MARCH 1996 NSLS NEWSLETTER

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Important Upcoming Dates

TABLE OF CONTENTS

Announcements and Reminders

NSLS Home Page............ BNL Home Page

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MARCH 1996 NSLS NEWSLETTER

IMPORTANT UPCOMING DATES

May 20 and 22, 1996

NSLS Workshops

May 21, 1996

1996 NSLS Annual Users' Meeting

May 31, 1996

Deadline for General User proposals

NSLS Home Page............ BNL Home Page............

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MARCH 1996 NSLS NEWSLETTER

Introduction by the NSLS Chairman

Michael Hart

As we all await with keen interest the final budget allocations of the 1996 Fiscal Year Scientific Users Facilities Initiative, work has started on the proposal for 1997.

At the half way point of the Fiscal Year users can already feel the impact of the initiative; the NSLS has been able to continue the expansion in numbers of user support staff, there was no slowing down of the infrared beam port installation program, nor of the developments on insertion device beamlines leading towards user beam time. Planned improvements to the storage rings such as improved global feedback control systems and the replacement of the 52MHz RF cavities and the beryllium window upgrade, which will allow operations at higher current, also continue. At the time of writing the outcome of the competition for funding based on peer reviewed proposals is not decided. We do know that the NSLS user community responded with vigor and that the needs documented covered the whole range of activities and objectives of the funding initiative. Beamline components, monochromators and mirrors were requested, as well as instrumentation which will increase resolution and data quality on both the VUV and X-Ray Rings, detectors and electronics to enable faster or new experiments and complete experimental rigs to undertake more complicated and difficult experiments. The proposals from NSLS users covered the full spectrum of radiation available at NSLS, from the infrared to hard x-rays and applications came from the full range of disciplines represented in the user community.

The success of the first Facilities Initiative stems not only from the fact that NSLS is able, this year, to sustain the program of facility use and development, nor alone from the enthusiasm with which the NSLS community responded. Its success stems rather (through the collective and constructive collaboration) from the ability of the DOE facilities and their users to make the case that relatively modest investment could have a substantial leverage on both the quality and quantity of science performed at existing facilities. We are already charged with the task of demonstrating the truth of our assertion by experiment - quarterly reporting of achievements in this the First Facilities Initiative Year compared with the same period last year. Let us turn the theory into practice!

There is no doubt in my mind that the lessons learnt in the first year will enable us to do even better next time. I have already discussed that with the Users Executive Committee and it will be an important topic too at the Annual Users' Meeting in May.

MARCH 1996 NSLS NEWSLETTER

Facility Report

Mike Kelly, NSLS Building Manager

On December 14, 1995, the X1-X4 Expansion addition was inspected and accepted by Brookhaven National Laboratory as an operational facility. This completes the current building expansion of the NSLS. Presently, every NSLS beamline has a laboratory or setup space in close proximity to their beamline (The Denis McWhan Plan). The Staff/User shop has moved from the basement of Building 535 to the experimental floor. It is located adjacent to the setup labs near the stock room. The Building Manager and the Safety Staff have moved into our new quarters opposite beamlines X1-X4.

We have improved the gas bottle storage facility. The Helium cylinder stock has been increased from 20 bottles to 50 bottles. All speciality gases are placed as indicated in the illustration. Empty gas cylinders are placed in the three racks next to the rollup door. Many gas cylinders haven't been used in years. It is quite possible you or someone is paying a monthly demurrage charge on certain cylinders. Please look through the speciality gas racks, and send back your old cylinders to the supplier. We can certainly use the room!

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MARCH 1996 NSLS NEWSLETTER

A User's Perspective

Paul Zschack (ORNL), Users' Executive Committee Chair

Let me begin by thanking those Users who took the time to write letters in support of the Science Facilities Initiative (SFI). This demonstration of widespread support, together with the hard work of the Facilities Directors and the DOE helped keep the SFI in the Basic Energy Sciences budget. A portion of these funds have been made available to individuals for "...new capabilities or for upgrading existing research capabilities...at DOE-supported synchrotron facilities.."; I am confident many outstanding proposals have been submitted by NSLS Users to the DOE for this grant support. We can also be hopeful that this Initiative results in a long-term increase in funding for synchrotron radiation research and is not simply a bump on the funding profile.

The last Town Meeting and UEC meeting were held on the 16th and 17th of November. A wide range of issues were reported and discussed in each forum. Michael Hart recon firmed his support for beamline confederations or consortia, and emphasized the importance of allowing scientific disci plines (such as powder diffraction, EXAFS, and protein crys tallography) to define these consortia.

A new Science Advisory Committee (SAC) is being formed and is due to meet this spring to begin the process of tenure review of PRTs. I believe we should applaud the resur rection of the tenure review process to help the PRTs identify the strength and weakness in their programs.

Despite the difficulties that many users have experienced with the electronic submission program for experimental ab stracts, our next Activity Report will have slightly more ab stracts than the previous year. Many users believe that the re quirement of electronic submission was premature. As the tech nology matures and more users become familiar with the elec tronic submission program, some of these problems may be alleviated. Perhaps some flexibility of the NSLS by also ac cepting some paper (hardcopy) submissions will make this a more friendly process next year.

Ring reports demonstrate again the reliability we have come to expect during operations. Development efforts on the x-ray ring include the small gap undulator, EPW, improved digital feedback, and reduced horizontal emittance. While life time improvements and deve> lopment work toward "top-off"; operations on the UV ring continue.

The final topic reported at the Town Meeting was the current status of the Be window project. By now, the short comings of many presently installed Be windows on the X -Ray Ring are well known. Don Lynch outlined a plan that calls for analysis of the new design in early 1996, and a proto type built by June. Expectations are that the first 17 replace ments could be installed in the fall (currents to 350 mA), and the remaining replacements by the end of 1996. This should then permit operating currents above 425 mA.

There were several other topics discussed at the UEC Meeting on November 17. The PRT/General User

Study Panel has completed its survey of the PRTs. A preliminary look at the responses indicates that the short term health of the PRTs is generally not too bad. There seems to be confidence in short -term funding prospects and many PRTs are upgrading their beamlines. The consortia concept was well received by many but not all PRTs. A commonly cited need was for equipment pools and software standardization. A final draft report will be discussed at the February UEC Meeting with specific recom mendations to be provided to Michael Hart. The survey of the General User program is due to be completed in February.

