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# The Cryogenic Cooling Program at the Advanced Photon Source

by C. Shawn Rogers, Dennis M. Mills, and Lahsen Assoufid

Experimental Facilities Division
Advanced Photon Source

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# The Cryogenic Cooling Program at the Advanced Photon Source

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**Abstract**

This paper describes the experimental and analytical program in cryogenic cooling of high-heat-load optics at the Advanced Photon Source. A prototype liquid nitrogen pumping system has been procured. This pump provides a variable flow rate of 1 to 10 gpm of pressurized liquid nitrogen and is sized to handle up to 5 kW of optic heat load. Also, a high-vacuum, double-crystal monochromator testing tank has been fabricated. This system will be used to test cryogenic crystals at existing synchrotron sources. A finite element analysis has been performed for a cryogenically cooled Si crystal in the inclined geometry for Undulator A at 100 mA. The inclination angle was 80°. It was set to diffract from the <111> planes at the first harmonic energy of 4.2 keV. The maximum slope error in the diffraction plane was calculated to be about 1 μrad with a peak temperature of 94 K. An analysis has also been performed for a cryogenically-cooled "thin" crystal oriented in the Bragg geometry which accepts 87% of the 1st harmonic photons at 3.866 keV. The total absorbed power was 131 W at 100 mA current and the peak temperature was 124 K.

## 1. Introduction

The combined requirement of an x-ray optical component to be simultaneously capable of withstanding extraordinarily high heat fluxes while maintaining minimal distortions makes x-ray optics designs for third-generation synchrotron sources one of the most challenging aspects of the development of such sources. We at the APS have mounted an aggressive R&D program to find solutions to this critical problem. Much of our initial effort went into the development of a liquid gallium cooling system because we saw this as an effective and relatively straightforward step to go beyond the cooling capabilities of water with only minor changes in the design of crystals and manifolds. A final, very important, reason for pursuing the liquid gallium cooling approach was that we had developed at the APS the technology to design and build effective systems for pumping liquid gallium. However, we are also aware that even with the larger thermal conductivity of liquid gallium, there is a limit to the amount that the temperature gradients, and hence strain, can be reduced. This limit is imposed by the physical properties of the optic material itself. The thermal resistances to heat flow from the crystal to the fluid are: (1) conduction resistance through the silicon and (2) convection resistance through the solid-fluid interface, or boundary layer. The ratio of these resistances is called the Biot number, *Bi*,

$$Bi = ht/k,$$

where $h$ is the heat transfer coefficient of the fluid, $t$ is the thermal path length in the component from the heat source to the coolant, and $k$ is the thermal conductivity of the component. Once a Biot number of approximately 1 has been attained, a region of diminishing returns is reached, because, even for large increases in the heat transfer coefficient, minimal reductions in the peak temperature will be achieved. Therefore, if the performance of the optical component when the Biot number is greater than 1 is not acceptable, alternative approaches other than increasing $h$ must be considered. For x-ray optical components, two approaches are currently being explored: changing the material of the components (from silicon, the most commonly used monochromating crystal, to diamond, for example), or modifying the properties of the existing material (silicon) through a change in operating temperature. Fortunately, the temperature dependence of the thermal conductivity and coefficient of thermal expansion of silicon both change in favorable directions as the operation temperature is reduced from room temperature (see Fig. 1). It is these properties that are the basis for interest in cryogenic optical components for third-generation synchrotron radiation sources.

The concept of using silicon at cryogenic temperatures for high-heat-flux optical components is not a new one. Bilderback first pointed out the potential for cryogenically cooled synchrotron radiation optical components in 1986 [1]. However, the use of silicon at low temperatures had been considered for high power laser mirrors in the seventies [2]. Recently, a considerable effort has also been put into the development of a liquid nitrogen cooling system for third-generation synchrotron sources, particularly by the European Synchrotron Radiation Source (ESRF) staff, but also by other groups. (The details of some of these efforts can be found in references 3-8.)

The APS cryogenic program began in earnest over a year ago when we developed a set of specifications and solicited bids for the development of a closed (primary) loop liquid nitrogen system for cooling high-heat-load optics. Because of the complexity of a cryogenically cooled optical system, our approach was to develop this technology in parallel with the liquid gallium system because we were concerned about potential difficulties in the development of the hardware, both cryogenically compatible monochromators and crystals, required to put the cryogenically cooled crystal concept into operation. During the liquid nitrogen pump design and fabrication period, we have been developing a test monochromator compatible with operation at liquid nitrogen temperatures and have been performing thermal analyses, both analytical and finite element, on crystals of various shapes and with different heat exchanger designs. The liquid nitrogen pump has now been delivered, and we have begun testing the pump and integrating it with the monochromator and crystals for further evaluation at existing synchrotron sources. This report chronicles the progress of that activity until the present (spring of 1994).

## 2. The Pros and Cons of Liquid-Nitrogen-Cooled Optical Components

As mentioned above, the primary motivation for operating x-ray optical components at cryogenic temperatures is the simultaneous reduction, in fact, a zero crossing at 125 K, of the coefficient of thermal expansion, $\alpha$, and increase in thermal conductivity, $k$, for silicon. The combination of these two effects provides an increase of a factor of 50 in the so-called thermal figure of merit (FOM) in silicon at 100 K as compared to silicon at 300 K and in fact is comparable to the FOM of room temperature diamond, another potential crystal material for use in high-heat-flux x-ray beams (see Table 1).

Table 1. Figure of Merit for Silicon and Diamond at Room Temperature and at Cryogenic Temperatures. Data taken from Thermophysical Properties of Matter [9].

| Material | Temp. (K) | $k$ (W/cm K) | $\alpha$ ($10^{-6}$ $K^{-1}$) | FOM ($k/\alpha$) |
|---|---|---|---|---|
| Silicon | 300 | 1.4 | 2.6 | 0.53 |
| Silicon | 100 | 13.4 | -0.5 | 26.8 |
| Diamond, type I | 300 | 9.0 | 1.0 | 9.0 |
| Diamond, type I | 100 | 30.1 | 0.05 | 602 |
| Diamond, type IIa | 300 | 23.1 | 1.0 | 23.1 |
| Diamond, type IIa | 100 | 100 | 0.05 | 2000 |

Along with the positive aspect of improved physical properties of silicon at cryogenic temperatures, there are some negative aspects that must be evaluated. Potential users of liquid nitrogen systems should be aware of both aspects, and so we list several difficulties that will have to be overcome in order to make this approach viable.

