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RESEARCH CENTER

AMERICAN OPTICAL COMPANY

Southbridge, Massachusetts

PREPARATION OF PLATINUM-FREE LASER GLASS

Semi-Annual Technical Report
Number Two

1 January 1965 - 31 July 1965

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Prepared by

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This research is part of Project Defender under the joint sponsorship of the Advanced Research Projects Agency, Department of Defense, and the Office of Naval Research

ABSTRACT

It has been demonstrated that the oxidation of platinum followed by the subsequent reduction of the oxide particles to the metal is a major source of inclusions in laser glasses melted in systems containing platinum. These glasses can be melted in platinum without inclusions if an inert atmosphere such as nitrogen or argon replaces oxygen in the system. Laser glasses prepared in this manner have given excellent performance in high energy laser devices.

In a parallel but different approach to the elimination of platinum inclusions an all-ceramic melting system has been designed and constructed. This system, which utilizes special refractories and stirring techniques, is undergoing preliminary tests.

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1. INTRODUCTION

The research being carried out under this contract has as its goal the preparation of glass of high optical quality, free of platinum inclusions, and of such composition as to be well suited for use in high energy laser applications. The investigation is divided into two tasks which are being undertaken concurrently. One task is the improvement of the techniques for melting glasses in platinum so that it might be used for laser glass production without causing inclusions; the other task is the development of an all-ceramic system, entirely free of platinum, that would be capable of producing homogeneous laser glass.

Platinum inclusions have been found in all types of laser glasses and optical glasses, both foreign and domestic, wherever platinum was a part of the melting system. These lacy, rounded particles are usually small (commonly from 5 μ to 50 μ in length) and often few in number (perhaps one or two in 10 cubic centimeters of a carefully prepared glass), but their presence is quite undesirable in laser glasses as they cause breakage of laser elements during high energy or high power operation.

In view of the need for glass laser materials which could be used in high energy and high power applications it was decided to attack the problem with two separate approaches; if one were unsuccessful it was hoped that the other would find a solution. First, the improved use of platinum in laser glass melting systems would be explored with the objective of preparing glasses without inclusions. Second, a melting system which contained no platinum would be designed and constructed entirely from ceramic materials. In addition, other optical glass manufacturers would be approached and encouraged to also try to produce some laser glasses free of platinum inclusions.

In the platinum improvement program it was reasoned that since platinum had been widely used since World War II in systems producing high quality optical glass, it would be ideal if it could somehow be protected or made inert with respect to the glass. The main advantage of platinum in optical glass melting systems is that it enables glass to be produced that is free of striae (threads of glass of different refractive index from the matrix glass) due to the superior resistance of platinum to attack by molten glass. This corrosion resistant character of platinum makes it possible to thoroughly blend and homogenize molten glass with platinum stirrers. Therefore, if the platinum could somehow be passivated, much of the optical glass melting equipment presently in use could be adapted to produce high quality laser glass.

In the all-ceramic melting system the greatest problem would be that of overcoming the solution of the refractory in the glass and removing the resulting striae. In the past, however, particularly before World War II, much high quality glass, even glass of astronomical objective quality, was made in ceramic pots without the assistance of platinum. The poor yield of the processes led to the use of platinum in melting. New ceramic refractory materials are now available that are considerably better than those in use before the War, and with the proper refractories and careful melter design and operation it would appear possible to prepare glasses that might meet the homogeneity requirements for laser use.

2. PLATINUM IMPROVEMENT PROGRAM

The purpose of this program was to determine the mechanism causing the appearance of platinum inclusions in laser glasses and to develop some technique for melting these glasses in platinum which would permit them to be prepared without inclusions.

2.1 SUMMARY OF PREVIOUS WORK

Several mechanisms were considered as possible sources of the platinum inclusions:

(1) Mechanical abrasion, where particles are scraped from the platinum by the contact of a hard object.

(2) Metallurgical changes in the platinum, such as inter-granular attack or grain growth and detachment.

(3) Solution of the platinum by the glass and subsequent reprecipitation.

(4) Oxidation of the platinum and subsequent reduction of the oxide.

A very small number of inclusions probably do originate from accidental scraping of the platinum, but the shaving-like shapes of these particles distinguish them from the lacy configration commonly found. Occasionally, a grain may separate from the main platinum body, but again, very few inclusions were found that had the size or shape of the platinum metal grain structure. Mechanical abrasion and metallurgical changes were therefore ruled out as major contributors to the inclusion problem and attention was concentrated on the solubility and oxidation mechanisms.

In this work and in the work that follows, the assistance of Battelle Memorial Institute of Columbus, Ohio, was obtained through a subcontract. Metallographic examinations, analyses and controlled experiments were carried out there under the guidance of J. L. McCall. The Battelle research supplemented and confirmed the investigations carried out at Southbridge by American Optical.