The UEC discussed the operation fill schedule on X-Ray Ring at November meeting and recommended to change to 12 hour fills regardless of LEGS operations. The filling policy on the UV Ring will remain unchanged. Several requests prompted this discussion and lead to a careful re-examination of old sur vey results. The new filling schedule will raise average current in X-Ray Ring. With much more reliable injection, excellent orbit feedback control, and higher injection currents, most us ers should realize improvement to their operations.

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MARCH 1996 NSLS NEWSLETTER

Synchrotron Radiation in Medical Research at the NSLS

W. Thomlinson
NSLS Associate Chairman

In the relatively short time that synchrotrons have been available to the scientific community, their characteristic beams of UV and x-ray radiation have been applied to virtually all areas of medical science which use ionizing radiation. The ability to tune intense monochromatic beams over wide en ergy ranges clearly differentiates these sources from standard clinical and research tools. The tunable spectrum, high intrin sic collimation of the beams, polarization and intensity of the beams make possible in-vitro and in-vivo research and thera peutic programs not otherwise possible. From the beginning of research operation at the NSLS, many programs have been carrying out basic biomedical research. At first, the research was limited to in-vitro programs such as the x-ray microscope, circular dichroism, XAFS, protein crystallography, micro-to mography and fluorescence analysis. Later, as the coronary angiography program made plans to move its experimental phase from SSRL to the NSLS, it became clear that other in -vivo projects could also be carried out at the synchrotron. The development of SMERF (Synchrotron Medical Research Facility) on beamline X17 became the home not only for an giography but also for the MECT (Multiple Energy Computed Tomography) project for cerebral and vascular imaging. The high energy spectrum on X17 is necessary for the MRT (Microplanar Radiation Therapy) experiments. Experience with these programs and the existence of the Medical Pro grams Group at the NSLS led to the development of a pro gram in synchrotron based mammography. A recent adaptation of the angiography hardware has made it possible to im age human lungs (bronchography). Figure 1 schematically depicts the broad range of active programs at the NSLS.

The research covers a very wide range of physical di mensions: atoms and molecules (XAFS and protein crystal lography), chromosomes and cells (x-ray microscopy), tissues and organs (angiography, computed tomography, bronchog raphy and mammography). Another way to differentiate the programs is between those which are in-vitro and those which are in-vivo. Of course the object of much of the in-vitro work is to impact the applications of medicine in the in-vivo health care world. This brief Newsletter article will not address any of the in-vitro research programs, but rather it will summarize the current status of the in-vivo programs: MECT, mammography, coronary angiography and bronchography, MRT.

MULTIPLE ENERGY COMPUTED TOMOGRAPHY (MECT)

Monochromatic synchrotron x-ray beams have several advantages over the wide-energy band bremsstrahlung radiation obtained from x-ray tubes for computed tomography (CT). First, monochromatic x-rays do not undergo "beam hardening", an effect caused by high attenuation of the low energy end of the photon spectrum, resulting in an increase in the spectrum's mean energy as the beam penetrates the body, particularly the bone. The effect is particularly troublesome in slices with irregular bone shapes, such as the lower part of the skull. Second, for the same absorbed dose to the subject as conventional CT, monochromatic CT at the right beam energy for a given patient size has larger image photon count and, therefore, lower image noise because of the absence of the low energy tail of the spectrum. At the same time better image contrast resolution is obtained because the monochromatic beam lacks the high end of the polychromatic spectrum which is associated with low image contrast. Third, the 0.2-0.5 % energy bandwidth of the monochromatic beam, compared to about 50% for conventional CT, and the wide energy range of beam intensities allow efficient implementation of the energy-selective CT methods of dual-photon absorptiometry (DPA) and K-edge imaging of contrast elements. Dr. Avraham Dilmanian from the BNL Medical Department and his co-workers are developing such a CT system, called Multiple Energy Computed Tomography (MECT), at NSLS beamline X17B2 for imaging the human head and neck [1], [2]. MECT has a fixed horizontal beam and a subject chair rotating about a vertical axis. It uses a fixed exit Laue-Laue monochromator employing flat [Si] crystals, and a modular CdWO_4/PIN-diode linear array detector.

DPA will probably be MECT's main mode of research application in the study of carotid artery atherosclerotic plaque composition. DPA separates the tissue into two components of low-Z and intermediate-Z, which allows characterization of the plaque in terms of lipid/cholesterol, collagenous (fibrous), and calcified tissues. DPA involves acquiring two images at greater than 40 keV and greater than 100 keV, and analyzing them to produce the low-Z and the intermediate-Z images. It is currently used in clinical diagnostics and research, in both the planar and the CT mode, mostly for bone densitometry. DPA's current use in conjunction with conventional CT is called Dual-Energy Quantitative CT (DEQCT). MECT DPA should also allow soft tissue characterization, where DEQCT has a poor performance.

MECT will be used in contrast imaging with I or Gd in a method called K-edge imaging, which involves tuning the beam energy immediately above the contrast element's K-edge. The method provides a 2-fold or more advantage in image contrast, depending on the contrast element and the polychromatic spectrum with which the comparison is made. MECT will be used for CT Angiography, combining K-edge imaging and the "Helical" CT mode.

MECT is at its preclinical stage, carrying out the imaging experiments in SMERF where it is possible to achieve the final field-of-view of 19.5 cm. Its recent images of phantoms and a live rabbit show a 2-fold smaller image noise than conventional CT for the same spatial resolution and dose. They also show a 2-fold larger image contrast in I K-edge imaging. A new bent Laue-Laue monochromator is under construction. MECT's clinical system will allow DPA at 40 and 100 keV, and K-edge Angiography with I and Gd. It will be used in the Helical CT mode with a 5 s/revolution chair speed, and 1 - 5 mm slice height. Figures 2a and 2b show a rabbit lower head imaged by MECT and by conventional CT, respectively. The MECT image was obtained in X17B1 at 43 keV, 2 mm slice height, and 6 cGy skin dose, while the conventional CT image was taken at 80 kVp and 800 mA (4 cGy), and 3 mm slice height. The two images have the same dose times slice height, which is an index for comparing image photon counts. The MECT image shows a better contrast resolution, apparent from its separation of muscle and subcutaneous fat. The dark bands in the conventional CT image along the continuation of the rabbit's jaws are most likely beam hardening artifacts.