Ignoring for the moment the plumbing and monochromator compatibility issues associated with cryogenically cooled crystals, the primary difficulty with the use of liquid nitrogen systems is the poor heat transfer properties of liquid nitrogen as compared with water or liquid gallium. One of the challenges that must be overcome with liquid nitrogen is the low value of its critical heat flux (CHF) as compared with other coolant fluids, which limits the amount of heat per unit area that can be extracted from the component by the coolant. The critical heat flux is defined as the value of the heat flux at which the cooling fluid is vaporized instantly upon contact with the wall, producing between the wall and the coolant a thin vapor film with poor heat transfer properties resulting in burnout, i.e., physical damage or even melting, of the component. Therefore, in order to avoid "burnout" in the optical component, two principles must be adhered to: (1) minimize the amount of heat absorbed in the optical component, and/or (2) incorporate large surface areas for removal of the heat loads that are deposited in the component. Due to the high thermal conductivity of cryogenic silicon, the nitrogen coolant may be placed further from the diffraction surface allowing the heat to diffuse through a larger volume, thereby, decreasing the heat flux at the solid-fluid interface. Liquid nitrogen has a critical heat flux (CHF) value in the range of only several tens of W/cm$^2$, depending on the details of the heat exchanger. In a recent report by Usui et al., a measured value of 20 W/cm$^2$ was reported [8]. This should be compared to water with typical CHF values of 200-500 W/cm$^2$. It should be noted the CHF is a function of many parameters, including the amount of subcooling, pressure, flow rate, geometry, and surface conditions. Hence the design of heat exchangers for the APS-compatible optics will require careful consideration due to the high power and power densities expected from APS undulator beams. Several approaches have been considered to reduce the total absorbed power in the component including the use of a mirror in front of the cryogenically-cooled optic to act as a power filter. This approach has been examined for the APS Undulator A by Yun et al. [10]. Also, using slits to permit only the central cone of the undulator radiation to impinge on the cryogenic crystal has been investigated. Another approach is to use thin crystals, a concept that has been considered by a number of researchers and looks to be promising [11,12,13]. Also, use of the inclined crystal geometry can provide an additional spreading of the beam by an order of magnitude, further reducing the impinging heat flux [14].

Another important consideration when using liquid nitrogen as a coolant is the pressure in the system. The liquid phase of nitrogen has a narrow temperature range at atmospheric pressure and a specific heat approximately half that of water. Consequently, the total amount of heat that the liquid nitrogen can transport before boiling occurs is limited when compared to water. To increase the temperature range over which the coolant is in the fluid state, the system can be pressurized. For example, with a flow rate of 2 gpm, a coolant pressure of 3 atm, and beam power of 1 kW, the average fluid temperature rise is about 5 K. The saturation temperature at 3 atm is 85 K, therefore, the bulk of the nitrogen should remain single phase. Because the properties of liquid nitrogen depend strongly on pressure, the pressure drop through the coolant channels must be carefully considered. This high coolant pressure (relative to water) has the potential to distort the optical component if the component is not carefully designed. Fortunately, faceplate thicknesses can be considerably larger for cryogenically cooled silicon as compared to water or gallium cooling, due to the improved thermal conductivity of silicon at cryogenic temperatures and, therefore, the higher pressure should not be a problem. Another approach is to have the high pressure coolant contained in a separate loop, i.e., not running through the component but cooling the silicon optics by contact to this loop. This technique alleviates the pressure-induced distortion in the optic but can result in a large thermal contact resistance and, consequently, a large temperature rise if the optical component and coolant loop are not in intimate contact.

Finally, although not necessarily in the "con" list but definitely in the nuisance category, is the fact that the lattice spacing, $d$, of the first and second crystals will be different if the second crystal is not cooled to the same temperature as the first. The fractional length change, $\Delta d/d$, of silicon upon cooling from 293 K to 100 K is -0.024% ($-2.4 \times 10^{-4}$) [9]. For a given x-ray energy, this change in length (or d-spacing) corresponds to a change in the Bragg angle given by:

$$\Delta\Theta = -(\Delta d/d)\tan(\Theta).$$

If the first crystal of a double-crystal monochromator is at cryogenic temperature and the second crystal is at room temperature, the twice diffracted beam will no longer be parallel to the incident beam, but will deviate from parallelism by twice $\Delta\Theta$, hence, the beam will walk (vertically) as a function of energy. Figure 2 shows the vertical beam motion as a function of Bragg angle, $\Theta$, at 10 meters from the monochromator. This walk can be corrected, in principle, by changing the separation of the two crystals in a prescribed way as the Bragg angle is changed.

## 3. The APS Liquid Nitrogen Pumping System

The Optics Group of the Experimental Facilities Division at the Advanced Photon Source has procured a liquid nitrogen pumping system for use in an R&D program to study the feasibility of cryogenically cooled high-heat-load optics. For a complete description of the pumping system refer to Ref. 15. The pumping system has been designed to provide a variable flow rate of pressurized liquid nitrogen. A flow schematic for the system is shown in Fig. 3. The system consists of a vertical dewar with a submerged rotary-vane pump. The dewar is enclosed within a vacuum-insulated heat exchanger. The pump speed is controlled by a variable-speed DC motor, which is coupled to the pump shaft through the top flange of the dewar.

Liquid nitrogen in the closed primary loop leaves the high side of the pump and passes through a coil in the heat exchanger tank removing any heat the pump added to ensure maximum cooling on exit from the system. It then flows through a turbine flow meter and out through a flexible line to the inlet of a double block and bleed valve box. The valve box is connected to the monochromator vacuum chamber and allows the flow to be bypassed so the monochromator is accessible while the pump continues to run. This device allows for quick crystal changes and safe access to the monochromator interior. After passing through the crystal, the nitrogen flows back through another flexible vacuum-jacketed line to the pump heat exchanger. The optic heat load is removed by boiling of atmospheric liquid nitrogen in the shell, or open-loop, side and the cooled optic-loop nitrogen is returned to the suction side of the pump well. The optic-loop liquid nitrogen inventory remains constant. The nitrogen level in the shell side of the heat exchanger is maintained automatically by an external storage dewar. The heat exchanger consumes approximately 0.1 gallons per minute per kilowatt of optic heat load. The system pressure is controlled by applying a helium pressure over the suction side of the pump. The primary design parameters are:

- Variable flow rate up to 10 gallons per minute
- Variable system pressure from 0 to 150 psig
- Maximum optic heat load of 5 kilowatts

The performance of the pumping system has been measured and is depicted in Fig. 4. This figure shows the pressure drop across the pump as a function of volume flow rate. Two cases are shown. The first case is for the flow short circuited through the bypass valve, and the second case is for flow into the monochromator and passing through a 9-hole crystal shown in Fig. 5. The hole diameter is 0.125", and the path length is about 2.4". It is shown that the pressure drop increases exponentially with the volume flow rate or, equivalently, the fluid velocity. This is expected for turbulent flow. For flow through the bypass, the pressure drop is about 18 psi at 10 gpm. The rate of head-pressure increase for flow through the crystal is significantly greater. The peak flow rate attainable was about 4.5 gpm at 30 psi differential pressure. The flow rate is limited because the present motor being used to drive the pump is too small to provide the increased torque at higher speeds. Most of the pressure drop inside the monochromator is due to the 1/2" flexible stainless-steel transfer lines. For the present tests, there were approximately 100" of transfer line inside the monochromator. The pressure drop can be significantly reduced by increasing the diameter of hose and decreasing the length. The hose diameter will be increased to 3/4" for future tests. If additional pumping power is still required, a larger motor can be easily fitted to the system.

