower Panel liquid nitrogen feed	LoPan_LN2_In	Blue	gray	SSR#1	Sol Valve Brn/W		LN2 manifold
DQ 9.2	Cryo pump liquid nitrogen feed	CPu_LN2_In	Blue	orange	SSR#2	Sol Valve Red/W		LN2 manifold
DQ 9.3	Liquid nitrogen dump	LN2 dump	Blue	purple	SSR#3	Sol Valve Or/W		LN2 manifold
DQ 9.4	Upper Panel liquid nitrogen Exhaust	UpPan_LN2_Ex	red	Brown	SSR#4	Sol Valve Yel/W		above pellet chamber
DQ 9.5	Lower Panel liquid nitrogen Exhaust	LoPan_LN2_Ex	red	gray	SSR#5	Sol Valve Grn/W		above pellet chamber
DQ 9.6	Cryo Pump liquid nitrogen Exhaust	CPu_LN2_Ex	red	orange	SSR#6	Sol Valve Blu/W		Cryo pump
DQ 9.7	Cryo Pump Nitrogen gas Inlet valve	CPu_N2_gas	red	purple	SSR#7	Sol Valve Pur/W		Cryo pump
SM1231 Analog Inputs								
Channel #	Name	tag name	Wire bundle tape color	wire color	goes to	goes to	wire color twisted pair	goes to
AI 0	112 upper panel LN2 temperature	UpPan_Kel		white/b 0	red 0 1 cable	Upper panel circuit box		
AI 1	114 Upper panel LN2 Level	UpPan_Lev		green/b 1	red 0 1 cable	"		
AI 2	116 Lower panel LN2 temperature	LoPan_Kel		white/b 2	green/yel 2 3 cable	Lower Panel circuit box		
AI 3	118 Lower panel LN2 Level	LoPan_Lev		green/b 3	green/yel 2 3 cable	"		
AI 4	120 Cryopump LN2 temperature	CPu_Kel		white/b 4	green 4 5 cable	Cryo pump circuit box		
AI 5	122 Cryopump LN2 Level	CPu_Lev		green/b 5	green 4 5 cable	"		
AI 6	124 Cryopump LN2 Pressure	CPu_LN2_Pres 760 Torr		clear/w 6	blue 6 cable	WIKA transducer cryo LN2 chamber		
AI 7	126 Cryopump Chamber Pressure	CPu_Pres 760 Torr		clear/w 7	yellow 7 cable	WIKA transducer cryo chamber		
SM1234 Analog Inputs/outputs								
AI 0	128 Methane Flow controller	Me_Flo_SLM		blu/br 0	red cable	Methane flow Controller		
AI 1	130 Ethane Flow Controller	Et_Flo_SCCM		blu/br 1	green/yel cable	Ethane Flow controller		
AI 2	132 Pellet chamber Pressure 100Torr	PelCh_Pres_100		white/blk 2	green/yel 2 cable	Baratron gauge Pellet Chamber		
AI 3	134 Pellet chamber Pressure 760 Torr	Pel Ch_Pres_760		clear/wht 3	green cable	WIKA transducer Pellet chamber		
AQ 0	136 Methane Flow controller Cont Voltage	Me_Flo_Con_ConVlt		red/blk 0	red cable	Methane Flow Controller		
AQ 1	138 Ethane Flow Controller Cont voltage	Et_Flo_Con_ConVlt		red/blk 1	green/yel cable	Ethane Flow Controller		

Figure 10.4 PLC Input and Output Connections

Figure 10.5: Pin Diode LN2 Level Detector

LN2 Level/temp Signal Amplifiers

LN2 Level/Temp Sensor
array of 12 PIN Diodes

Overview

The report summarizes the major accomplishments and findings from the activities performed at Indiana University under the STTR contract with Cryogenic Applications F Inc. (CAF): Application No. 86232T08-II, “Development and Neutronic Validation of Pelletized Cold and Very Cold Moderators for Pulsed Neutron Sources”. The primary goals of the project were to demonstrate the ability to produce methane pellets using a device constructed by CAF and then transfer those pellets into a moderator cell at the Low Energy Neutron Source (LENS) so that neutronics measurements could be performed on the resulting “pelletized” moderator. To the best of our knowledge, only one other attempt has been made to characterize the neutronic performance of a granular moderator, that being a study of a polyethylene pellet moderator by the Kiyanagi group in 1999 [1]. We compare the results obtained from the neutronics measurements on the pelletized moderator to the standard LENS moderator as a benchmark. However, we did not attempt to optimize the geometry or fill pattern of the pelletized moderator in this study, so relative comparisons between the LENS and pelletized moderators should be taken as suggestive. They should not be used as the basis for definitive conclusions regarding the relative performance of pelletized and monolithic methane moderators.