2.2 CONSIDERATION OF THE SOLUBILITY MECHANISM

Several experiments were conducted to determine the solubility of platinum in a typical K_2O-BaO-SiO_2 laser glass base. Solutions of $PtCl_4$ in water were added to the raw batch materials which were then melted in ceramic crucibles at 2700°F to make approximately 100 cc of glass. The glass was cast into flat slabs and examined for inclusions. The solubility of platinum in the glass appeared to be insignificant as lacy metallic platinum inclusions were found in glasses where the concentration of $PtCl_4$ by weight was as low as 1 part in 10^{10}.

The glasses containing the small inclusions were given heat treatments in which they were heated to the softening point, soaked, then either slow cooled or quenched as rapidly as was possible without breakage. No difference in the number or size of particles was observed as a function of cooling rate indicating again that no appreciable solubility existed for platinum in this glass.

In another series of experiments small ($\sim$ 10-20 μ) crystals of platinum were placed in a bead of molten glass of the same composition as that used above and observed with a hot stage microscope at 2700°F. In the 20 minute periods of observation the particles did not go into solution. When the samples were cycled to room temperature and back repeatedly, there was no apparent solution of the particles, indicating that the platinum inclusions must exist as metallic crystals at the laser glass melting temperature and that they do not precipitate from the glass. Some agglomeration of the particles was observed; if they contacted each other at the high temperatures they tended to sinter together in linked globules (Figure 1).

Consideration of the results of these experiments plus the fact that repeated careful spectrographic analyses showed no platinum in solution in the glass convinced the investigators that the solution and reprecipitation mechanism was not the source of the inclusions observed when laser glasses were prepared in platinum. It was decided at this point to concentrate on the platinum oxidation mechanism as the major cause of inclusions.

2.3 CONFIRMATION OF THE OXIDATION MECHANISM

In the previous report on this research[1] it was suggested that the oxidation of platinum and the subsequent introduction of either this oxide or its metallic decomposition product in the glass was one of the principal mechanisms for the formation of

Figure 1. Coalescence of Platinum Crystals in Glass as Observed by Hot Stage Microscope. Mag. 400X

platinum inclusions. This hypothesis was based upon the known slow, but definite, oxidation of platinum at high temperatures and upon experiments which demonstrated that the rate of weight loss of platinum at glass melting temperatures (2400-2700°F) was dramatically increased by the presence of oxygen. Subsequent experiments showed that platinum inclusions were not found in laser glasses melted in platinum crucibles under conditions where oxygen was excluded by a nitrogen atmosphere.

The surface of the platinum is believed to oxidize to PtO_2 at the glass melting temperatures and this oxide volatilizes quite readily. The oxide may decompose at a hot surface to produce platinum crystals and oxygen or condense on a cold surface.

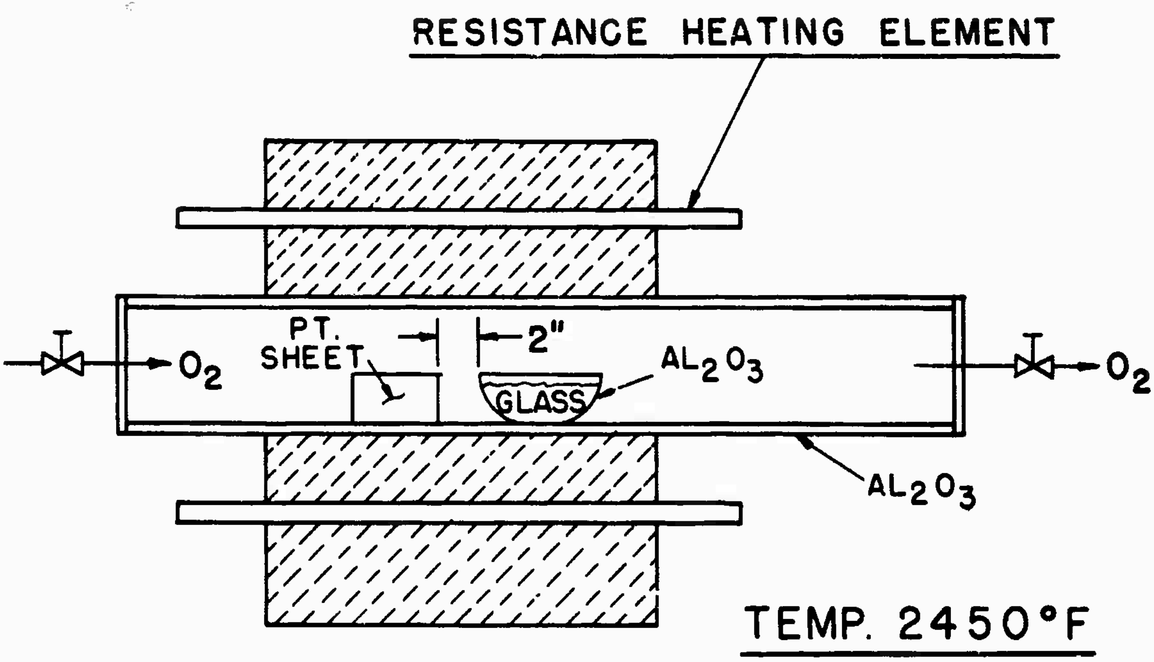
at hot surface: $Pt(s) + O_2 \rightleftharpoons PtO_2(g)$

at cold surface: $PtO_2(g) \longrightarrow PtO_2(s)$

The oxide crystals may fall into the glass where they are reduced in situ to form metallic platinum having the same morphology as the oxide crystal. Metallic platinum formed in the hot parts of the system may also fall into the melt to form inclusions.