## 4. Thermal Analysis of Cryogenically Cooled Crystals for the APS

The design considerations for cryogenic monochromator crystals are significantly different than for room-temperature crystals. The two main differences are that the thermal conductivity of silicon is about an order of magnitude larger at cryogenic temperatures, and the transport properties of liquid nitrogen are far worse than those for either water or liquid gallium. The resistances to heat flow from the crystal to the fluid are conduction resistance through the silicon and convection resistance through the solid-fluid interface, or boundary layer. For room-temperature crystals, the conduction resistance usually dominates. Therefore, it is often desirable to minimize the thermal path length in order to decrease the magnitude of the thermal gradients. However, the situation is reversed in the cryogenic case. The cooling channels can be placed further from the diffraction surface due to the higher thermal conductivity and smaller heat transfer coefficient. Locating the cooling fluid further from the diffraction surface has the added benefit that the heat diffuses throughout the crystal, thereby, reducing the thermal flux at the cooling channel interface and the likelihood of boiling.

Two general classes of crystals are often considered for cryogenic use: thin and thick. Thin crystals are designed so that only a small portion of the x-ray power is absorbed.

The absorption coefficient is a function of the x-ray energy, and, accordingly, the power is deposited into some finite depth. Much of the hard x-ray energy passes completely through the silicon. Roughly speaking, thin crystals are usually < 1 mm thick. Thick crystals, > 1mm thick, absorb a large fraction of the energy in the beam.

The advantages of thin crystals are:

- Less absorbed power
- Less liquid nitrogen consumed
- Smaller thermal gradients perpendicular to the diffraction surface

The disadvantages are:

- Heat must flow through a thin membrane rather than a full 3D solid
- Fabrication is more difficult and care must be exercised not to mechanically distort or strain the crystal
- Cooling channels are necessarily further from the hot spot

The advantages of thick crystals are:

- Easier to fabricate
- Crystals are more substantial, less susceptible to mechanical distortions and so easier to manifold
- Cooling geometry is not as limited as the thin case
- Inclined geometry can be readily used

The disadvantages are:

- Most of the x-ray power is absorbed
- More liquid nitrogen is consumed
- Crystal heat exchanger must be larger
- Larger thermal gradients normal to the surface

### 4.1. Finite Element Analysis

Finite element techniques are being used to investigate the performance of both thick and thin crystals for undulators and wigglers. At this point , analyses have been performed on several crystal geometries for undulator radiation. A summary of the results of those analyses is given below.

#### 4.1.1 Wigglers

Although the normal incidence power density from Wiggler A is smaller than that from Undulator A, the design of cryogenic wiggler crystals is complicated by other factors. The wiggler beam is wider than the undulator beam; therefore, the inclined geometry cannot be used unless only a smaller portion of the beam is taken. Also, the total absorbed power is higher. A variable asymmetric crystal may be a better way to spread out the incident beam [16]. The size of the beam accepted should be limited to only what is absolutely required in order to minimize the liquid nitrogen consumption. Computational analyses are in progress for cryogenically cooled wiggler monochromators.

### 4.1.2 Undulators

The two best candidate crystal designs for use with APS undulators are inclined crystals using porous-media-enhanced channels [15, 17] and thin crystals. Taking the entire central cone of radiation from Undulator A gives about 750 W of power and a peak power density of 150 W/mm$^2$ at 30 m from the source at closed gap conditions for 100 mA stored current where the deflection parameter, K, is 2.17, and the first harmonic energy is 4.21 keV.

Figures 6, 7, and 8 show the results of a finite element analysis for a cryogenically cooled crystal in the inclined geometry for APS Undulator A at 100 mA. A more extensive analysis will be presented at the 1994 SRI conference [18]. The crystal is 25 mm wide, 50 mm long, and 10 mm thick. Using slits, the beam is cut down to allow 50 $\mu$rad vertically and 120 $\mu$rad horizontally to pass. The crystal is inclined at 80$^o$ and set to diffract 4.2 keV photons in the first harmonic from the <111> planes. The distance from the source is 30 m. The crystal is uniformly cooled from the bottom surface with a heat transfer coefficient of 5 W/cm$^2$ K. This magnitude of heat transfer coefficient should be readily achievable by using porous copper inserts in the cooling channels [15]. Figure 6 shows that the peak temperature is about 94 K, and the minimum temperature is 82 K. The absorbed heat is diffusing quite readily throughout the entire crystal volume. Figure 7 shows the a.) displacement, and b.) slope along the centerline of the surface of the crystal in the beam direction. It is shown that instead of the normal "thermal bump" for room temperature crystals, the cryogenic crystal has a "thermal dimple". This is a result of the negative thermal expansion coefficient below 125 K. Figure 8 shows the a.) displacement, and b.) slope along the centerline of the crystal for the diffracting planes. The crystal was treated as free-standing for all calculations. The peak slope error at the beam position is about 1 $\mu$rad. A full-scale analysis is underway to calculate the performance of cryogenic crystals for beam currents up to 300 mA.

A thin crystal has also been analyzed [19]. This particular crystal diffracts from a thin membrane approximately 0.6 mm thick. The crystal is cooled from the side by liquid nitrogen. The heat transfer coefficient was chosen to be 0.6 W/cm$^2$ K. The membrane is 2.4 mm wide, which allows about 87% of the central cone of radiation from Undulator A to be accepted. The membrane absorbs about 1/3 of the beam power at 3.866 keV. The remainder is transmitted through the crystal. The peak temperature is 124 K. The structural analysis for this geometry is in progress.

## 5. Crystals and Planned Tests

To date, two crystals have been fabricated for synchrotron tests at CHESS in the summer of 1994. The high-heat-load crystals have seven 1/4" diameter holes core-drilled through them approximately 1 cm below the diffraction surface as shown in Fig. 9. One of the crystals will have a porous matrix of copper wire bonded into the holes. The other crystal will be unenhanced. These crystals will give a direct comparison of the heat transfer enhancement due to the porous media. The enhancement of the effectiveness of the heat exchanger is expected to be about 500% to 600% [15]. The crystal surfaces have been oriented in the <111> direction. They can also be used in the inclined geometry using the <11$\bar{1}$> reflection with an inclination angle of 70.529°. Fabrication of the cryogenically cooled thin crystal of the geometry described above is in progress. We plan to test these crystals on a focused wiggler beam at CHESS having a power and power density comparable to the APS Undulator A at 100 mA.