The LENS facility is a pulsed neutron source based on the (p,n)Be reaction with a 13MeV proton beam with a peak current of roughly 20 mA and pulse lengths of from 14 to 600 μ sec [1,2]. The facility was constructed to be suitable for research on neutron moderator by making exchange of different cryogenic moderators a relatively straightforward process. The moderators may be exchanged using an overhead crane after that same crane has been used to remove the shielding that sits above the moderator vacuum vessel. This process can begin as little as a few days after the facility has been running at full power, or after 12-24 hours after the facility has been running at the power levels typically needed for collecting spectral and emission time distribution data from a moderator of interest. LENS normally operates with a solid methane moderator held at roughly 6K to provide the maximum possible flux at wavelengths longer than 1.0nm since the instruments fed by the LENS source are primarily interested in the study of large-scale structures in materials for which longer wavelengths carry certain advantages. For the purposes of this project we take the LENS moderator as the standard against which we will compare the neutronic performance of the pelletized moderator. Neutron spectra were collected using standard ^3He -based detectors on the SANS beamline at LENS, with the neutron time-of-flight providing the neutron’s velocity (and therefore energy). We also mounted a time-focused spectrometer (based on a large Ge single crystal with roughly a 0.35 deg mosaic spread) at the sample position of the SANS instrument to provide information on the neutron pulse shapes that would arise from such a pelletized moderator.

The heart of the LENS facility is the Target Moderator Reflector assembly (TMR), which is shown schematically in figure 1. The moderator is held within a vacuum can and

cooled by a closed-cycle helium refrigerator to which it is connected by a high-purity aluminum bar. Combining this refrigerator with heaters attached to the moderator vessel, transfer lines and various support structures, we are able to control the temperature of the moderator over a range from 6 K to 300 K.

Fig. 1 shows the interior of the LENS TMR. The proton beam enters from the left and hits the target a few cm from the moderator which is held in a vacuum can in the center of the water reflector. The moderator is connected to the helium refrigerator (recognizable by the two parallel cylinders its top). The yellow “caramel corn” blocks provide the primary shielding of the source.

Fig. 2 gives an overview of the LENS instrument hall, showing the TMR (yellow circle on the left), main poured-concrete shielding wall (turquoise arc) along with grey concrete wall blocks that can be moved with an overhead crane, and the three instruments. All the measurements reported here were collected on the SANS beamline (on the bottom right).

Figure 2 shows an overview of the LENS facility, and figure 3 shows how the pelletizer was mounted on top of the TMR in order to feed pellets into the moderator vessel. Before running the pelletizer within the TMR vault for these neutronics tests, several tests were performed in which the pelletizer was connected to the moderator system outside the vault to assure that an adequate transfer of pellets between the two devices could be achieved. Figure 3 shows the pelletizer connected to the moderator system outside the vault for these tests in one picture and the support of the pelletizer over the TMR in the other.

Fig. 3 The pelletizer connected to the moderator test assembly at LENS. On the left, student Zach Hunt is seen next to the assembly in which the pelletizer (equipment above Zach's head) is supported above a cart holding the moderator assembly. On the right, Chris Foster is shown preparing the pelletizer for its initial growth of methane within the TMR vault. In the vault, the moderator is located in the center of the TMR (the beige block structure with the red bands encircling it behind the platform on which Chris is standing).

Personnel

Activities performed at IU under this contract were overseen by Prof. David Baxter, and included completing a Safety Assessment document for the operation of the pelletizer within the Center for the Exploration of Energy and Matter (CEEM) facility (including operation next to the LENS neutron production target), construction of a suitable moderator cell and pellet transfer system, and a monitoring system to track the fill level of the pellets in the moderator during the filling process. The neutronics measurements

and monitoring system construction were performed with one graduate student (Tracy Steinbach) and three undergraduate students (Ben Nicholson, Michael Schevitz, and Zach Hunt). Therefore the research described herein was part of the education of four physics students. In addition to these academic personnel, several members of the CEEM support staff were employed in various aspects of the project (including writing the safety assessment, designing and constructing the support structure to hold the pelletizer over the moderator system, and machining various parts of the pellet transfer system). The key CEEM staff members involved were Dr. Helmut Kaiser (safety documentation), Dr. Tom Rinckel (accelerator operations, logistics), Jack Duskow (mechanical design and construction), and Tom Todd.