The oxidation of the platinum also appears to take place in contact with the liquid glass below the surface if oxygen is present above the glass. If no oxygen is present above the glass the partial pressure of the oxygen in the glass soon becomes negligible and the oxidation of the platinum stops. This phenomenon is demonstrated by the following experiment. Specimens of platinum were completely submerged beneath the surface of the glass in ceramic crucibles and heated to 2450°F in both air and argon atmospheres. The specimens heated in glass under an air atmosphere showed a weight loss comparable to the weight loss of platinum in air. The platinum specimen submerged in glass under an argon atmosphere showed no measurable weight loss.

Another experiment was carried out to demonstrate that the transport of platinum oxide alone can cause the formation of inclusions in a glass physically separated from hot platinum. A sheet of platinum was placed in the hot zone of an aluminum oxide tube furnace and a ceramic crucible of laser glass was placed two inches away in the same tube (see Figure 2). A stream of oxygen was passed slowly through the tube so that it went over the hot platinum (2450°F) and then over the molten glass. When the glass was removed for examination it contained platinum inclusions. When the same experiment was performed with nitrogen, no inclusions were observed.



APPARATUS TO CHECK

ATMOSPHERIC TRANSFER

OF PLATINUM

Figure 2. Schematic Representation of Experiment Demonstrating the Transfer of Platinum Through Furnace Atmosphere

2.4 PREPARATION OF PLATINUM-FREE GLASS UNDER NITROGEN

As previously reported, laser glasses melted in 100 cc capacity platinum crucibles under a nitrogen atmosphere appeared to be essentially free of platinum inclusions and resisted breakage when irradiated by bursts of laser light. After these encouraging results it was decided to construct a system large enough to prepare enough glass to produce large laser rods that could be tested as active laser elements. Such a system was built and is shown in Figure 3.

The platinum crucibles are fabricated by rolling platinum foil into a cylinder one foot tall and 2-1/4 inches in diameter and welding the side seam and welding the bottom into place. A crucible made in this manner is shown in Figure 4. The platinum crucible is loaded with laser glass that has been previously reacted in a high-purity ceramic crucible and is remelted under nitrogen. The glass is then held under nitrogen for up to 90 hours at 2450°F before slow cooling to room temperature. The striae quality of the glass improves considerably during the remelting although the only homogenization actions present are diffusion and weak convection currents. The platinum foil is stripped off and the glass is then drawn into a laser rod 1 meter long and 18 mm in diameter. These rods contained no visible inclusions and subsequently produced some of the highest energy outputs ever achieved with neodymium glass lasers. The absorption at 1.06 μ of the glass prepared with this technique is quite low, being around .1-.2%/cm.

The glass produced in this manner still contains some striae (see Figures 5 and 6), and present efforts are directed toward improvement of the homogeneity by means of different forms of stirring. The nitrogen atmosphere, of course, will be maintained during any homogenization cycle.

While this technique developed as a test for the feasibility of melting laser glasses under inert atmospheres, it appears to have promise of becoming a process for the routine production of platinum-free laser glass. As the homogeneity of the product is improved, the size of crucible could be scaled up to produce much larger glass cylinders.

If conditions in the glass become excessively reducing, however, it is possible that some problems may be encountered with the reduction of some of the more easily reducible constituents. Experiments performed at Battelle show that antimony, which is commonly added to glasses as a refining or bubble eliminating agent, can be reduced in a glass under a 7% hydrogen, 93% nitrogen atmosphere and can alloy with the platinum to form the brittle intermetallic compound Pt_4Sb (see Figure 7).