## 6. Summary

The use of cryogencially cooled crystals as a solution to the thermal distortion problem from third-generation synchrotron sources is clearly an attractive one. The combination of enhanced thermal conductivity and reduced coefficient of thermal expansion bodes well for improved optical performance. As a testament to this fact, several beamlines at the ESRF are currently operating routinely with cryogenically cooled crystals [20], and the diagnostics team in the Insertion Device Group at the APS is currently considering cryogenically cooled monochromator crystals to measure the source properties of the beam, a measurement that requires monochromator optics of the highest quality [21].

We now have an operating prototype liquid nitrogen pumping system and will in the near future, test the performance of cryogenically cooled silicon crystals on a focused wiggler beamline at CHESS. If the mirror on this beamline performs as expected, it will provide a total power and power density comparable with that of a slitted APS Undulator A. This system will be used for R&D activities and hence was specified to operate over a large range of pressures, flow rates, and heat removal capacity. However, a more compact version may be useful for many undulator applications where slits and thin crystals are used to limit the total power absorbed, and we plan to pursue this along with upgrades to our current prototype system in the very near future.

Applying liquid nitrogen cooling to synchrotron optics is still a relatively novel concept in practice. Much experimental work is still to be done to determine where the operational difficulties lie. One of the main areas of concern is the susceptibility of nitrogen to boil. The design of cryogenic-compatible double-crystal scanning monochromators will have to be addressed. It will probably be necessary for both crystals to be cooled to eliminate the vertical walk problem discussed earlier. Strain-free and thermally isolated mounting techniques will be required.

Looking to the future, the APS aspires to operate with a stored current of 300 mA with 5-meter-long insertion devices installed in the ring. The combined effect of these two improvements will result in power densities approaching 1 kW/mm$^2$ at 30 meters from the source. Handling such astounding heat fluxes may require not cryogencially cooled silicon but cryogenically cooled diamond, which has a figure of merit as high as 2000! (see Table 1). This "ultimate monochromator" will permit experimenters to keep pace with and handle the beams that will be in store for us in the not too distant future.

## 7. Acknowledgments

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(a)

(b)

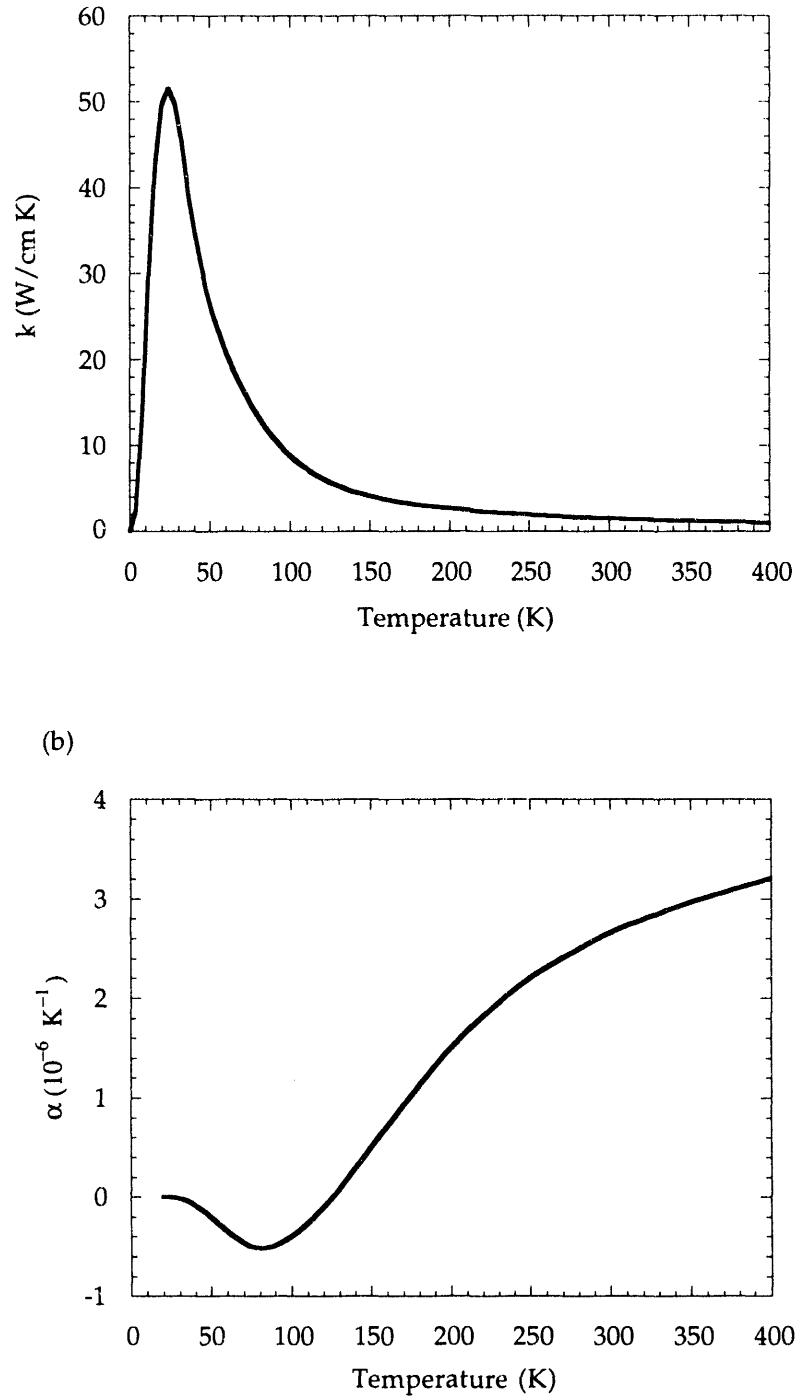



Figure 1 (a) Thermal conductivity and (b) coefficient of thermal expansion of silicon as a function of temperature. (Data taken from Ref. 9.)