MAMMOGRAPHY

Screening mammography has proven to be an effective procedure in identifying early breast cancer. The cancers found by mammography tend to be smaller and less advanced than those found by physical examination, resulting in better survival rates. Mammographic technology has dramatically improved in the last two decades, but approximately 10% of clinically obvious breast cancers are not visible with mammography. Further improvement in detection is expected with the advent of digital mammography which utilizes better source geometry and improved detector systems. It has been suggested very frequently that perhaps the use of the synchrotron source with its inherently highly collimated, tunable radiation could increase the signal to noise and increase the contrast resolution in the images, possibly at lower dose to the patient.

Over the past year, a set of preliminary experiments has been done by Dr. R. Eugene Johnston from the University of North Carolina and his collaborators using monoenergetic x -rays to explore the potential of monoenergetic photons for mammographic imaging [3]. The experiments done on the X27 beamline have shown that superior image contrast can be obtained relative to the conventional film-screen techniques. As an example of the results, Figure 3 shows a comparison between contrast measured in a contrast detail phantom at 18 keV and the same data obtained on a conventional system. Images of various mammographic phantoms and real tissue have been carried out in the energy range 16 to 24 keV.

In these early experiments, it was clear that improved contrast at equivalent or less dose is obtained. Scoring of the phantom images according to American College of Radiology criteria shows improvement over the conventional systems, with similar or less mean glandular dose. The future plans include optimization of the monochromator, sample orientation and translation system, and detector. The early work at the NSLS has utilized available image plate and conventional mammographic film detectors. It is planned to study digital detector systems, scanning monochromators and imaging technology incorporating an analyzer crystal to obtain scatter free images. The elimination of scatter is expected to produce images with higher contrast than conventional imaging systems. A preliminary experiment has been reported by Chapman, *et al*. at the NSLS [4]. Direct comparisons between the synchrotron system and conventional systems will continue to be made using tissue samples obtained from patient specimens containing different types of cancers (masses, calcifications, and architectural distortions). In the long term, it may be possible to advance the program to human studies in the medical research facility at the NSLS.

CORONARY ANGIOGRAPHY

The most advanced of the applied medical research programs at synchrotron facilities are those doing human coronary angiography. The field traces its origins back to the proposal by Rubenstein, *et al.* that the intensity of the synchrotron x-ray beams would be high enough to allow imaging of the coronary arteries following venous injection of an iodine containing contrast agent [5]].

The reason such a procedure is desirable is that the standard arterial catheterization method (contrast agent injected directly into the coronary arteries) presents significant enough risks that it is not used for clinical screening or research even though the images obtained are excellent. For coronary artery disease research to use humans subjects for imaging, the venous technique is highly desirable. Using conventional sources, this method proved to be a failure due to motion artifacts in the images and not enough flux to allow sufficient contrast to image the small arteries containing highly diluted contrast agent overlying the large coronary structures. Even applying digital subtraction imaging at the iodine K-absorption edge proved a failure with conventional sources.

The concept of synchrotron based coronary angiography was first developed at Stanford University and the early human studies were done at the Stanford Synchrotron Radiation Laboratory [6], [7]. In 1989 the project moved to the NSLS where the hardware was installed in the SMERF medical research facility [8], [9], [10]. The NSLS program has been a collaboration between Stanford University, North Shore University Hospital and SUNY Stony Brook. Thus far a total of 28 patients have been imaged, 7 at SSRL and 21 at the NSLS.

The technique takes advantage of the wide horizontal, narrow vertical beam profile from the superconducting wiggler on X17. The patient is aligned and then translated vertically through the cross-over point of two fan beams, one above the iodine K-edge energy and the other below the edge. An iodinated contrast agent is injected into the Superior Vena Cava, the large vein near the entrance to the heart. After a delay time for the iodine to reach the coronary arteries, the image scan is started. As the patient moves through the beams, successive lines of data are taken, with each line of data taken in 4 milliseconds with a complete image built up from 256 lines. Spatial resolution is either 0.25 or 0.5 mm. Excellent images of the right coronary artery (RCA) and of the left anterior descending coronary artery have been obtained. The circumflex artery has been more difficult to image, but can be seen in particular patients with the proper angulation for the image. Figure 4 is an example of an intravenous angiogram taken at the NSLS showing the full RCA.

In Germany, Dr. Rainer Dix and his co-workers at HASYLAB have made two major advances in this technology which will soon be implemented at the NSLS as well. They have developed a system by which the images are gated from the patient's ECG signal. In addition, they have shown that the contrast agent can be injected into a peripheral vein [11], [12] and result in images comparable to those with injection near the heart.

The recent work at the NSLS has centered on determining the optimal projection angle for studying each artery. These studies, along with the demonstrated gating of the images from the ECG and peripheral injection in Germany, have advanced the technology to a point where definitive medical research can begin. A workshop at HASYLAB in October 1995 demonstrated that the synchrotron technology is presently well ahead of both MRI and ultra-fast CT for coronary artery imaging. Based on the very positive outcome of that workshop, HASYLAB is undertaking a major program to validate the synchrotron angiography against the standard arterial procedures. At Brookhaven National Laboratory, discussions are underway to assess the future of angiography at the NSLS. Independent of the outcome of those deliberations, the project is continuing to commission an advanced low noise image acquisition and display system which will lead to increased contrast in the images [13].

BRONCHOGRAPHY

Recently, Rubenstein *et al*. have described a medical imaging procedure using xenon as a contrast agent for K-edge dichromography of the respiratory air passages [14]. The process could provide the opportunity to image anatomic structures and pathologic processes that cannot be visualized by conventional x-ray based imaging methods. For example, detection of lung cancer, the leading cause of cancer related deaths in the US, is an important application. At present, standard x -ray procedures cannot detect tumors less than 1 cm in diameter. It has been calculated that synchrotron imaging with xenon could detect significantly smaller, earlier tumors leading to enhanced five-year survival. For the synchrotron bronchography, the airway structures are imaged after inhalation of a gas mixture containing stable xenon. The amount of inhaled gas is limited to the anatomic dead space volume of the upper and lower air passages. The subjects hold their breath for several seconds while the images are recorded using the dual-energy imaging system developed for coronary angiography. Initial studies on human volunteers have been carried out at the NSLS in a recent experiment [15]. For these studies, the X17 beamline was aligned to bracket the xenon K-edge at 34.56 keV. The procedure was identical to the angiography imaging except that the contrast agent was inhaled instead of being injected. The results are very promising and additional volunteer imaging experiments are scheduled.