Major Activities

Pellet transfer system and moderator cell

The standard LENS moderator design consists of a 1cm-thick solid methane rectangle held at a temperature of roughly 6K and surrounded by a 50 cm diameter water tank. This design was chosen on the basis of extensive MCNP simulation using the best methane kernel that was available at the time that the facility was under construction (suitable for 22K). Since that time, we have developed our own kernel for methane at 6K, and with this kernel we found that the optimal thickness was closer to 2 cm than 1 cm for methane in its low-temperature phase (phase II, below 20.5K). To account for this, and to facilitate the transfer of pellets, we chose a cell of roughly 3 cm in thickness with a pellet fill tube entering from at the geometric center of the top. Initial tests with polyethylene pellets suggested that our original 0.65" diameter transfer line was too narrow to avoid jamming of the pellets in the first 90 deg. bend of the transfer line, so in the final design we used a transfer line with an inside diameter of roughly 0.75" (see fig.4). A second, smaller diameter, line comes into the top of the cell to accommodate the cables needed for the microwave resonance system used to monitor the fill level of the pellets. This line also allowed an alternative exit for gas should the main fill line become plugged during a fill. Figure 4 shows the cell and its connections to the pelletizer and monitoring systems, along with the location of the thermometers and heaters used to control the temperature profile of the fill line during pellet loading. Thermometers were also located on the base of the moderator cell itself as well as on the cold finger used to cool the moderator.

A mixture of methane and a small amount of ethane gas is condensed into a honeycomb structure in the pelletizer (referred to as a cryopanel in the following) to produce the hexagonal pellets. The pellets are subsequently released from the cryopanel by reducing the pressure in the pelletizer (thereby causing the pellets to sublime slightly and release from the honeycomb). Gravity then feeds the pellets into the pellet transfer line via a series of funnels in the pelletizer. An electro-mechanical vibrator was attached to the fill line and the moderator can to promote pellet flow and redistribution within the moderator cell (to minimize the tendency of the pellets to jam in the lines or pile up in the middle of the cell). The temperature of the transfer line is maintained above 90K at all monitoring points (see fig. 4B) in order to assure that pellets do not stick to the walls.

Fig. 4 The cell used to hold the pelletized moderator is shown on the left (A). The pellets enter into the cell through the central flange. On the right (B) we show how the cell is connected to the outside world (including the bottom of the pelletizer (the gate valve at the top of the figure). A mechanical vibrator was mounted on the pellet transfer line just above the moderator vessel (thereby agitating both the transfer line and the cell itself during the fill process), but it is not shown in this figure.

Microwave Pellet Monitoring System

The apparatus built for monitoring the fill of methane pellets inside the moderator consisted of two antennae with coaxial inputs, a Voltage Controlled Oscillator, a Power detector, and a NI-DAQ unit in conjunction with a National Instruments Virtual Instrument (VI) written in the LabView programming environment. The antennae were mounted inside of the vacuum canister with lacing tape to secure them to an aluminum support structure. The transmitting antenna swept over a range of frequencies, received by the receiving antenna. The signal received will be enhanced at resonant frequencies for the cavity defined by the metal walls of the moderator cell. The key to this system is that the resonant frequency will shift as the cell is filled with a material with a dielectric constant greater than 1 (methane has a dielectric constant of roughly 1.7). The signal from the receiving antenna is monitored by the VI as a function of transmission frequency. Each scan over a frequency range from 1.5 to 1.85 GHz takes approximately 10 seconds, so this system provides a real-time monitor of the fill level in the moderator.

Figure 5 provides a schematic outline of the microwave resonance circuit used to probe the methane pellet fill level by looking for a shift in the resonant frequency of the cavity defined by the moderator cell due to the dielectric properties of the pellets.

During the fill, pellets can be observed through a viewport as they fall from the cryopanel into the funnels that guide them into the pellet transfer line on their way to the moderator itself. As pellets start to enter the moderator, the resonant frequency also shifts (confirming that the pellets observed being released from the cryopanel are, in fact, making it into the moderator). Figure 6 shows the frequency shift in a 3-D plot for two different methane fill procedures, and these measurements clearly indicate that the two procedures resulted in moderators filled with a very different amount of methane. Below we will show that this is confirmed by the neutronic measurements on these two moderators. Our attempts to provide an absolute calibration of the frequency shift to allow the data in figure 6 to be interpreted directly in terms of a moderator fill level were not successful for reasons that are not presently clear to us. Nevertheless, the system proved invaluable in confirming the transfer of pellets into the cell (in particular, in a few cases the pellets formed a plug somewhere in the system and in these cases the resonant frequency did not shift despite the observed “dropping” of pellets past the viewport on the pelletizer).