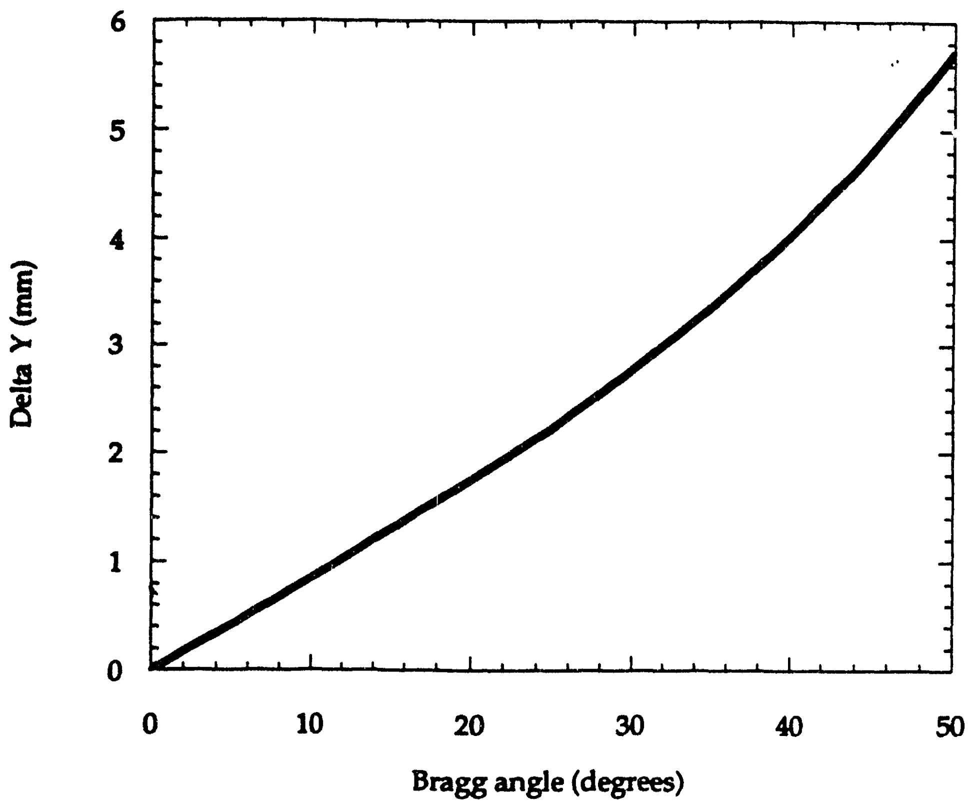



Figure 2 Vertical walk of beam at 10 meters from the monochromator with first crystal at 100 K and the second crystal at 293 K.