RADIOTHERAPY

Radiotherapy is that process whereby ionizing radiation (gamma-rays, x-rays, or charged particles) are targeted to a tumor in order to kill or retard growth of cells. The dose can be delivered by external beams generated by machines or by the decay of radioactive sources such as Co-60. The dose can also be delivered by use of radioisotopes which are transported to the tumor site pharmacologically via antibodies or chelators. Some radioisotopes are encapsulated and inserted surgically or by stereotaxic injection into the region of the tumor. Radiotherapy is limited by the radiation dose tolerance of normal tissues in the region of the tumor or, in the case of external beam therapy, of normal tissues between the skin and the tumor and, often, beyond the tumor.

There are two ways synchrotron radiation may be advantageous in radiotherapy. The first is to increase the sensitivity of the target tumor cells to the radiation, leading to increased death of the cells while sparing normal cells. The tunability and monochromaticity of the synchrotron radiation makes this approach possible. An example of an application in the field of radiobiology is Photon Activation Therapy (PAT) [16]. The second approach [17], is to use the inherent collimation of the synchrotron beams to create a beam geometry that optimizes dose delivery to the tumor site and effectively avoids damaging the intervening normal tissue.

One of the most effective means of increasing the dose to the tumor and sparing intervening normal tissue is to use stereotactic radiosurgery. In that procedure one or more highly collimated radiation beams are directed at the tumor from varying directions. The crossing point of the beams is at the target tumor, thereby delivering a dose equal to the sum of all the beams to the target and delivering a fraction to all other tissue.

Synchrotron radiation beams can be very highly collimated in either planar or cylindrical beam geometry and can be either focusing or non-focusing. With the development of high energy sources it is now possible to have beams with energies in the range of 50 keV and above. Microbeam Radiation Therapy (MRT) is a concept developed at BNL by which a lesion is irradiated in a stereotactic fashion using bundles of multiple, parallel, microscopically narrow beams of x-rays [18], [19], [20], [21]. The energy range required is 50-150 keV. The micro beams are planes several millimeters high and 25-75 mm wide. The beams in each bundle are separated by 75-200 mm on center. The detailed spacing and beam widths are determined by experimentation and by Monte Carlo photon- and electron -transport simulations. The central phenomenon is that endot helium and other kinds of vital self-renewing cell systems that are destroyed by high absorbed doses within the paths of mi crobeams regenerate from similar cells in the minimally irra diated contiguous segments between the microbeams. Tissue necrosis is thus avoided except in the crossfired zone (a super position of parallelepipeds), where the tissue-sparing effects of the microbeams are eliminated.

Experiments have been carried out at the NSLS in which it has been shown that MRT is effective in increasing the survival of rats with imminently lethal brain tumors [20], [21]. In these experiments, the beams were 4 mm high and 25 microns wide, filtered by Gd on the X17 beamline at the NSLS. The microplanar beams were separated by 100 micron intervals, center to center. The skin entrance dose rates were about 400 Gy/s. The present efforts at the NSLS and in Grenoble at the ESRF [22] are continuing both experimentally and theoreti cally in order to understand optimal beam parameters for MRT and to study dose distributions theoretically.

CONCLUSIONS

The projects discussed in this article are, for the most part, still in their infancies and no one can predict the direction in which they will develop. Both the basic research and applied medical programs are sure to be advanced at the new facilities coming on line, especially the ESRF and SPring-8. However, success is not guaranteed. There is a lot of competi tion from advances in conventional imaging with the develop ment of digital angiography, computed tomography and func tional magnetic resonance. The synchrotron programs will haveto provide significant advantages over these modalities in order to be accepted by physicians and their patients. Advances in image processing and the development of compact sources [13], [23] will be required in order to move the synchrotron developed technologies into the clinical world. In any event, it can be expected that the images produced at synchrotrons will establish "gold standards" to be targeted by conventional mo dalities. More work needs to be done to bring synchrotron radiation therapy and surgery to the level of human studies and, subsequently, to clinical applications.

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Infrared Studies of Single Crystal C_60 : Silent and Higher Order Modes

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Carbon, one of the most abundant elements on earth, has been known for a very long time to exist in only two crystalline forms: graphite and diamond. However, within the past ten years a third form of carbon has been discovered: Fullerenes [1]. And after learning how to make and purify fullerenes in quantity [2], these novel cages of carbon have become quite heavily studied.

One of the most attractive features of fullerenes is their high symmetry. C_60 , the most common fullerene, has the shape of a truncated icosahedron with *I_h* point group symmetry (Figure 1). This high symmetry can be exploited to predict the vibrational modes andelectronic molecular orbitals of the molecule. While a 60 atom molecule will in general have up to 174 vibrational modes (3 x 60 - 3 translational - 3 rotational = 174), the *I_h* molecular symmetry causes many to be degenerate. The symmetries within Ih are

$$I_h = 2A_g \oplus 1A_u \oplus 3F_{1g} \oplus 4F_{1u} \oplus 4F_{2g} \oplus 5F_{2u} \oplus 6G_g \oplus 6G_u \oplus 8H_g \oplus 7H_u \ ,$$

a total of 46 modes. The capital letters denote the degeneracy of the mode (*A* is singly degenerate, *B* is two-fold degenerate, *F* is 3-fold, *G* is 4-fold, and *H* is 5-fold) and the subscripts are *g* for *gerade* or even symmetry and *u* for *ungerade* or odd symmetry modes. Of these 46 only 10 are Raman-active (2 *A_g* and 8 *H_g*) and 4 are infrared-active (4 *F_1u*). These four *F_1u* modes were experimentally observed [2] at 527, 576, 1182, and 1438cm^-1.

High-purity single crystals of C_60 were grown using a vapor transport method [3], and had sizes of approximately 1 x 1 x 0.5 mm. These crystals were then measured in the mid-IR (500-4000cm^-1) at the U2B beamline and in the far-IR (50 - 600cm^-1) using the U4IR beamline. Some crystals were placed inside a diamond anvil high pressure cell which was placed at the IR beam focus for measurements under pressure. These two beamlines' high IR brightness made good resolution measurements quick and easy to obtain. We present the infrared transmission data on a C_60 single crystal at 300 and 77K in Figure 2. There are approximately 150 vibrational mode absorptions visible in the room temperature spectrum and many of the modes have a fine structure at low T. The four *F_1u* modes are seen to be so strong that they saturate with zero transmission in a range around the usual positions enumerated above. All the other modes are approximately a factor of 100 times weaker than the 4 IR-active modes, and are now visible because of the significant thickness of the C_60 crystal. A similar set of weak vibrational modes was observed in Raman spectroscopy using a high laser power [5].

