New Synchrotron Light Sources Sputtering

Murphy's Law, high expectations, and a shortage of funds and accelerator physicists add up to exasperating delays at Brookhaven and Wisconsin

High-energy physicists in Europe have started to build an immense electron storage ring that will be 27 kilometers in circumference and will have a beam energy of 50 billion electron volts (GeV). So should it not be child's play for U.S. scientists to construct and operate a much smaller, lower energy machine to produce synchrotron radiation? Apparently not, as anxious biologists, chemists, physicists, and materials scientists are discovering as they wait to irradiate their samples with photons from the National Synchrotron Light Source (NSLS) at Brookhaven National Laboratory and from the Aladdin storage ring at the University of Wisconsin's Synchrotron Radiation Center.

Brookhaven's NSLS consists of two rings, one (0.75 GeV) that produces ultraviolet radiation and the other (2.5 GeV) that generates x-rays. Prospective users were once told they could expect to have enough light to begin aligning their ultraviolet instruments in April 1981 and their x-ray equipment in October of the same year. Although both machines were completed by their respective deadlines, the ultraviolet source began operating reliably only 2 months ago, and there is still no firm indication when there will be x-rays.

Wisconsin's Aladdin, a 1-GeV ring designed to penetrate into the soft x-ray region of the electromagnetic spectrum, is even farther behind. Once scheduled to be running well enough for users to begin hooking up their gear in May 1980, the machine's most recent projected starting date is this December.

What is causing the trouble? Several accelerator physicists contacted by *Science* agreed that there is no intrinsic reason why these storage rings should require the amount of time they have to be commissioned. Although several factors have combined to make the job tougher than anticipated, the storage ring technology is not the main one.

Nonetheless, both the NSLS and Aladdin represent significant advances in accelerator design, says Ednor Rowe, the director at Wisconsin, and for that reason alone no one should have expected them to emit copious quantities of light the first time operators threw the ON switch. According to Ewan Paterson of the Stanford Linear Accelerator Center, the giant machine LEP that is being built by the European Laboratory for Particle Physics (CERN) near Geneva is primarily a scaled-up version of existing storage rings for high-energy physics, whereas NSLS and Aladdin push the technology in a new direction.

The NSLS and Aladdin aim to maximize the production of synchrotron radiation by making densely packed electron beams of small cross section. High-energy physicists want to minimize synchrotron radiation, which depletes the particle beam of energy and for technical reasons cannot use small, dense beams in machines like LEP anyway.

The intensity of synchrotron light depends primarily on the electron beam energy, the beam current, and on the radius of the storage ring. High energies and small radii translate to higher intensities and also to the ability to generate shorter wavelengths. For many experiments, source brightness is a more important figure of merit than intensity. Roughly speaking, a small-sized source emitting light into a narrow cone is brighter than a large source spewing forth the same number of photons.

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The jargon term for the type of electron beam needed to enhance the brightness of synchrotron radiation is low emittance. About a decade ago, the late G. Kenneth Green of Brookhaven pointed out the importance of low emittance to achieving high brightness. Then Green and the late Renate Chasman, also of Brookhaven, devised a particular way to achieve this property in a storage ring in the course of designing the NSLS.

Rowe independently took a similar approach for Aladdin. Since then, several other synchrotron radiation sources have been planned in the United States and overseas that also aim for a low emittance. The idea has been so well studied by now that no one doubts that it is workable. And John McTague, who became NSLS director in early 1982, argues that the performance of the ultraviolet source there now constitutes a proof of principle for Green's concept. A second proof resides in Berlin. An 0.8-GeV storage ring named BESSY, which works about as well as the NSLS ultraviolet source, has been open to users for over a year.

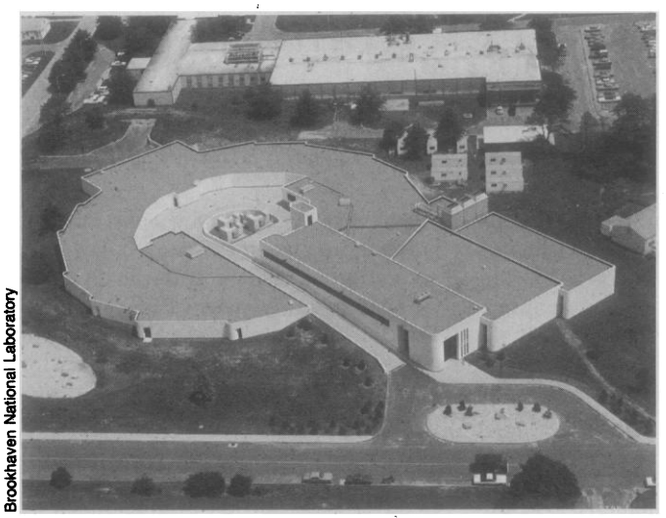
The problem from the user point of view is not that the NSLS x-ray ring and Aladdin are not delivering light as bright as advertised, but not even as much as existing machines. None at all, in fact.