A

B

Figure 6. shows a 3-D plot (color corresponds to deviation of the signal strength from a moving average in order to enhance the visibility of the resonance). The cycle number refer to evolving time. Clearly, the two fill procedures produced different frequency shifts (and therefore correspond to different fill levels in the moderator). The typical fill process takes roughly 15 minutes. The microwave data shown in figures A and B were collected during fills B and C respectively (see figure 7 below in the discussion of the moderator spectral performance).

Safety Assessment

A safety assessment of the pelletizer's design and operation was conducted prior to the production of pellets at IU. This evaluation was compiled in LENS document# L000353 "Safety Assessment – CAF Pelletizer" [4], and consisted of a review of the pelletizer design and operations in the context of the LENS facility. This included consideration of various accident scenarios, an evaluation of the engineering design and the controls incorporated into that design to mitigate against possible accidents, and the identification of operations procedures to provide additional mitigations. Examples of the procedural controls imposed on the operation included establishing a limit on the total flammable gas inventory (less than 2200 STP liters of methane gas) as well as on the gas condensed into the moderator cell (equivalent to no more than 225 STP liters of methane), restrictions on the operation of a vacuum gauge during pelletizer operation, and restricting access to certain areas around the pelletizer during operation.

Neutronic Characterization and Measurements

We report results from three different attempts to load pellets into the moderator while it was in position within the LENS target/reflector/moderator assembly (TMR) so that neutronic measurements could be performed. The figure below shows the spectra collected from these three fills at a moderator temperature of 30K. In one case (B), the mass flow controller feeding methane into the pelletizer failed during the pellet growth cycle, and consequently very little methane was actually transferred to the moderator cell. The spectrum shown for this case then represents the spectrum provided by the water reflector that surrounds the moderator system within the LENS TMR. As expected, in this case the spectrum is dominated by neutrons above 10meV in energy, and the cold spectrum (below 10meV) is very weak, and this portion of the spectrum may therefore provide information about the fill level of the moderator. We notice that fill A, for which we believe we transferred the greatest number of pellets into the moderator, shows the smallest spectral intensity near 50 meV, the lowest 1eV coupling, and the largest (by a small factor over fill C) intensity at 5 meV.

Fig. 7 displays neutron spectra from three different fills of the moderator cell with methane pellets and the cell held at roughly 40 K on a linear scale to emphasize the differences among various fills. During “Fill B” there was a failure of the mass flow controller that fed methane into the pelletizer, and consequently very little methane was transferred (and hence the spectrum is substantially warmer than for the other two). Fills A and C both succeeded in transferring a substantial amount of methane into the cell, but the fill total was greater for A than for C, and this is reflected in the spectra.

For fills A and B, we were able to measure the amount of gas recovered from the cell after the neutronic measurements. In the case of fill A, roughly 8.0 moles of methane were recovered, which corresponds to an amount of methane that is about 25% more than would be needed to fill the moderator (assuming a random packing efficiency of 0.63).

We therefore assume that in this case, some pellets were also situated in the fill line above the moderator, but that the beam line viewed a full moderator. In contrast to this, for fill B only about 1.4 moles of methane were recovered (this case involved a mass flow controller failure during the fill, resulting in far less than the requested amount of methane condensing on the cryopanel of the pelletizer). This moderator was, therefore, no more than 22% full. Due to an operator error, we were unable to quantify the amount of recovered gas from fill C, but the neutron spectrum from that moderator suggests that it was not completely full, although certainly it was substantially more full than was the case for fill B. Fills B and C are the ones for which microwave data are displayed in figure 6.