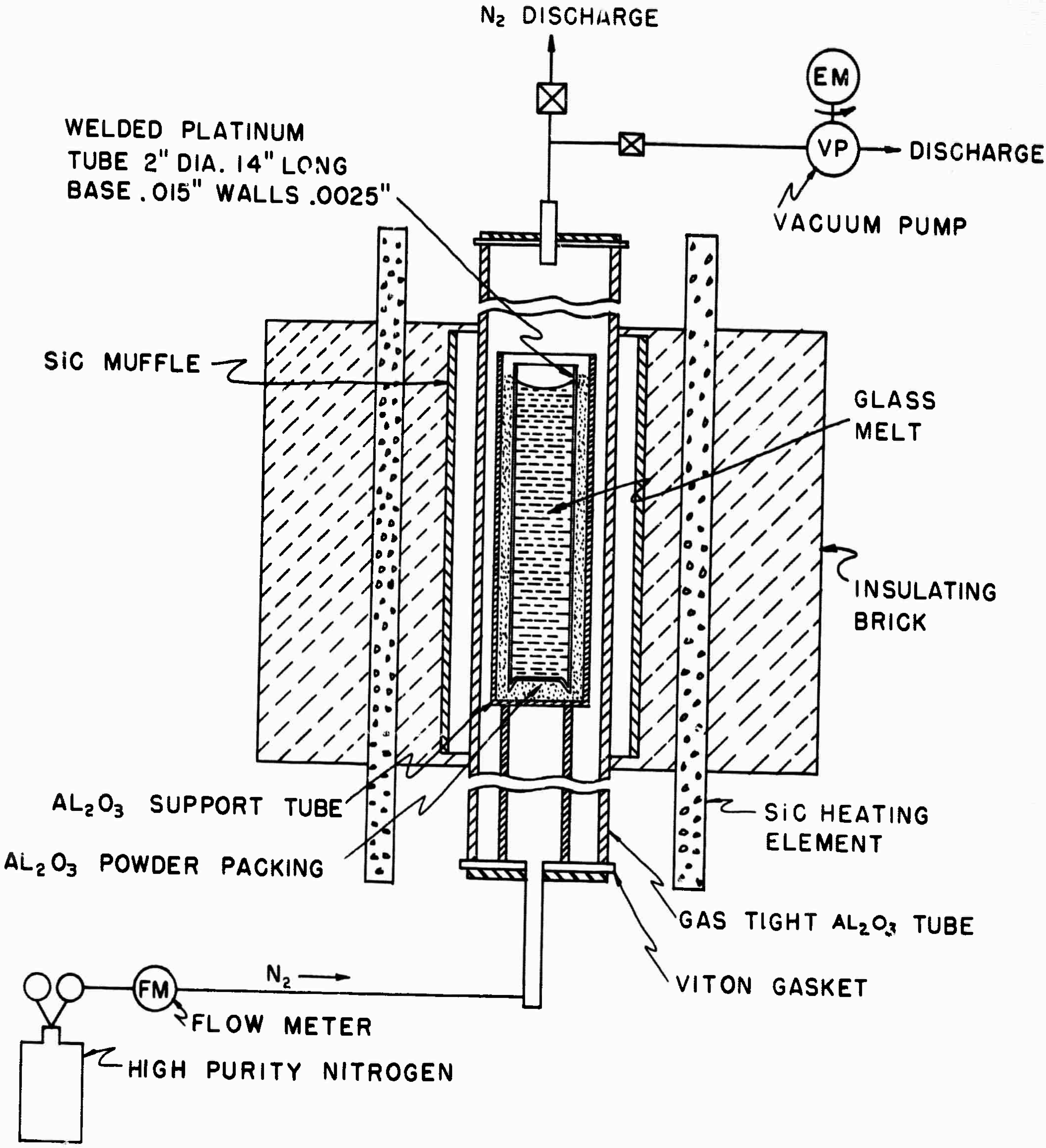


Figure 3. Schematic of System for Melting in Platinum Foil Under Inert Atmosphere

