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Semiannual Progress Report
(March 1 through September 1, 1994)

Large Area Soft X-ray Collimator to Facilitate X-ray Optics Testing

Prepared by

Physitron, Inc.
3304 Westmill Drive
Huntsville, AL 35805
Samuel L. Espy

Prepared for

NASA/Marshall Space Flight Center
Marshall Space Flight Center, AL 35812

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Semiannual Progress Report for

LARGE AREA SOFT X-RAY COLLIMATOR TO FACILITATE X-RAY OPTICS TESTING

The tasks for this program can be divided into two broad categories, those relating to the design of a high-performance soft x-ray collimator and the tasks involving the demonstration of reflector fabrication techniques. Since the program's initiation on March 1, 1994, we have made significant progress in both areas.

A design procedure has been developed for both single and double reflection x-ray collimators which use nested reflectors having conical geometry. This procedure relies on two different software tools. The first is a program which we developed in-house with Mathematica; it contains a generalized representation of the intricate geometrical issues involved in producing a design which maximizes optic throughput and the acceptance angle of the optic while producing an optic with minimal "shadowing" and exit divergence as well as addressing most of the other tradeoffs that exist. The Mathematica program uses the Henke data set for determining the reflectivity as a function of photon energy and grazing angle. The second software tool is a commercial raytracing program called Opticad. For a particular optic design, Opticad receives input from the Mathematica program and conducts a raytrace with a large number of rays to simulate the nearly infinite number of ray/surface intersections that are possible. We believe that the procedure that we have developed for the single-reflection collimator represents one that is optimized for the intended use, but we believe that we can "squeeze" a little better performance from the double-reflection designs by altering the procedure slightly so we are still working toward this. The details of the final designs as well as the results of the raytrace calculations will be given in the final report on this program.

We are very pleased with the progress thus far in the fabrication related tasks. When we initiated the program, cost was of primary concern and a methodical study had not been reported in the open literature which determined the quality of the master surface required in order for lacquer-smoothing to produce sub 10 Å final surfaces. We, therefore, chose to develop our reflector fabrication techniques using aluminum mandrels that were hand-polished rather than using expensive, diamond-turned mandrels. We had four aluminum mandrels machined and polished to a bright finish using conventional machine shop techniques. The mandrels were then coated with a thin layer of lacquer with a technique whereby the mandrels are slowly drawn out of a lacquer-filled tank. Following this, the lacquered mandrels were then rotated at a constant speed in a vacuum chamber and evaporatively coated with 750 Å of copper. Copper was chosen for this first attempt since it was considerably less expensive than gold and we wanted to test the rotation fixtures which we made for both the vacuum chamber and for our electroplating tank. The four mandrels, which at this point had an electrically conductive layer on top of the lacquer, were then coated with a thick (nominally 0.020") layer of nickel with a zero-stress electroforming technique. While this step was successful in that it allowed us to test the zero-stress electroforming process, the 750 Å copper layer proved to be a poor substitute for gold since the copper introduced nonuniform deposits due to electric field aberrations introduced by the heavily oxidized copper surface. We should also note that the zero-stress nickel electroforming technique was used in place of the copper electroforming process described in the original proposal for this effort because the sulfamate nickel chemistry proved to be more stable and easier to maintain zero-stress metal growth. Furthermore, the replacement of the copper electroforming step with the nickel electroforming process does not in any way adversely affect the initial goals of this program or the quality of the x-ray reflectors produced.

We initially tried to release these electroformed reflectors from the mandrels by dissolving the lacquer with various organic solvents, but this process proved to be too slow and also led to damage of the reflector surface. The damage mechanism seemed to be caused by capillary effects in the 5 μm gap between the mandrel's outer surface and the vacuum deposited copper/electroformed nickel shell. This space is, of course, initially filled with lacquer . Ultimately, we released the reflectors from the mandrels by immersing the mandrel/reflector combination in liquid nitrogen. The aluminum mandrel then contracted more than the nickel reflector during cooling and a complete separation occurred at the lacquer/copper interface. Furthermore, we determined that separation without damage to the reflector surface could be achieved when the lacquer remained adhered to the mandrel rather than separating from it and releasing with the reflector.