We looked for any sign of contaminants such as solvents, C_70 and C_60 O, but do not find evidence for any of these based on their known spectral features.

Many of the known Raman vibrational modes can readily be identified in our infrared spectra using their well known positions:
H_g (2) at 431cm^-1 , *H_g* (3)at 709cm^-1, *H_g* (4)at 775cm^-1,*H_g* (5) at 1102cm^-1, *H_g* (8) around 1576cm^-1, and *A_g* (2)at 1470cm-1. *H_g* (7) at 1425cm^-1 might also be active, but it is hidden under the extremely strong *F_1u* (4) mode nearby. *H_g* (1), *H_g* (6), and *A_g* (1) do not appear in our IR spectra.

The appearance of the Raman lines in the IR spectra strongly suggests that other IR-inactive resonances could appear in the spectra as well. However, the total number of lines far exceeds the number of vibrational modes, and the extension of the spectral features up to about 3200cm^-1 indicates that combination modes are also present. Combination modes are weakly active in all molecular systems due to theanharmonicity of the bonds (they are not simple springs). This causes resonances at the sums and differences of two fundamental frequencies. Again group theory can be used to understand which combinations will be IR-active or Raman-active[3]; out of 2116 total possible combinations, 380 are IR-allowed and 484 are Raman-allowed. The binary combinations (involving two fundamentals) are expected to be stronger than third or higher order combinations. Indeed, the apparent absence of significant features above 3200cm^-1 is in good agreement with the expected highest frequency of ~1600cm^-1 for the fundamental modes [4].

The energy of a particular combination mode *v*_1 *v*_2 is generally expected to be about E(*v*_1) ± E(*v*_2) (where E(*v*) is the energy of mode *v*). The Bose factors for fundamentals at *v*_1 and *v*_2 , n_1 and n_2 , respectively, appear in the intensity of the sum mode at E(*v*_1) + E(*v*_2) as 1 + n_1 + n_2 , whereas for the difference mode E(*v*_1) - E(*v*_2) the intensity is |n_1 - n_2 |. Consequently, for the wavenumber range studied, the intensity of the difference modes should exhibit a strong decrease with temperature. None of the lines exhibit this behavior, therefore we must conclude that all of the resonances seen in our measurement are either fundamentals or should be close to the sum of two fundamental frequencies.

In many cases a broader high temperature line splits to several sharper low temperature resonances. This is illustrated in Figure 3, where spectra at several temperatures are plotted over an expanded scale for some particularly interesting frequency ranges. In the two middle panels of Figure 3 a few lines where the behavior is non-typical, in the sense that the oscillator strength seems to increase dramatically below the phase transition, are also pointed out. These lines were either very broad, or forbidden at high temperature. The significance of these resonances will be discussed later.

We used the group theoretical results of which combination modes are allowed together with the observed weakly active fundamental vibrations to come up with an assignment for all 46 vibrational modes [3]. This assignment enabled us to account for every observed vibrational mode in Figure 2, in the Raman higher-order data[5], and in agreement with neutron time-of-flight measurements [6]. The fact that we have observed Raman lines becoming weakly IR-active implies that there is a symmetry breaking occurring in the crystal; a given C_60 molecule no longer has its full *I_h* symmetry. Several possible symmetry breaking mechanisms exist:

- Isotopic Impurities: ^(13)C is present in about 1.1% of natural carbon meaning that approximately 1/2 of all fullerenes will have one or more ^(13)C atoms, greatly reducing that molecule's symmetry.
- Solid State Effects: Each C_60 molecule is at an FCC crystal site which imposes an additional FCC site symmetry on it. Other:
- Dislocations, foreign impurities, and surface effects can all change symmetries.

We can tackle the last item first. Since these are large single crystals the surfaces are a very small amount of the volume being probed and if they were the cause of spectral features, thin film measurements should have shown the same modes (they do not). Furthermore the intensity of all weakly-active features scales with the volume of the crystal. Random impurities and dislocations can be ruled out because of the detailed confirmation of our spectra shown in Figure 2 obtained on samples grown by several groups around the world [7], [8]

Isotopic activation of previously silent modes is possible to test. We obtained a sample that was grown from 8% enriched ^(13)C fullerenes and repeated the measurements [9]. The spectra obtained were quite similar to the ones obtained in Figure 2. The relative intensities of each feature did not significantly change, and each mode was broadened somewhat. These effects could be well modeled by simply assuming the known mass distribution and applying this broadening function point-by-point to the natural abundance C_60 spectrum. Furthermore a theoretical model of C_60 was used to calculate the IR spectrum with 1.1% and 8% probability of any atom being a ^(13)C [9]. The resultant calculated spectrum is displayed in Figure 4 and shows firstly that the modes activated by this mechanism are approximately 10^4 times weaker than the allowed *F_1u* modes whereas our measurements find modes only 10^2 times weaker implying that the true symmetry breaking is significantly stronger than that due to the addition of a proton. Secondly, the calculation shows that indeed the intensity of each weakly active mode should be enhanced by an average factor of 4.45, which we do not observe. We can therefore conclude that while ^(13)C may cause a symmetry-lowering, this is a much weaker effect than we are observing.

Finally that leaves us with the effects of the fullerene molecules being located in a crystalline solid. The FCC site symmetry that is imposed on a C_60 will reduce the symmetry, however in all cases it will retain the inversion symmetry. This means that *gerade* and *ungerade* modes will retain their even and odd characters, and therefore Raman modes will not be active in the infrared as well as the reverse. Since we clearly do observe Raman modes in Figure 2, this again seems to not be our symmetry-breaking mechanism. Furthermore, in the high temperature state (greater than 250K) the fullerenes are freely rotating which would "smear out" any solid state effects. If on the other hand the vibrational states were sensitive to the neighboring molecules on a much faster time scale than the rate of rotation, each molecule would see a "snapshot" of its neighbors having essentially random orientations. In this case the site symmetry will be much lower than FCC and the inversion symmetry would no longer hold.