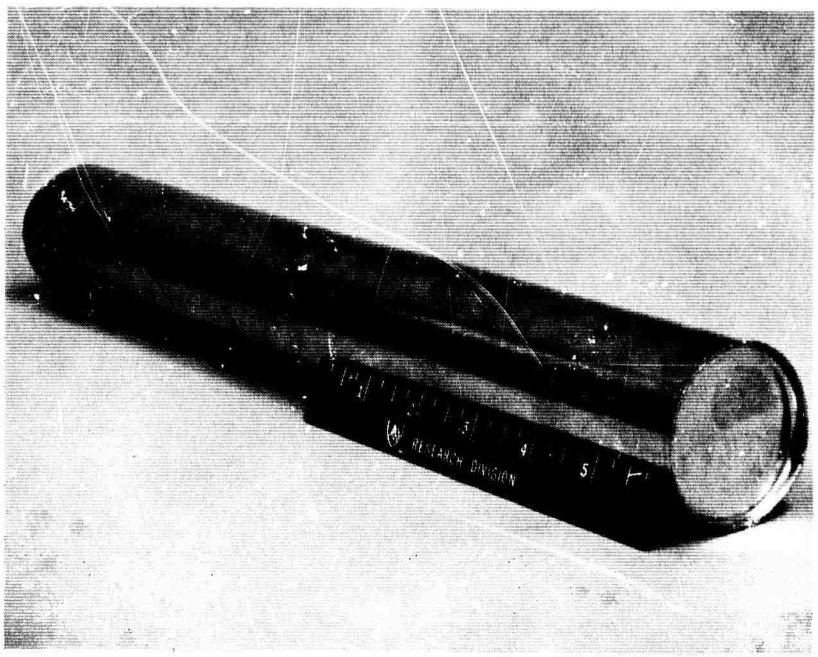
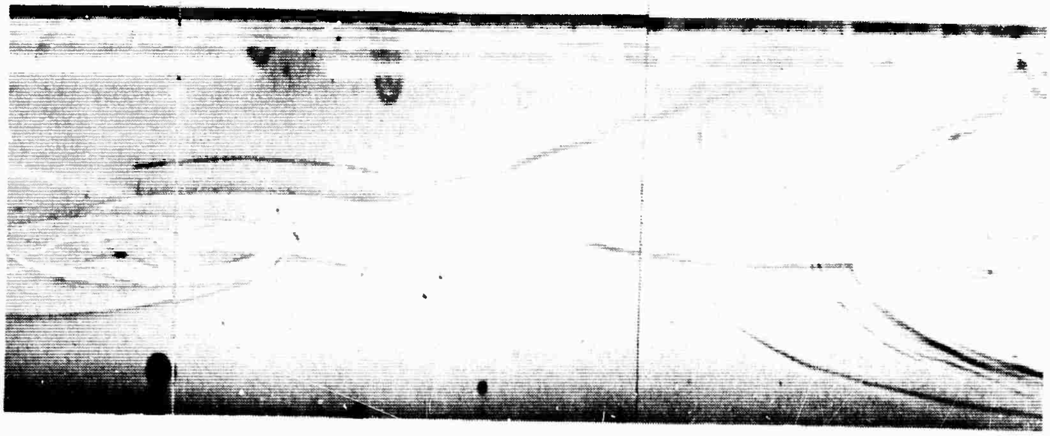


Figure 4. Platinum Foil Cylinder



(Light, parallel lines attop and middle of each picture are reflections from glass immersion tank containing oil of matching refractive index. Short, dark arcs at left center are scratches on the immersion tank surface.)

Figure 5. Shadowgraph of Longitudinal Section of a Portion of Laser Glass Rod Before Treatment in Platinum. Mag. 1X

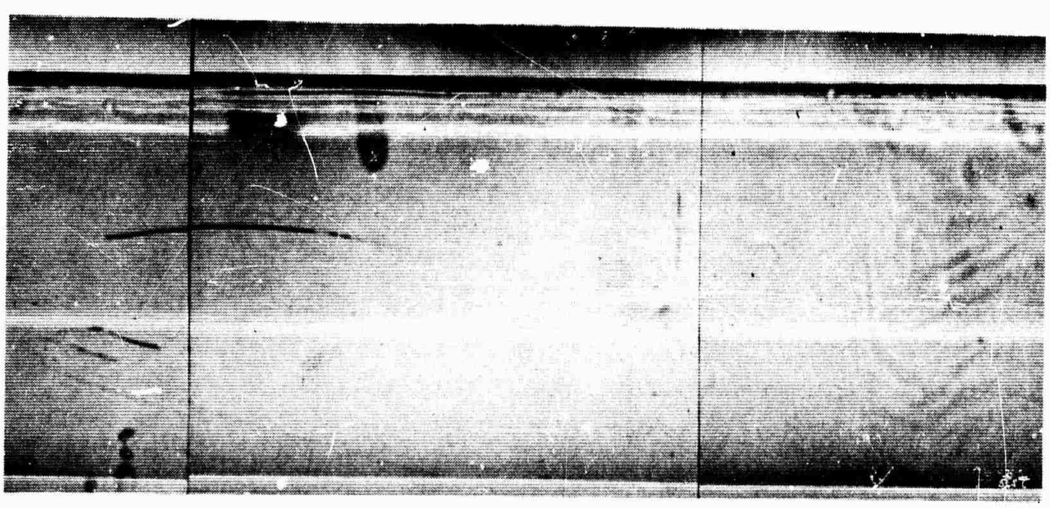


Figure 6. Shadowgraph of Glass Shown in Fig. 5 After Remelting in Platinum Foil Crucible. Mag. 1X