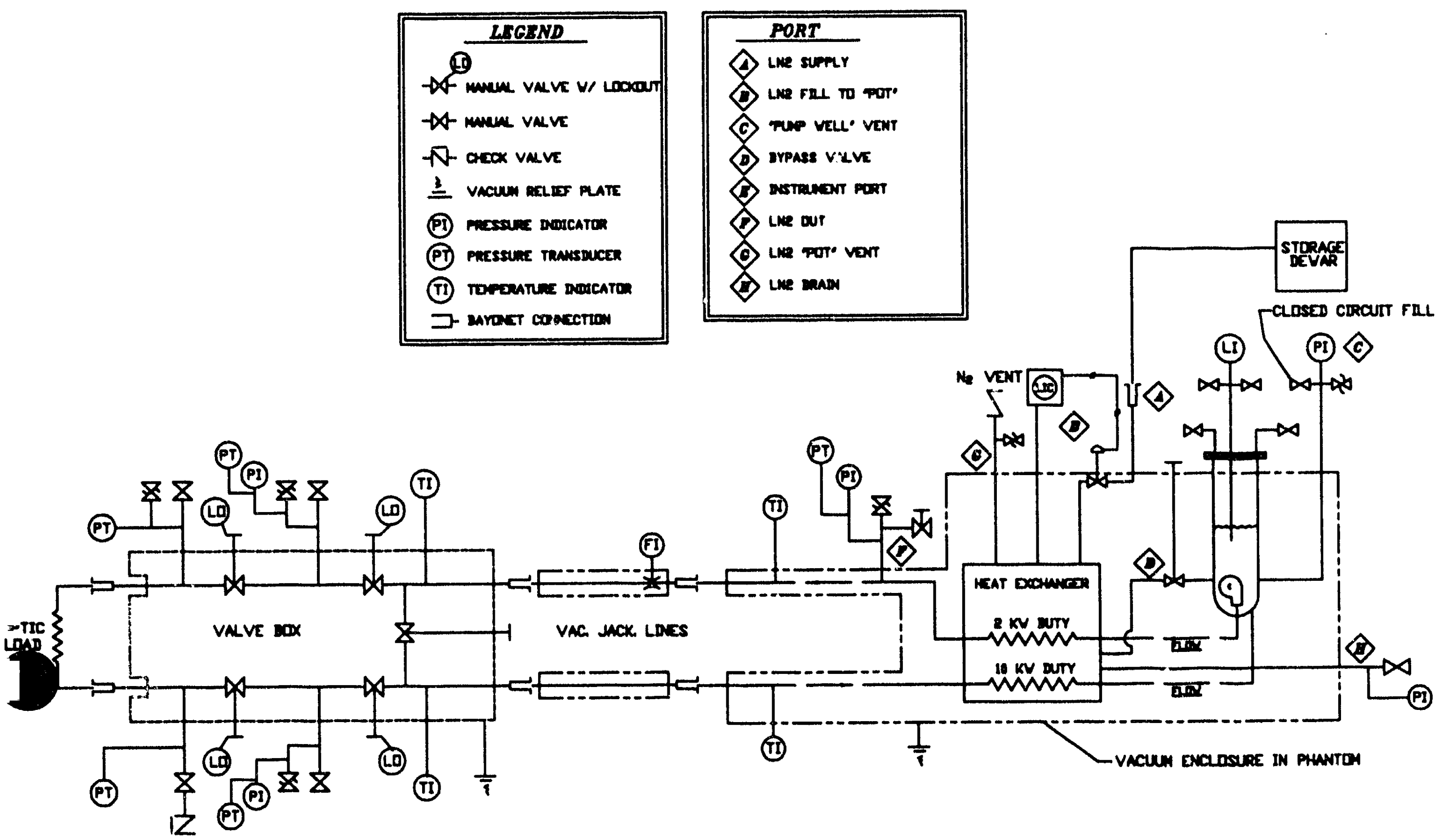



Figure 3 Schematic of APS liquid nitrogen pumping system

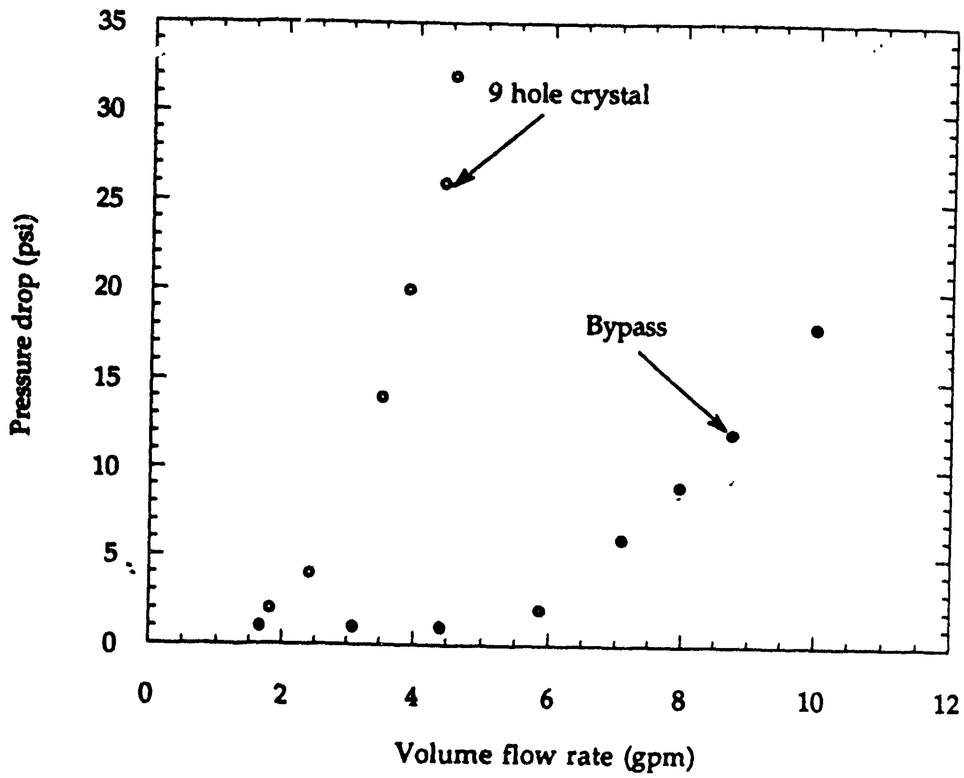



Figure 4 Liquid nitrogen pumping system performance for flow through bypass and a 9-hole crystal

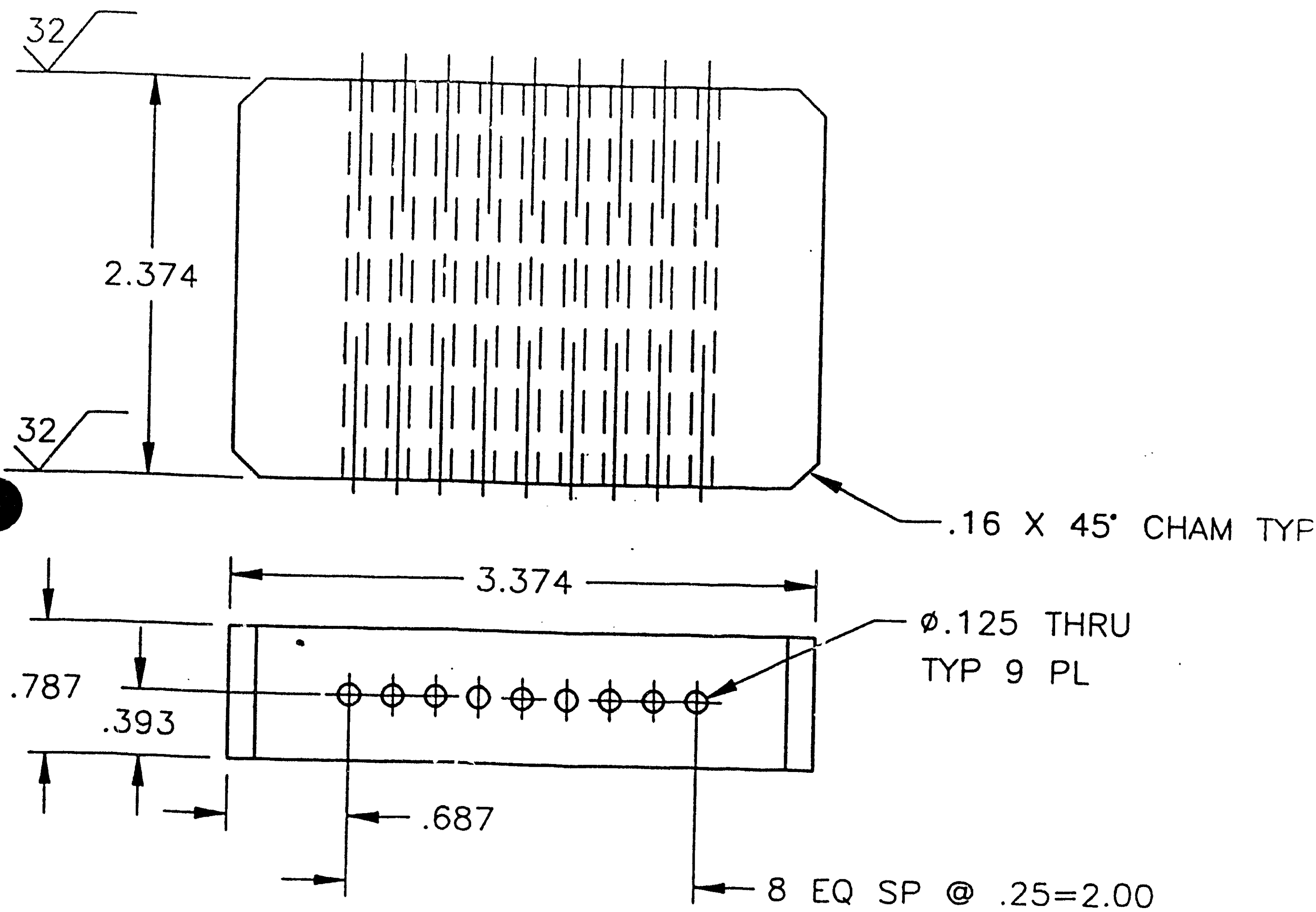



Figure 5 Schmatic of a 9-hole cryogenic crystal used to test the liquid nitrogen pumping system performance