To explore if these "snapshot" ideas are viable, we developed a model system where we could calculate what the relevant time scales are [10]. We found that activation of symmetry-forbidden modes indeed does occur as long as the lifetime of the vibrational mode, as deduced from its width, is shorter than the rate of motion of the molecules. We have termed this effect "motional diminishing", in analogy with motional narrowing in NMR, since the result of increasing the motion will remove symmetry lowerings and forbidden vibrational modes will again be disallowed (they will diminish).

To apply these ideas to the present case, we need to know the natural lifetime of a particular vibrational mode, and the correlation time of the fluctuations of the neighboring fullerenes. The lifetimes of the weakly active fundamentals can be inferred from measured line widths, approximately 3 - 6cm^-1, corresponding to lifetimes of 5 - 10psec. The correlation time is related to the rotational diffusion constant which was masured by neutron scattering measurements to be *D_R* = 1.4 x 1010sec^-1 at T=260K [11], just above the rotational phase transition. Since we have observed forbidden modes at this temperature, the correlation time must be greater than 10psec, which corresponds to a rotation by a C_60 by an angle of greater than 5 degrees.

As the temperature is raised, the rate of rotation of the fullerenes will increase. However the lifetime of the vibrational modes will also decrease since in insulators the primary channel of phonon decay is due to the interaction between phonons and the enhanced population of phonons at higher temperatures will lead to shorter lifetimes. Therefore as the temperature is further increased the "forbidden" lines should either

1. motionally diminish (if the lifetime of the resonance has a weaker temperature dependence than the rotational motion), or
2. the integrated intensities will not change while the line widths increase (the mode lifetime remains shorter than the fluctuation time at all temperatures).

To test these predictions we measured the IR spectrum of a C_60 crystal at elevated temperatures. All observed modes do broaden without a significant change in integrated intensities up to a temperature of 650K.

Motional diminishing can, however, be observed in a few narrow lines which are only active in the low temperature (less than 250K) state. Figure 3 demonstrates such modes at 1566, 1314cm^-1 (middle panels with arrows) and four very sharp modes in the 750 - 765cm^-1 region. These modes are active in the low temperature state where rotation is inhibited, then completely disappear just above the rotational transition temperature. The linewidths of these modes, 0.7 - 1.5cm^-1, are narrower (and therefore they have longer lifetimes) than the other weakly active fundamentals which persist to higher temperatures. We can now establish an experimental limit on the correlation time *tau* between neighboring C_60 molecules in the rotating state: *tau*between 10psec and 20psec. Combining these times with the measured rate of rotation [11] we find they correspond to rotation angles of between 5 and 10 degrees.

The merohedral disorder observed in the low temperature phase suggests that fullerene [12] molecules are sensitive to whether a neighboring ball has a bond or a pentagon or hexagon center facing it. The amount of rotation needed to move between these two arrangements is approximately 15 degrees so it is reasonable that a 10 degree rotation will significantly change the crystal field perturbations being applied to a fullerene molecule.

To conclude, we have shown how experiments at the NSLS have led to the discovery of weak symmetry breaking in the IR spectra of C_60 single crystals. In addition, this molecule is found to be a complex system where second and higher order combination modes can be studied. In trying to elucidate the mechanism of the weak symmetry breaking we first ruled out the most obvious symmetry lowering due to ^(13)C atoms within individual C_60 molecules. The effective crystalline environment was found to be much more disordered than initially thought, making fullerite above the rotational transition temperature a plastic crystal system. The lifetime of nearly all fundamental vibrational modes turns out to be shorter than the rotational diffusion time constant and therefore the molecules "see" a static, disordered configuration of neighbors giving rise to the symmetry breaking which is observed. A few fundamental modes are found to exhibit the newly understood motional diminishing phenomena.

This work was done in collaboration with Laszlo Mihaly, Xiaoqun Du, John Kwon, Jaroslav Fabian (SUNY @ Stony Brook), J. Goddard (Orsay, France), P. Bernier and J.M. Lambert (Montpellier, France). Support was primarily by NSF grant DMR9202528, and also by NSF grant DMR9118414 (J.F.) and from CNRS through the Groupe de Recherche 1019 (J.G, P.B, J.M.L.). The NSLS is supported by the U.S. Department of Energy, under contract DE-AC02-76CH00016. [1] H.W. Kroto, J.R. Leath, S.C. O'Brien, R.F. Curl, and R.E. Smalley, Nature 318, 162, (1985).

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MARCH 1996 NSLS NEWSLETTER

FOCUS ON..... NSLS VACUUM GROUP

Conrad Foerster, NSLS Vacuum Group Leader

Click here to see a photograph of the Vacuum Group.

"Much ado about nothing", a popular title from English literature, has been quoted often to vacuum technologists relative to our work. In the real world there is much to do to obtain the nothingness of a good vacuum! There is even more to achieving the good ultra-high vacuum (UHV) needed for the NSLS storage rings. Today's vacuum technologist must be familiar with a large variety of pumps, gauges, chambers, materials, processes, valves, controls, diagnostics, etc., related to vacuum. A practical example would be to measure, find, and fix a system vacuum leak - a task easily compared to finding a needle in a haystack. A good knowledge of vacuum systems and skill with mass spectrometers is required for success. The vacuum technologist must also be familiar with the workings of the entire Light Source and its beamlines in order to resolve vacuum issues and to ensure smooth operation.

The ongoing mission of the NSLS Vacuum Group is to provide the best possible vacuum for the operation of the Light Source. Good vacuum is one of several main ingredients for good stored beam lifetime; without it, the many collisions between electrons and gas molecules in poor vacuum would result in short lifetimes and excessive radiation. The Vacuum Group is a wing of the Mechanical Engineering Section. Members of the group are: Conrad Foerster (Group Leader) Walter deBoer (Technical Supervisor), Peter deToll (Assistant Technical Supervisor), Chris Lanni (Technical Associate), with Technical Specialists Mike Caruso, Norman Cernyar, Richard Freudenberg, and Malry Tardd.

Along with the Operations Group, we continually monitor beamline front ends in both rings to ensure that one beamline problem does not affect an entire ring. Pressure and partial pressure requirements must be met to open a beamline to VUV or the X-Ray Ring. The rings and front ends are continually monitored with residual gas analyzers to protect beamline optical components from hydrocarbon and other contaminants. Clean dry nitrogen is used for controlled vent up of vacuum sections when necessary and pressure interlocked portable turbo molecular pumping stations are used to recover vacuum. Special procedures have been developed by the Vacuum Group for bake out, argon plasma cleaning and conditioning of parts and assemblies for installation in the rings or beamlines. These procedures were used during the recent VUV shutdown where one quarter of the bending chambers were replaced to add new IR beam lines. Without these procedures quick recovery of the stored beam lifetime would have been impossible.