After this first series of coating experiments, we stripped the four mandrels and hand-polished their surfaces attempting to produce a surface which was smooth enough for the lacquer to reduce the rms microroughness below 10 Å. Our polishing attempts were not as successful as we would have liked. We have since found that bare aluminum is considered by experts in the field to be very difficult to polish to the level which we require. Typically, researchers deposit, via an electroless nickel bath, a hard coating comprised of 89% nickel and 11% phosphor on aluminum to produce a surface which can be polished to the required degree. Since this was not possible given the funding constraints of this effort, we settled for the best finish which we could achieve with the aluminum mandrels and our hand-polishing technique. Until the mandrels were lacquer-dipped and evaporatively coated with gold, we could not determine the quality of the mandrel surfaces since the lacquer is transparent. Only after the 750 Å layer of gold was deposited could we determine the success of our polishing efforts. This, of course, further complicated our polishing efforts.

We conducted the lacquering/gold coating/nickel electroforming process three times with the set of four mandrels for a total of twelve electroformed reflectors. The problem with the surface quality of the mandrels was discussed earlier, but one of the mandrels was successfully polished to a degree that the surface of the reflectors which were electroformed onto it were very good as seen by the human eye. The surface roughness of one of these reflectors was measured by TMA Technologies, Inc. using a total integrated scatter technique. The rms roughness reported by this technique was 90 Å. While this is not nearly as good as we must achieve for x-ray reflectors, we have evidence that the surface quality of our aluminum mandrels is now the limiting factor. We have used qualitative, light-scattering measurements to measure the surface quality of lacquered/gold coated samples both before electroforming a thick nickel layer and after reflector separation. These measurements indicated that no damage was occurring either during the electroforming process nor during the release from the master substrate. Since the state of the art in polishing metal mandrels currently produces sub 10 Å surfaces, we feel that it is only a question of obtaining mandrels that have a surface quality which is better than our current mandrels produced. It is certain that since we use lacquer-smoothing the mandrels will not have to be as good as 10 Å to achieve reflectors with sub 10 Å surfaces.

With this in mind, Physitron has initiated an IR&D program (funded up to $100k) in which we are obtaining four additional aluminum mandrels coated with a nickel/phosphor alloy which is polished to 10 Å. The reflectors which were produced from the hand polished mandrels and were paid for with NASA funds will be tested at the Naval Research Laboratory at the same time that we test the reflectors made with funding from the IR&D effort. Any possible future x-ray collimators which we fabricate for NASA will benefit from our IR&D research since our IR&D effort is an extension of this NASA program and is focused on improving reflector fabrication techniques.

For the IR&D effort, we will make several sets of reflectors from the four diamond-turned mandrels using our lacquer-smoothing replication process. A set of reflectors made from these mandrels will be assembled into a four channel collimator. The four channel collimator along with individual reflectors produced from the diamond-turned reflectors and those which we have already produced from the conventionally machined mandrels will be tested at an x-ray source at the Naval Research Laboratory in Washington D.C. in the near future. The surface microroughness of the reflectors will also be measured.

In a related project, we fabricated two types of ultrasmooth foils, one with gold as the reflecting surface while the other has a nickel reflecting surface. Both reflectors have an electroformed nickel layer to provide structural integrity. Both were made by evaporatively depositing 750 Å of the reflecting metal, nickel or gold, onto a superpolished silicon wafer and then electroforming a thicker layer of nickel onto the metal coated wafer. After removal from the electroplating bath, release of the ultrasmooth foil from the wafer was accomplished by taking advantage of the differential thermal contraction that occurs between the metal foil and the silicon substrate when the combination is immersed in liquid nitrogen. Separation occurs between the metal reflecting layer and the silicon wafer surface. Surface roughness measurements of ultrasmooth foils with both gold and nickel surfaces were conducted by Zygo Corporation using their NewView 100 system employing scanning white light interferometry. The rms surface microroughness varied between 5 to 7 Å.

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Samuel L. Espy

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Physitron, Inc.
3304 Westmill Drive
Huntsville, AL 35805

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The first objective of this program is to design a nested conical foil x-ray optic which will collimate x-rays diverging from a point source. The collimator could then be employed in a small, inexpensive x-ray test stand which would be used to test various x-ray optics and detector systems. The second objective is to demonstrate the fabrication of the x-ray reflectors for this optic using lacquer-smoothing and zero-stress electroforming techniques.

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