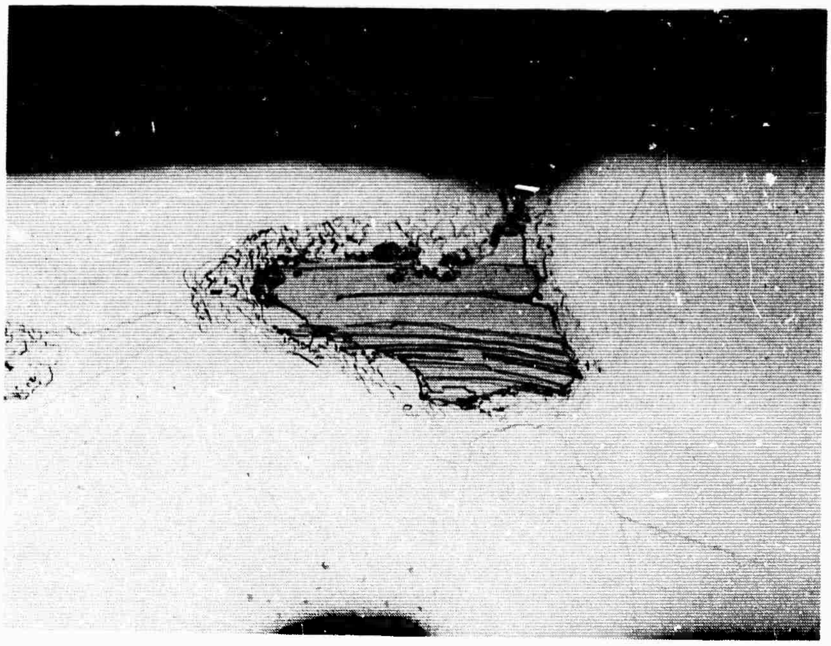


Figure 7. Photomicrograph of Platinum - Antimony Intermetallic Compound in the Wall of a Platinum Crucible

In the absence of antimony, zinc and even silicon can be reduced in reducing atmospheres and can alloy with the platinum. If particles of these platinum intermetallic compounds are present when the glass is reoxidized, the antimony, zinc or silicon can go back into solution in the glass leaving the platinum. Thus, a cycle of reducing atmosphere over a laser glass followed by an oxidizing atmosphere could cause the formation of platinum inclusions. This is not likely under normal melting conditions, but serves to emphasize the point that the platinum attack is rather sensitive to the melt atmosphere and that both oxidizing and reducing atmospheres are to be avoided.

3. ALL-CERAMIC MELTER PROGRAM

The purpose of this program was to develop as quickly as possible a melting system for producing laser glass of high optical quality and free of platinum inclusions. In addition, manpower and budget limitations determined that radical innovations in glass melting could not be explored, so the best existing state-of-the-art knowledge of optical glass melting was combined with special materials and special techniques to minimize contamination and striae.

3.1 SUMMARY OF PREVIOUS WORK

Once the decision was reached to eliminate platinum inclusions by avoiding platinum parts in the system, two major problems remained.

There must be no unwanted absorption of light in the glass, in particular transmission should be as high as possible at the 1.06 μ neodymium laser wavelength, and the glass must be as homogeneous as possible. These requirements meant that ceramic materials of unusual purity and high resistance to attack would have to be selected and provisions made to blend the glass very effectively. Accordingly, a ceramic evaluation program was carried out and experiments were conducted to select a stirring technique that was highly efficient.

About two dozen candidate ceramic materials were tested for their contributions to absorption at 1.06 μ, their resistance to attack by laser glasses, and their resistance to thermal shock. The availability of the materials in the sizes and shapes desired was also considered. From this test program a high purity mullite was selected as the material which possessed the optimum combination of properties for practical use both as a crucible and as a stirrer.

Although homogeneous optical glasses had been produced in large pots in the past, there are problems in the melting of laser glasses in such systems. The special high purity refractories are not always available in the large pot sizes and the volume of glass involved represents a considerable investment in expensive, high purity glass making raw materials. It is also difficult to pour the glass from a large pot without introducing striae. Accordingly, the study of homogenization in this program pursued techniques for blending glasses in smaller systems and casting from a bottom orifice. Laboratory experiments with model systems were followed by actual stirring and casting experiments with molten glass to verify the effectiveness of the methods chosen.

3.2 DESIGN AND OPERATION OF SYSTEM

The design chosen utilizes a thin-walled mullite crucible heated by radiation from vertical silicon carbide resistance elements. The crucible is separated from the heating elements by a ceramic muffle so the atmosphere around the crucible can be controlled if this ultimately proves necessary. The crucible, which holds approximately 50 pounds of molten glass, has a hemispherical bottom to favor stirring and to assist in emptying all of the glass in casting. The same chamber is used for both melting and stirring. The raw materials are added from the top and when all have melted, homogenization of the glass begins. The glass is kept from running out of the chamber by a plug consisting of cooler, highly viscous glass. The system is shown schematically in Figure 8.

After the glass is thoroughly homogenized by stirring, it is cast from the bottom of the chamber by heating the plug of glass. The glass passes out through the tube at the bottom of the system and its flow in this tube is controlled by a helical wire resistance heater.

Figure 9 shows some of the special high purity mullite parts. The stirrer was formed with blades to provide a more positive displacement of the molten glass, and the shaft of the stirrer is hollow for the purpose of air cooling to reduce the rate of attack by the glass.

The overhead stirring mechanism is so designed as to rotate the ceramic stirrer at any speed desired. The stirring mechanism will also oscillate the stirrer back and forth through an arc at speeds and amplitudes independent of the rotation. The crucible itself is supported on a hearth that rotates, again at an independently variable speed, so the combination of movements permit the stirrer to sweep out all parts of the melt with a variety of different stirring patterns possible (overlapping circles, figure 8's, etc.).