In figure 8 we compare the spectra from the pelletized moderator with those from the standard LENS moderator which is based on a 1-cm-thick slab of solid methane. LENS normally operates at a temperature of 6K, but we were unable to cool the vessel containing the pellets to below 30 K during our tests, so we collected a few spectra from the standard LENS moderator at non-standard temperatures as well. Since the above discussion suggests that fill A had the most complete fill of pellets, we make the comparison to the LENS moderator using fill A. We conclude that the spectral intensity and 1-eV coupling seen for this fill of the pelletized moderator are both slightly below that of the standard LENS moderator (despite our attempts to make the effective thickness of the pelletized moderator greater than the 1cm standard LENS moderator). This may reflect some loss of moderator volume to the antennae used for the microwave measurement of the moderator filling, but it may also be influenced by some of the extra polyethylene pieces that were placed around the standard LENS moderator to enhance its coupling, but which were not present for the pelletized moderator.

Fig. 8 shows spectra from the standard LENS moderator (at 6 K and 30 K) compared to fill A of the methane pellet moderator (at 30K). We note that the pellet spectrum is slightly warmer than the standard moderator even when at the same nominal temperature.

The observed difference between the 1-eV coupling for the two moderators (standard LENS and pellets fill A), about 11 %, is roughly twice the typical variation we observe in this quantity in standard LENS operations (where the coupling can be influenced by such factors as the normalization to the proton current, the positioning of the moderator vacuum vessel within the TMR, and variations in the condensation process used to fill the standard moderator). By looking back to figure 7, we see that the variation in 1-eV coupling among various fills of the pellets is greater than this difference between the fill A and the standard LENS moderator. We conclude, therefore, that the variation in the spectrum resulting from fill to fill variations with the present system for loading pellets into the moderator is bigger than any difference between the spectrum from a pelletized and homogeneous methane moderator.

We also note that the available spectrum for the LENS moderator at 30 K was inadvertently taken with an attenuator in the beam (consisting of a BN plate with a small number of holes drilled in it to reduce the thermal flux by roughly an order of magnitude). The data for LENS at 30 K have therefore been rescaled to account for the presence of this attenuator, and the difference seen in the slope of the spectra in the slowing-down regime (above a few hundred meV) comes from a few such energetic neutrons starting to penetrate the BN itself (and therefore this run should not be used to compare spectra above 100 meV).

The emission time characteristics of the pelletized moderator are compared to those of the standard LENS moderator in figures 9 and 10. These data were collected a spectrometer consisting of a mosaic Ge crystal held at 9K at the sample position of the SANS instrument and a GS-20 scintillation detector arranged in a time-focused geometry [5]. In this geometry, the emission time distribution can be measured at energies corresponding to the available orders in the Ge(111) family of reflections, and the figures show the fundamental (2.7 meV) as well as the 3rd, 4th and 5th orders (24, 43 and 67 meV respectively). The data from the pelletized moderator, as well as data collected from the standard LENS moderator at 6K, were collected with a proton pulse width of 50 μ sec. We notice that the long-time decay seen in both the cold and thermal portions of the spectrum are identical in the monolithic and pelletized versions of the moderator. There is a slight change in the cold peak shape, for which we don not have a ready explanation. We also note that when, as in fill C, the pelletized moderator is not completely full (red curves in figures 9 and 10) the cold intensity is slightly lower, and the thermal intensity is slightly higher (in agreement with the spectra shown in figures 7 and 8).

Figure 9 shows the emission time distribution near an energy of 2.6 meV. Note that the tails of the standard LENS moderator and fill A overlap exactly. The fill-C data were taken over a shorter time, and in this set we start to see a contribution from the detector background that is not present in the other (longer) runs.

Figure 10 shows the emission time distribution from three moderators in the thermal neutron range of energies (23.7, 42.2 and 68 meV). Note the fill A of the pelletized moderator and the standard LENS moderator have essentially identical peak shapes (so identical it is difficult to see the former behind the latter in the figure).

Recommendations

The results of this project demonstrate that the neutronics performance of a moderator consisting of methane pellets is very similar to that achieved by the monolithic moderator in use at the LENS facility. Spectra from the pelletize moderator are only slightly less intense and have essentially the same spectral temperature as those seen from a monolithic methane moderator held at the same temperature. We also see no measurable difference between the emission time distributions of the two moderator types. It is also true that any of the observed spectral differences are small enough that they may be corrected by more careful optimization of the pellet cell and fill system. The difference in observed neutronics performance is not so great as to suggest that a moderator based on methane pellets would be uninteresting at higher-power sources where radiation damage would preclude the use of a solid methane moderator such as that employed at LENS.