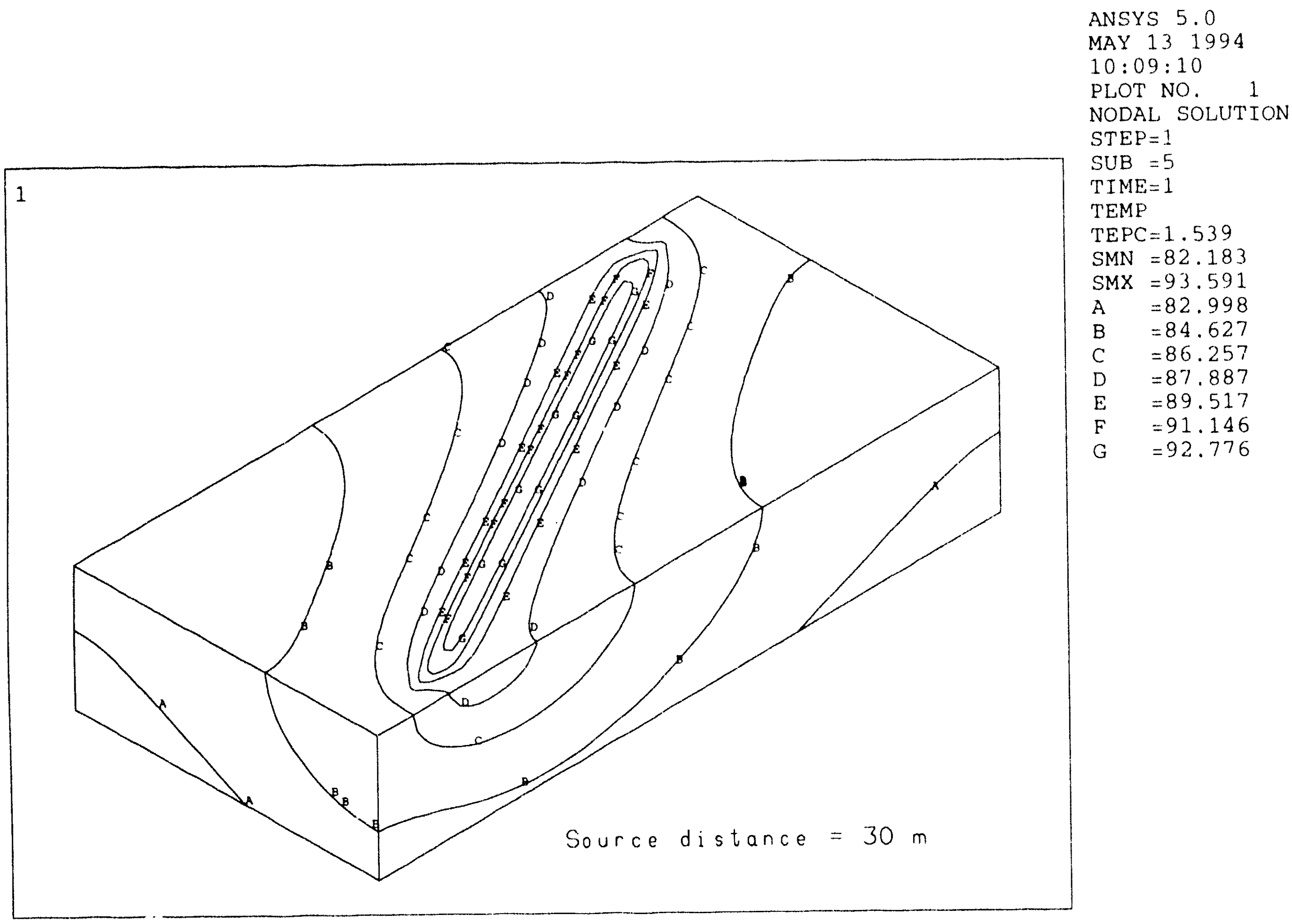



Figure 6 Thermal results from finite element analysis for 80° inclined cryogenic crystal for Undulator A

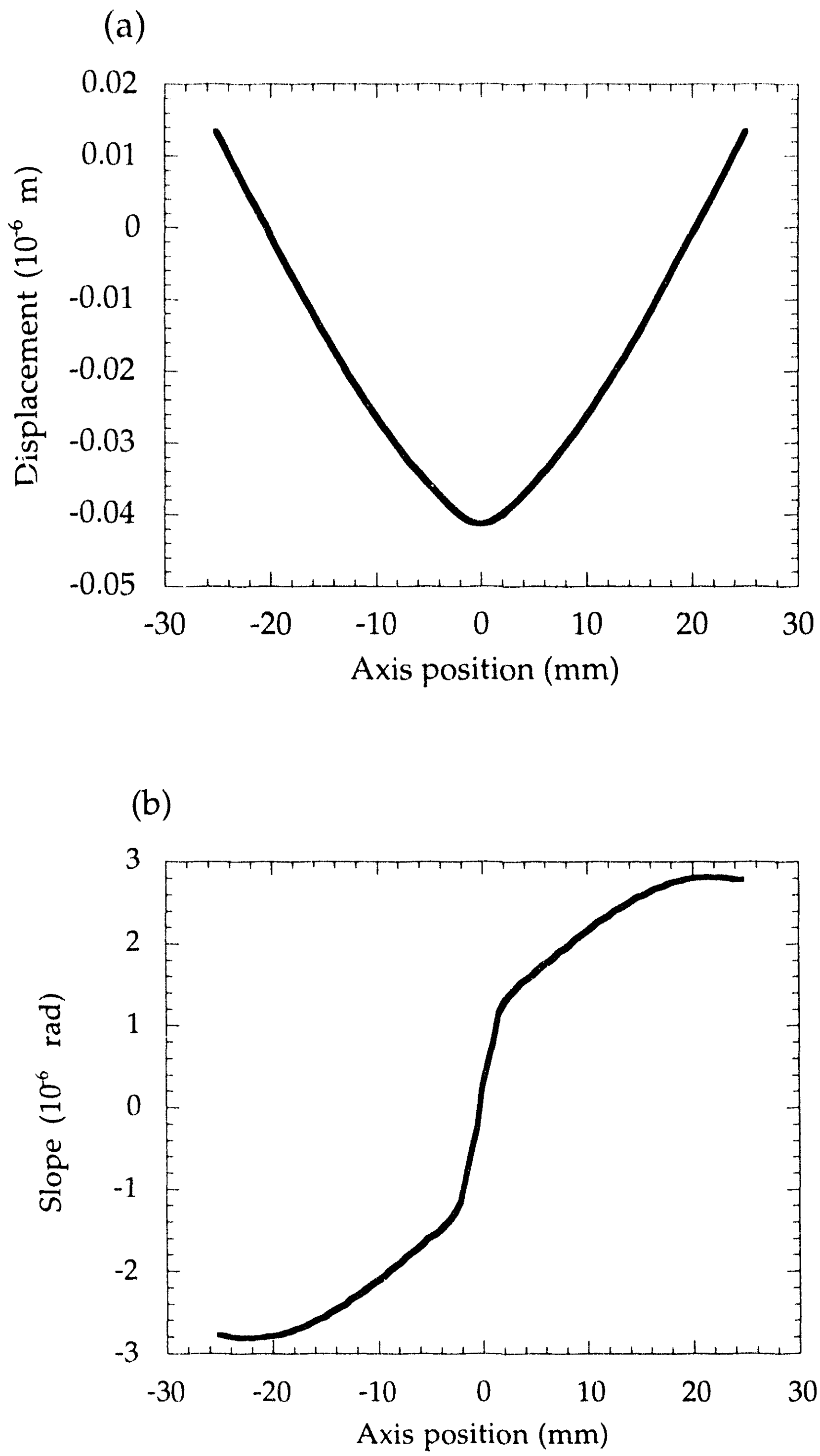
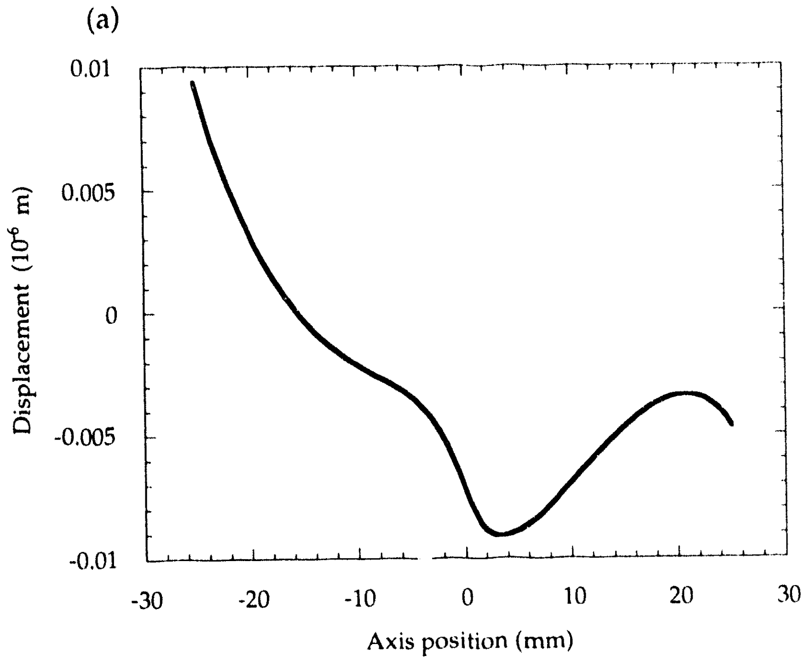