In addition to the NSLS storage rings, booster and injection vacuum systems, and front-ends, the Vacuum Group works on other projects. We have been involved in vacuum R&D programs for the Advanced Light Source, The Advanced Photon Source, B-meson Factory, Daphne, LHC and SSC, UHV beam chambers. Measurements of accelerator construction materials were studied and measured on beamlines U10B and X28A. The measurements involved photon interaction with internal vacuum surfaces. This photon interaction causes the pressure to rise which limits the stored electron beam lifetime. In addition to measuring internal vacuum surfaces, we have coated internal surfaces with various metals, for example, ceramic beam chambers.

The Vacuum Group currently operates the NSLS cleaning facility and will continue to do so umtil a new cleaning facility, scheduled to open this summer and managed by the BNL Central Shops, is in operation. To help support the present facility we clean vacuum parts for other BNL Departments such as Physics, AGS, RHIC, and Central Shops. New cleaning procedures which have been evaluated and tested for NSLS UHV will be incorporated in the Central Shops facility.

The Vacuum Group interfaces with the NSLS staff and User community to resolve vacuum issues or problems. We routinely Helium leak test vacuum parts and assemblies for NSLS staff and Users. In addition, time and schedule permitting, we rebuild various types of vacuum pumps and electronics for ourselves and NSLS users. Included are: mechanical pumps, Balzer turbo molecular pumps Varian ion pumps, and Perkin Elmer ion pumps and controllers.

The Vacuum Group is located in the basement of Bldg. 535 on the east side. If you have a question or need assistance with vacuum, call us on extension 4754 or 7168.

MARCH 1996 NSLS NEWSLETTER

National Industrial Users Group

Richard Harlow (DuPont)

The National Industrial Users Group is a newly formed organization consisting of industrial synchrotron and neutron users, representatives of the National User Facilities, academ ics who have industrial interests, and independent consultants. Its formation was the product of an NSLS Industrial Users Workshop held at Brookhaven on Dec. 5, 1995. The Workshop was organized by Dick Harlow (DuPont)and Jerry Hastings (NSLS) and was attended by some 50 people, roughly split between the industrial users and representatives of the NSLS and other synchrotron/neutron facilities. The mission of the workshop was to explore a wide variety of industrial user conserns and, where possible, to explore possible solutions to the problems that presently limit industrial usage of the NSLS. The issues raised at the workshop were more varied and com plex than could possibly be resolved at this one meeting and so it was felt that a national organization of industrial users should be formed to bring issues of industrial importance to the attention of the user facilities on a continuing basis. Notes from the workshop covering such topics as proprietary vs non-propri etary experiments, liability of industrial members of PRT's, instruments dedicated to industrially important methods of analyses, cooperative PRT's, etc. are available by e-mail from "harlow@esvax.dnet. dupont.com", or here.

The industrial users are also in the process of forming an Industrial User Special Interest Group at the NSLS to bring our issues to the attention of the UEC. The group will soon have its own WEB page (on the NSLS home page, under Users' Organization) which will communicate information on industrial concerns, future meetings, and list job openings and potential synchrotron/neutron consultants who specialize in various analytical techniques. The industrial users will prob ably have their first follow-up meeting during the NSLS Users' Meeting in May. Anyone who is interested in the meeting and/or interested generally in the activities of the industrial users can send a note to Dick Harlow at the above e-mail address.

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MARCH 1996 NSLS NEWSLETTER

X-Ray Status

Roger Klaffky, X-Ray Ring Manager

There has been considerable activity on the X13 straight section over the last year. During the December 1994 shutdown, the NSLS Prototype Small Gap Undulator (PSGU) was removed so that vacuum chamber flanges could be re-machined to reduce the minimum electron beam aperture from 3.8 mm to 1.0 mm. The Elliptically Polarized Wiggler (EPW) was installed in the X13 straight section during this same shutdown. The EPW began operation at 2 Hz in April after studies shifts had demonstrated that it had a negligilble effect on users' beam. During the May '95 shutdown trim magnets were installed to better compensate for vertical orbit motion caused by the EPW. The PSGU was reinstalled during the May '95 shutdown and, in August, the photon spectrum showed that the design goal was achieved: a 2.5 keV fundamental, and a 7.5 keV third harmonic for a 2.5 mm electron beam aperture and a 5.6 mm magnet ap. The partial lifetime contribution was 36 hours. Shortly afterwards, the PSGU began operations at a 4.0 mm electron beam aperture and 7.5 mm magnet gap. Due to the reduction in beam lifetime at gaps less than 4mm, any operation at less than 4 mm will require approval by X-ray users.

The maximum operational current of the X-Ray Ring increased from 250 mA to 300 mA at the end of June 1995. In order for operations to occur at currents above 300 mA for all beamlines, a sizeable number of beryllium windows will have to be replaced. Operation at 438 mA requires replacement of a total of 34 additional windows. A new beryllium window design effort was underway in FY 1995 with the goal of installation of 17 new windows during the December 1996 shutdown, which is halfway to the number required for 438 mA operation and more than required for 350 mA operation. Operations at 350 mA would commence in January 1997.

There was progress in further stabilizing the electron beam position and size during FY 1995. In late '94 the global feedback gain was increased by a factor of 3 at low frequencies and the bandwidth was increased from 60 Hz to 200 Hz. To avoid changes in horizontal and vertical beam size occurring at specific XRF2 tuner positions (i.e. higher order modes) during a fill, the XRF2 temperature ramp was adjusted. The XRF3 tuner temperature was adjusted so that its resonance occurred during the first 1/2 hour after the fill which is the time that orbit corrections are made. During the December 1995 shutdown, a change in the XRF2 damping antennae configuration successfully removed the effects of the higher order modes. Changes in several load resistors on the XRF3 antennae resulted in a several-fold decrease in the beam size and orbit changes occuring at the resonance position. To track these beam size changes, the X28 profile monitor Spiracon was interfaced to the NSLS computer system to keep a daily history of beam size and position.

To enable high current operations at the end of June, the X-Ray Ring active interlock beam position monitor electronics were also upgraded. The AGC dynamic range was extended from 35 dB to 55 dB to allow prefill tests at 7 mA and to permit operations up to 434 mA. In the future, the improved electronics will be installed on the other ring bpms not in the active interlock system.