Our experience suggests that a simple gravity feed system with a vibrational assist is capable of transferring pellets from the pelletizer to a moderator cell over a relatively short distance (total transfer distance was on the order of 6 feet). However, our results may not translate directly over to applications at a user-oriented neutron scattering facility where significant longer and more convoluted transfer lines may be required. We note that a similar pellet transfer system is being deployed at the IBR-2M pulsed reactor facility in Dubna Russia. At this facility, toluene/mesitylene spheres are being used as the moderating material (due to the enhanced resistance to radiation damage of these materials compared to methane), and they are transferred to the moderator cell over a much longer (and geometrically more complicated) transfer line with the aid of a cold helium gas flow [6]. Such a design should also be considered should a decision be made to pursue the pelletized moderator idea further for a high-powered spallation source.

Finally we note that the operation of the pelletizer at the LENS facility required some procedural controls that may not be deemed adequate for routine operation of the system at a major scattering facility.

Presentations

During the course of this project, the following presentations were made at international conferences.

1. “Neutron Moderator Studies at the Low Energy Neutron Source”, Inaugural Conference of the Union of Compact Accelerator-Driven Neutron Sources-I, Beijing China, 16 Aug. 2010.
2. “Status of Moderator Research at LENS”, delivered at a joint Coordinated Research Project and Technical Consultation Meeting at the IAEA, Vienna, Austria, 6 Dec. 2010.
3. “Moderators at LENS: Performance and Development Research”, David V. Baxter et al., Union of Compact Accelerator-driven Neutron Sources-II, Bloomington, IN, 6 July, 2011.
4. “Innovative concepts in Cold Neutron Moderation and Science at Small Neutron Sources”, David V. Baxter, First NANO collaboration meeting, Kolkata India, 21 July 2011.
5. “Exploring new Moderator Concepts at LENS”, American Conference on Neutron Scattering, Washington, DC., 26 June, 2012

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Project Summary

This project demonstrated, for the first time, the capability of producing a methane pelletizer, installing it on an operating pulsed neutron facility, filling a cold moderator with methane pellets and producing a cold neutron beam that was similar to that of a solid methane block moderator. While solid methane moderators are used at many neutron facilities, they are not suitable for high power spallation sources since they cannot be adequately cooled. Because of this large sources such as the SNS use supercritical hydrogen moderators. This work opens the possibility of achieving a liquid hydrogen cooled methane pellet moderator on these high power sources which would have the potential of doubling their cold neutron brightness. However, before this happens a lot of work and testing has to be done, in both the pellet fabrication area and the transport of the pellets into and out of the moderator.

This project and the work done in two previous SBIRs could form the basis for implementing a methane pellet moderator on a high power neutron source. In the first project[1,2,3] we demonstrated the successful production of both ammonia and methane pellets. It was found that the methane pellets readily stuck together, so the phase II work focused on reducing this by cooling the pellets to low temperature prior to loading them into the moderator cell using cold helium gas. While this method may be eventually worked out, we failed to do this to the fact that the methane pellets readily stuck to cold surfaces. We did however find that adding 1% of ethane to the methane reduced the stickiness of the pellets and allowed the filling of the moderator cell. A vibrator was also used during the loading of the cell to settle the pellets. Once loaded, the pellets will start to sinter together. While moderators held at temperatures below 20K might sinter at a slower rate, they would probably sinter over the long periods. In this project we also developed a method of removing the sintered pellets from the moderator cell by the injection of methane gas which rapidly (seconds) melts the solid pellets and flushes out the remaining liquid. Removing the pellets in liquid form, as opposed to evaporation, would have the advantage of flushing out higher chain hydrocarbons formed in the moderator. This method leaves the moderator cell and the transfer lines above 90K which is an ideal temperature to reload the moderator with fresh pellets.

In the second SBIR[4,5] the focus was transporting the pellets and developing methods of rapidly filling a large moderator cell. The honeycomb pelletizer requires a rather long time to release the pellets, so methods were tried to accumulate the pellets without having them sinter together. A cryogenic pellet tumbler was constructed which partially worked, but still had problems with the pellets sintering. We finally developed a method of storing them separated in the honeycomb prior to release, which resulted in the capability of loading 1.1 liters of pellets into a moderator cell in 10 seconds. We found that the pellets could be loaded by keeping the temperatures of the cell to ~77K and the transfer line above this. At these temperatures the pellets float on the warmer surfaces with essentially no friction and can be transferred through smooth tubes or slides at fairly low angles. The best transfer lines were made with a series of “waterfall” breaks so that the pellet speed was kept low. Fast pellets impacting into a partially full moderator cell tend to stick more on the pellet impact.

References (see also IU report).

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