The ultimate causes of the delays in getting on line seems to lie in low finances, the problems they caused, and Brookhaven management's responses to the problems. The laboratory had bad luck from the start. The NSLS designers, Green and Chasman, coincidentally died in August and October 1977, just as the funding of the project began. Adding to the impact was a worldwide shortage of electron accelerator specialists because of four new machines starting up in the United States and Europe.

But McTague and others at Brookhaven point to a low budget as underlying their problems. A flat figure of $20 million was set in Washington as the maximum the NSLS could cost. The laboratory would have to design a facility to fit that amount. The final figure was higher, $24 million, but in the late 1970's inflation was rampant. By contrast, the ultraviolet facility BESSY cost about $32 million with only one ring. A number of observers have praised Brookhaven and project director Arie Van Steenbergen for "building a $40-million machine on $24 million."

The major variable in the design was the number of rings. The higher energy x-ray ring also produces ultraviolet radiation, and Brookhaven's original proposal included only one ring to serve both user groups. When ultraviolet users demanded a separate ring, the laboratory acceded, but the construction budget did not grow correspondingly. A number of compromises had to be made.

Several of these comparatively small cost-shaving measures have already slowed progress. Symptomatic of the situation is the large number of surplus parts from decommissioned accelerators incorporated into the system that injects electrons into the storage rings. An electron linear accelerator gives an initial kick to the particles to get them to 70



Brookhaven National Laboratory

NSLS

About 60 percent of the instrumentation there is being built and paid for by so-called participating research teams from universities, industry, and government laboratories. Altogether, they have contributed almost $20 million toward the facility.

million electron volts, and an electron synchrotron boosts these to 0.7 GeV, the injection energy. Some of the parts are rather old. "Parts of the electron linac should be in a museum," comments Samuel Krinsky, who was recently put in charge of the NSLS x-ray ring.

In fact, the booster synchrotron was not able to achieve its 0.7-GeV energy. After a first, feeble beam was stored in the ultraviolet ring in August 1981, there was a shutdown while a power supply for the synchrotron was rebuilt. Not until December were NSLS operators able to store a beam and build up its intensity by accumulating electrons from several synchrotron pulses.

Another 6-week delay came when operators determined that they had to examine the radio-frequency cavity that replenishes energy lost from the beam as synchrotron radiation. To save money, designers left out vacuum valves at the ends of the cavity. This meant that the entire beam pipe of the storage ring, which is normally kept under ultrahigh vacuum, had to be exposed to air. To reachieve the lost vacuum, it is necessary to "bake out" the beam pipe by heating it to a few hundred degrees for long periods. The synchrotron radiation itself is the final purge as the ultraviolet light desorbs contaminants from the walls of the beam pipe. The requisite vacuum valves have since been installed.

One of the most aggravating inconveniences traceable to cost cutting resulted from the transport system that directs electrons from the booster synchrotron to either the ultraviolet or the x-ray rings. The NSLS strategy was to get the ultraviolet source operating reasonably well before devoting much attention to the x-ray side. Unfortunately, when this stage was reached last year, the ultraviolet source was not reliable. Researchers understandably became annoyed when their light disappeared, and they demanded its restoration.

However, to save $30,000, the transfer beam pipes that connected the synchrotron to the two storage rings had only a single set of controls, which had to be set differently according to which ring was to be filled with electrons. For various reasons, it sometimes took as long as 4 hours to switch the settings from those appropriate to the x-ray ring to those needed to inject in the ultraviolet source, fill the ring, and switch back again. The delays occurring when NSLS operators responded to the ultraviolet ring users meant that relatively little progress was made on the x-ray ring commissioning, according to Van Steenbergen, who now is deputy director of the facility. The first beam was stored in September 1982 and an energy of 2 GeV was reached 2 months later. But since then it has been difficult to raise the beam current. Additional controls have just been ordered.