There have been several successful trial runs of the digital orbit feedback system in the horizontal plane. The sampling frequency of the orbit micro was increased from 60 Hz to 500 Hz to allow the digital feedback system to work. The higher sampling frequency has proven useful in diagnosing rapid orbit motion problems.

There were a number of steps taken to reduce downtime. A significant improvement resulted from running the RF systems with gap voltages adjusted so that if one system drops out, the three remaining systems can carry the beam load (up to 280 ma) without a beam dump. Also, the X-Ray Ring, VUV Ring, and Linac/Booster vacuum systems were placed on emergency power.

Considerable progress in the reliability of the NSLS water and compressed air systems was achieved by adding redundancy, and improving control and servicability. During the December '94 shutdown, a third High Pressure Copper System water pump was installed allowing the pumps to operate within their design specifications. Pressure control was added to this system to maintain constant flow. The addition of cooling and compressed air from the BNL Central Chilled Water Facility provided needed reduncancy to the NSLS chillers and air compressors. The four NSLS chillers and air compressors have stood alone when the CCWF was down for maintenance/repair. Digital proportional integral controllers have significantly improved temperature control. Temperature jumps of 8 degrees F in the Low Pressure Copper water after an X-Ray dump or fill were reduced to 1 degree. Additionally, the rms temperature changes in the water systems during stored beam conditions were reduced by a factor of 3 to 5. There was an ongoing effort to improve the serviceability of the proteus flow rate monitors by moving the proteus units from under the ring magnets and redesigning the plumbing to maintain a constant pressure boundary during testing and maintenance. To better track failure rates and pinpoint possible problems areas, there was implementation of color coding (according to time period of installation) of water and air hoses, and of a air solenoid inventory.

MARCH 1996 NSLS NEWSLETTER

CALL FOR GENERAL USER PROPOSALS

Deadline for proposals and requests for beam time on the NSLS X-Ray and VUV Rings is Friday May 31, 1996 for scheduling September through December 1996

Prior to Submitting a Proposal

> You must contact the beamline personnel responsible for the beamline(s) selected in order to verify technical feasibility on the beamline(s) and discuss any special arrangements for equipment. Your chance of getting beam time is improved by being able to use more than one beamline.

Preparing Your Proposal

> The same form is used for new proposals and for beam time requests against existing proposals. Follow the instructions on the proposal information sheet. All information must be typed or printed legibly. Be sure all of the required sections are completed and submitted at the same time. MAIL OR FAX ONE COPY of the proposal form, Safety Approval Form, and any attachments to the NSLS User Administration Office. Only one copy is required - do not mail a hard copy or fax a second if you have already faxed one.

Proposal Deadline

> The complete proposal package must be received by the User Administration Office on or before 5:00 pm Eastern Time Friday May 31 in order to be considered for the January - April cycle. The fax machine is always extremely busy on the deadline date; please do not rely on faxing the proposal successfully on May 31. We encourage submitting new proposals by mail prior to the deadline. Beam time requests for active proposals will be accepted after the deadline, but will be allocated beam time only after requests received on time have been allocated. Late requests are not eligible for a rating upgrade if beam time could not be allocated to them.
>
> Each proposal will receive a prompt preliminary review to verify that it is complete and legible. If there is a problem with the proposal, you will be contacted immediately. Submitting your proposal well in advance of the deadline date assures that the User Administration Office has time to reach you and that you will have enough time to correct any deficiencies.

Additional Information and Forms

> Blank proposal forms and instructions, a guide to the NSLS beamlines, and more information about the General User Program are available by contacting the General User Program Coordinator. Office hours are Monday through Friday, 8:00 am to 5:00 pm Eastern Time.

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MARCH 1996 NSLS NEWSLETTER

NEW X-RAY RING FILL POLICY

Approved at the X-Ray Users' Meeting on February 15, 1996:

- Scheduled fills will be maintained at 12-hour intervals, scheduled at 7:00 am and 7:00pm (0700 and 1900 hours).
- If the X-Ray Ring has been filled less than 6 hours before a regular fill time, skip the next scheduled fill.
- If the beam dumps early in a 12-hour fill period and is back up with more than 6 hours remaining before the next scheduled fill, beam will bea dumped at the regularly scheduled time for re-injection.

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MARCH 1996 NSLS NEWSLETTER

END-OF-RUN SUMMARY

Please fill out and turn in your end-of-run summary to the User Administration Office; the NSLS needs feedback on how well we are answering user needs during your experimental runs. Comments, both positive and negative, and suggestions are encouraged! The form is available from the User Administration Office, the NSLS Lobby, or the NSLS Home Page.

NSLS Home Page............ BNL Home Page............

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MARCH 1996 NSLS NEWSLETTER

NEW BNL PHONE SYSTEM

The new system was put in place over December and January of 1995. In the BNL telephone numbers, the 282 and 341 prefixes have been replaced with 344. Effective February 1, the old numbers were retired and any callers using those numbers are now being told to reach their party using the 344 prefix.

The change-over was not flawless. There were some troubles with phones outside BNL or in certain area codes getting calls through, and also with class markings which determine how the phone can be used to call out. Fax machines had to be set up to use the new phone lines. If you are experiencing trouble with your BNL phone, you should call teh BNL phone service center at x4031. Special adaptors for analog lines can be purchased or rented. Additional technical information about the new telephone system can be obtained at x4031, and also on the Web via the NSLS and CCD (BNL Computing and Communications Division) Home pages.

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MARCH 1996 NSLS NEWSLETTER

NEW FAXING PROCEDURE

Previously, to send a fax to a number outside BNL you entered

6xxxxx9 then area code and number,

where xxxxx is you telephone account code. The **6** is now replaced with ***2** so would would enter

*2xxxxx9 etc.

This new procedure is related to the new BNL telephone system

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NSLS Home Page............ BNL Home Page............

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MARCH 1996 NSLS NEWSLETTER

A BIG THANK YOU....

...to all who used the NSLS Electronic Submission System to submit an experimental summary, or abstract, for the 1995 Activity Report. We have received 488 abstracts, which is about **40 MORE** than the previous year!

There were a few difficulties with the system, which became evident under the heavy and varied usage. Plans are underway to iron out those problems and also include some of the very useful suggestions made by the authors.

NSLS Home Page........... BNL Home Page...........