Figure 7 (a) Displacement, and (b) slope of crystal surface along the central axis from finite element analysis of inclined crystal for Undulator A



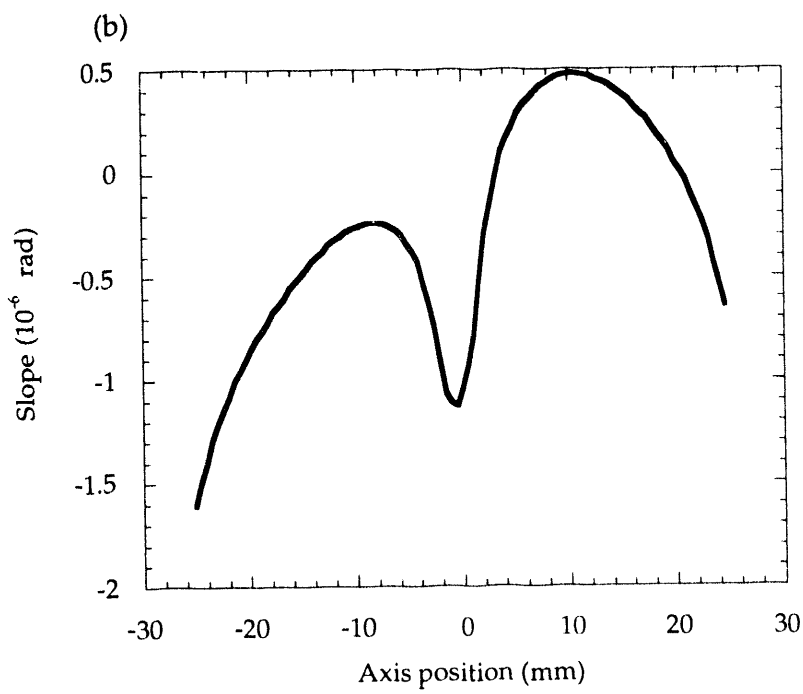




Figure 8 (a) Displacement, and (b) slope of diffraction plane along the central axis from finite element analysis of inclined crystal for Undulator A

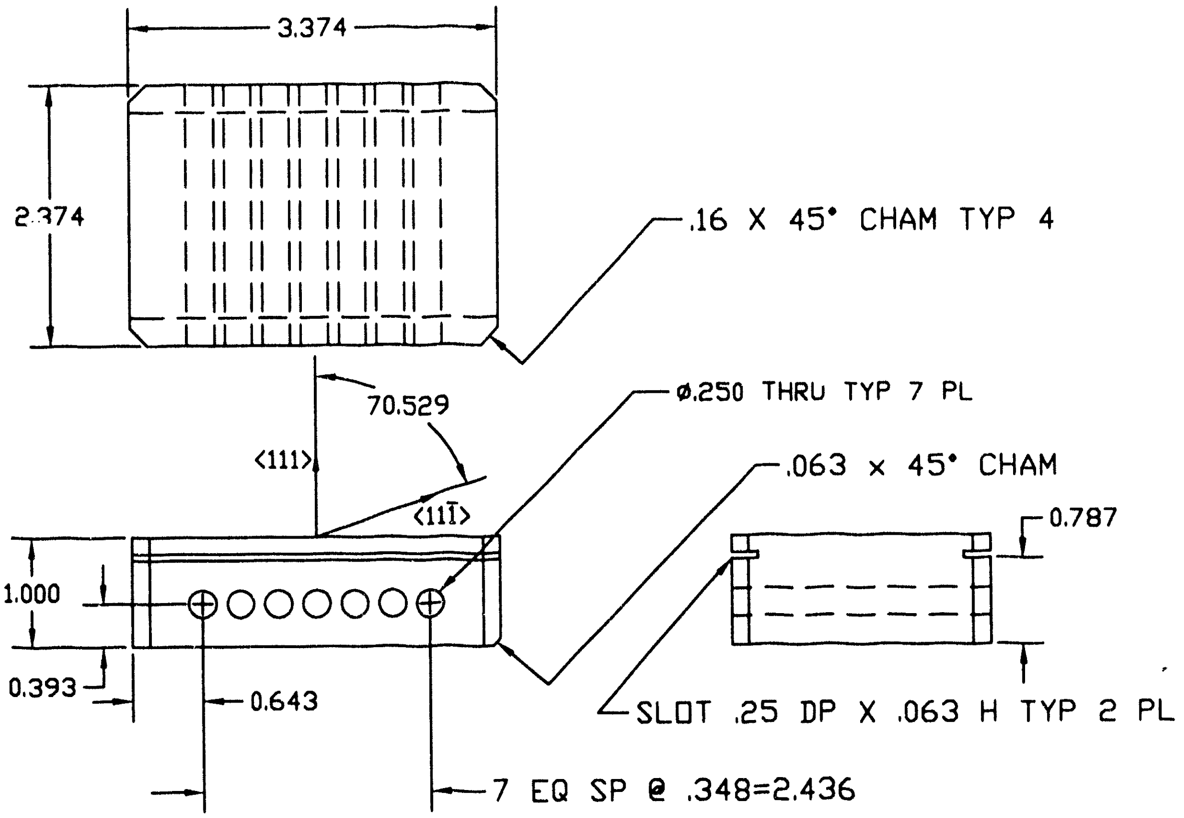



Figure 9 Schematic of cryogenic crystal for use with Cu mesh inserts

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