The lack of resources and the dearth of available accelerator physicists combined to take another toll. NSLS officials emphasized hardware in their tight budget situation. Everyone involved now admits that they badly miscalculated the effort needed to get the facility running once it was actually built. Maury Tigner of Cornell University recalls that there were from 6 to 12 people capable of making "primary decisions" during the construction and commissioning of the CESR storage ring there. "You're in trouble if you have much less than that," he adds. The rest of Brookhaven was not in a position to help out, as trying to save the troubled ISABELLE/Colliding Beam Accelerator project was taking most of the laboratory's attention.

Two pertinent examples of the effects of not being flush with machine experts spring to mind. One concerns the ultraviolet source's reliability. After a month-long search, Kenneth Batchelor of the NSLS staff found that the small magnets for correcting the electron orbits produced fields with small but unpredictable variations. The synchrotron light therefore missed some of the waiting users. Discovering this effect led to its cure and the currently reliable operation of the ultraviolet source. But a shortage of experienced electron machine physicists and time delayed the search until this summer.

The second example has to do with the power supplies for the quadrupole magnets that focus the electron beam of the x-ray ring. Some of these power supplies were poorly regulated, so that the current oscillated or rippled slightly. The effect was again small (1 percent) but was partially responsible for the inability of the x-ray ring to accumulate an intense electron beam. And again, the discrepancy was not found until this spring when a detailed component-testing program was instituted when Krinsky began his tenure as x-ray ring supervisor.

As might be surmised, a differently managed NSLS might have made faster progress despite the numerous obstacles. "There were some imprudent choices made in the course of time at the NSLS; it took longer than it should have," admits Nicholas Samios, director of Brookhaven. With the ISABELLE affair resolved (*Science*, 9 September, p. 1038), Samios now says that getting the NSLS x-ray ring going is the laboratory's first priority. Two accelerator specialists have been detailed to the NSLS, which also has authorization to hire some of its own, along with engineers and technicians.

Last spring, there was a bit of a shakeup. McTague put Krinsky in charge of commissioning the x-ray ring. At the same time, Claudio Pellegrini, who started out at Brookhaven on the ISABELLE project, was made overseer of the ultraviolet source. Pellegrini is an electron accelerator expert and was involved in the workings of the first GeV-energy electron storage ring, ADONE, at the Frascati laboratory near Rome, in the early 1970's. Krinsky is, comparatively speaking, learning on the job. He began his career as a theoretical physicist before becoming an apprentice to Green when the NSLS was being designed.

Nonetheless, Krinsky is highly regarded, and his group has made significant progress. It was his idea to cease further development of the x-ray ring until thorough testing of much of the equipment could be carried out. A team of experts from outside the NSLS in a subsequent review concurred with this decision and

one wrote to McTague that true commissioning of the ring could not be considered to have begun until problems with "flaky hardware" were overcome.

What remains to be done? Both the ultraviolet and x-ray rings need to store higher current electron beams. All other conditions equal, the more electrons in the beam, the more light is emitted. Both machines' design specifications call for considerably higher currents than high-energy physics storage rings can tolerate. There are instabilities that are unique to the high-current machines, and so they have not been addressed in high-energy physics accelerators. Some of these have already been encountered in the ultraviolet source, where the circulating current in the ultraviolet ring should eventually reach 1 ampere. It is about 0.3 ampere now. The NSLS x-ray ring has a lot farther to go. The maximum current so far is 14 milliamperes, with the goal being 500.

Moreover, the x-ray ring is not fully completed. A vacuum bake-out system is just being installed this month. Without it, the vacuum has been below specifications. Collisions between the electrons in the beam and residual gas molecules eventually deplete the beam, limiting its lifetime. And the existing radio-frequency cavity is insufficient to accelerate a high-current beam to the 2.5-GeV design energy. A second one has yet to be tested and put in place.

As for when x-ray users can expect photons, Krinsky cautiously predicts this January, but only if nothing unexpected goes wrong. Spring is more likely, he concludes. Krinsky's bottom line is that it is "only when you know what the problems are that you know how long it will take to fix them."

The same situation pertains at Wisconsin. Aladdin does everything it is supposed to do except store a high beam current. Rowe is telling his users to plan on a December starting date for hooking up their equipment. But Wisconsin's resources are, if anything, slimmer than Brookhaven's for the NSLS. Aladdin's construction budget was $4.5 million.

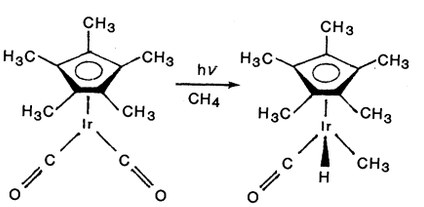
Everyone has to stretch dollars when funding is tight. Perhaps, however, there is a limit to the stretching. If, as presidential science adviser George A. Keyworth, II, has repeated more than once, the NSLS is an example of the excellence to be supported in times of lean budgets, then those proposing such facilities should have the courage to ask for adequate resources to build and operate them, and Congress and the funding agencies should be willing to provide the wherewithal.—**ARTHUR L. ROBINSON**

Methane C–H Bonds Activated

One of the foremost goals of many organometallic chemists is the use of transition metal complexes to activate—insert a functional group into—saturated and aromatic hydrocarbons. Activation allows further reactions to be carried out on the hydrocarbon, particularly reactions that would make it useful as a precursor to organic chemicals. Such activation has been achieved for most types of hydrocarbons (*Science*, 17 June, p. 1261) with a single important exception—methane. Methane has the strongest C–H bonds of any simple hydrocarbon and has proved exceptionally resistant to activation. This is unfortunate because methane is a widely occurring hydrocarbon gas that would, in many ways, be ideal as a source of both synthetic fuels and chemicals.

At the recent meeting of the American Chemical Society (ACS) in Washington, two groups of investigators reported independently that they had finally coaxed methane into adding oxidatively to a transition metal complex. The reaction described by one group was photochemical and that of the other was thermal, but the two approaches were nonetheless very similar and similar complexes were used in each.

William A. G. Graham and his colleagues at the University of Alberta used a simple iridium complex, $[\eta^5 - C_5(CH_3)_5]Ir(CO)_2$. When this complex is dissolved in perfluorohexane at room temperature under about 8 atmospheres of methane and illuminated, methane is added oxidatively to form the new complex $[\eta^5-C_5(CH_3)_5]Ir(CO)(H)CH_3$. This complex is very unstable, but can be readily converted to the very stable complex $[\eta^5-C_5(CH_3)_5]Ir(CO)(Cl)CH_3$ by reaction with carbon tetrachloride. Formally, this complex might be considered to be one in which chloromethane has added oxidatively, but Graham has not yet been able to reverse the process and expel chloromethane. Similar results have been obtained in a complex with a cyclopentadienyl ligand instead of the pentamethylcyclopentadienyl ligand.



Robert G. Bergman and his colleagues at the University of California, Berkeley, first tried to photolyze a similar complex, $[\eta^5 - C_5(CH_3)_5][P(CH_3)_3]IrH_2$, in perfluoroalkane solvents, but they were not successful. Bergman then reasoned that the desired methylated complex should be thermodynamically more stable than a complex with a secondary alkyl group, such as a hydridocyclohexyl complex, $[\eta^5 - C_5(CH_3)_5][P(CH_3)_3](C_6H_{11})(H)$, that he and Andrew H. Janowicz had prepared last year. When this complex was dissolved in cyclooctane and heated to about 150°C under 20 atmospheres of methane, the corresponding hydridomethyl complex was formed. This product was also unstable, but it too could be readily converted to the chlorine complex.

Strictly speaking, neither of these two cases represents the first time that methane has been activated with an organometallic complex. At last spring's ACS meeting, Patricia L. Watson of the Du Pont Company reported that certain lutetium and yttrium complexes undergo exchange reactions in which unlabeled methane bound to the complex exchanges with $^{13}CH_4$ in solution. This exchange suggested that methane C–H activation was possible, but it was a nonproductive reaction in the sense that there was no net increase in the amount of activated methane. Some investigators, furthermore, argue that the reaction of methane in Watson's case was not an oxidative reaction of the type sought.

The next step, which has also proved difficult with other hydridoalkyl complexes, is to force release of a functionalized hydrocarbon from the complex. If it should be possible to add a carbonyl to the methyl moiety, for example, the reaction would have great utility for synthetic purposes. For now, however, such a development still seems to be some time away.

—**THOMAS H. MAUGH II**