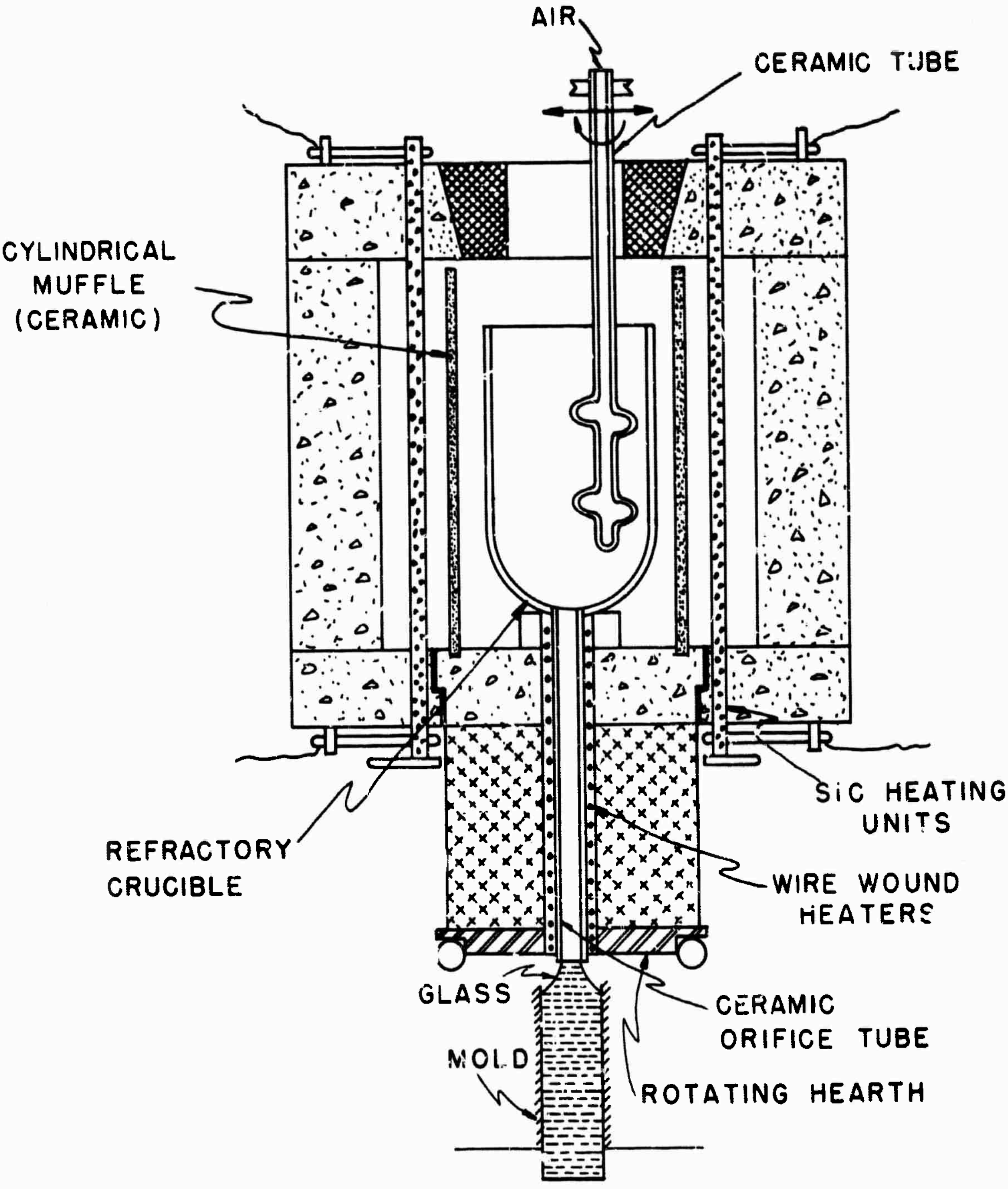


Figure 8. Schematic Representation of All-Ceramic Melter

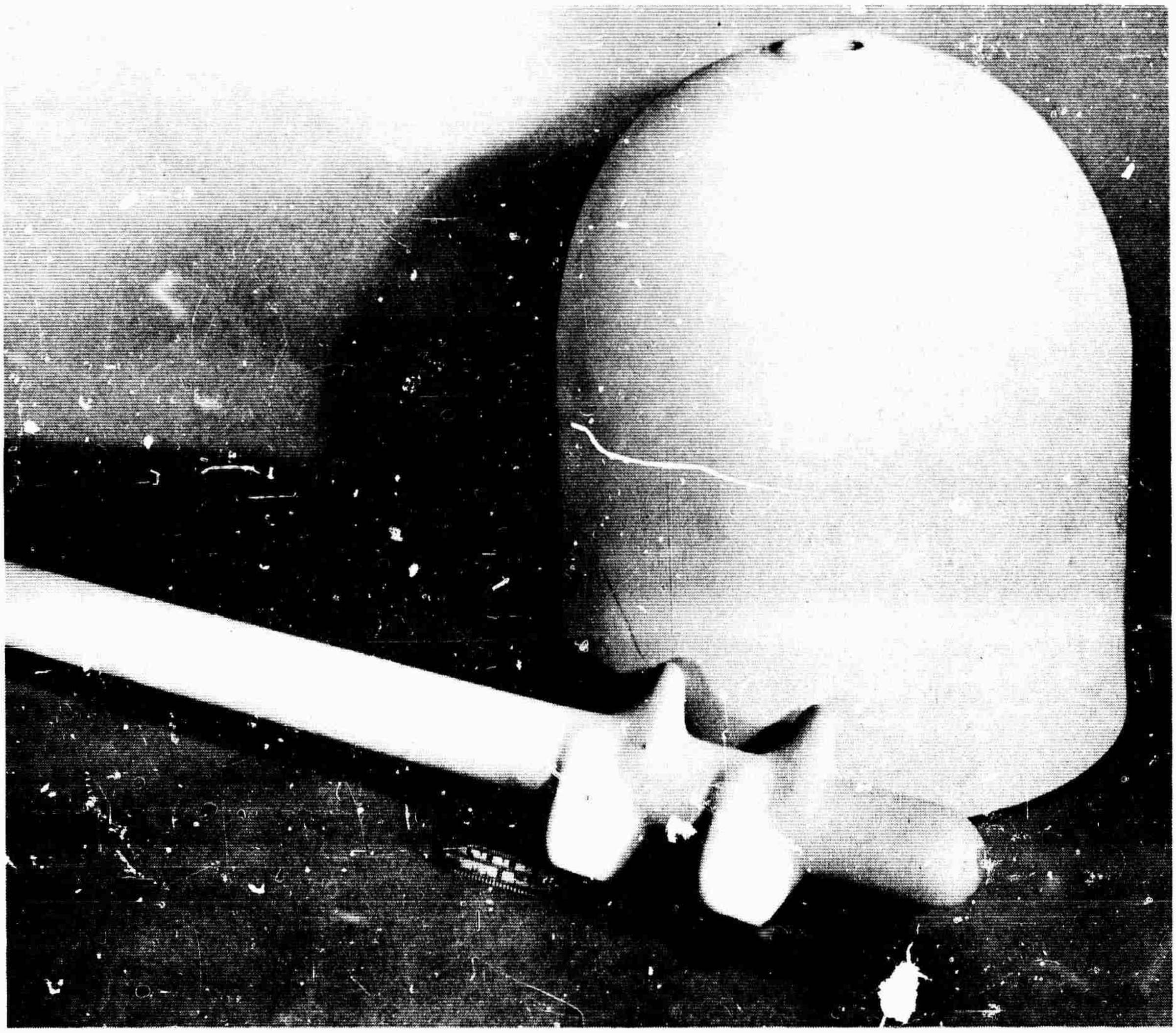


Figure 9. High Purity Mullite Crucible and Stirrer

The casting of the glass will be made into a vertical molding machine, shown in Figure 10, to produce a billet 3 inches in diameter and approximately 42 inches long. A billet of glass this size can be redrawn into a laser rod which will have finished dimensions of 2 inches in diameter and 80 inches in length. The device has been tested and has demonstrated its ability to produce glass in the size stated without adding striae during the casting process.

A view of the finished system is shown in Figure 11. The control panel shows the number of temperature control devices required to maintain the proper distribution of temperatures in the system. It is necessary to avoid overheating and accidental cooling as well as loss of control during casting. Overheating is undesirable as the rate of attack of the refractories by the molten glass increases rapidly with temperature. Excessive cooling, such as that leading to the solidification of glass in the system, is also undesirable as this would cause rupture of the system. The extra temperature control equipment and the complexity of the safety circuits retarded the completion of the unit due to delays in the delivery of the many components. The system is now complete and ready for operation.

3.3 TESTING AND UTILIZATION OF SYSTEM

The new unit will now be operated to determine the optimum time and temperature cycles for high quality glass production. These variables will be studied for the melting, homogenizing and casting phases of operation, and homogenization rates and stirring patterns will be investigated.

The successful operation of the unit will be closely related to the minimum residence time for glass in the system. The longer the molten glass remains in the system, the more material is dissolved from the crucible walls and the more volatile constituents are lost. A compromise must therefore be found between refractory attack at high temperatures and long homogenization times at low temperatures.

To assure prompt melting and the formation of a homogeneous melt it is possible that some time may have to be spent in raw material adjustments and pretreatment. Studies in raw material ratios, particle sizes and mixing as well as batch sintering or pelletizing may be required to obtain the proper melting characteristics.

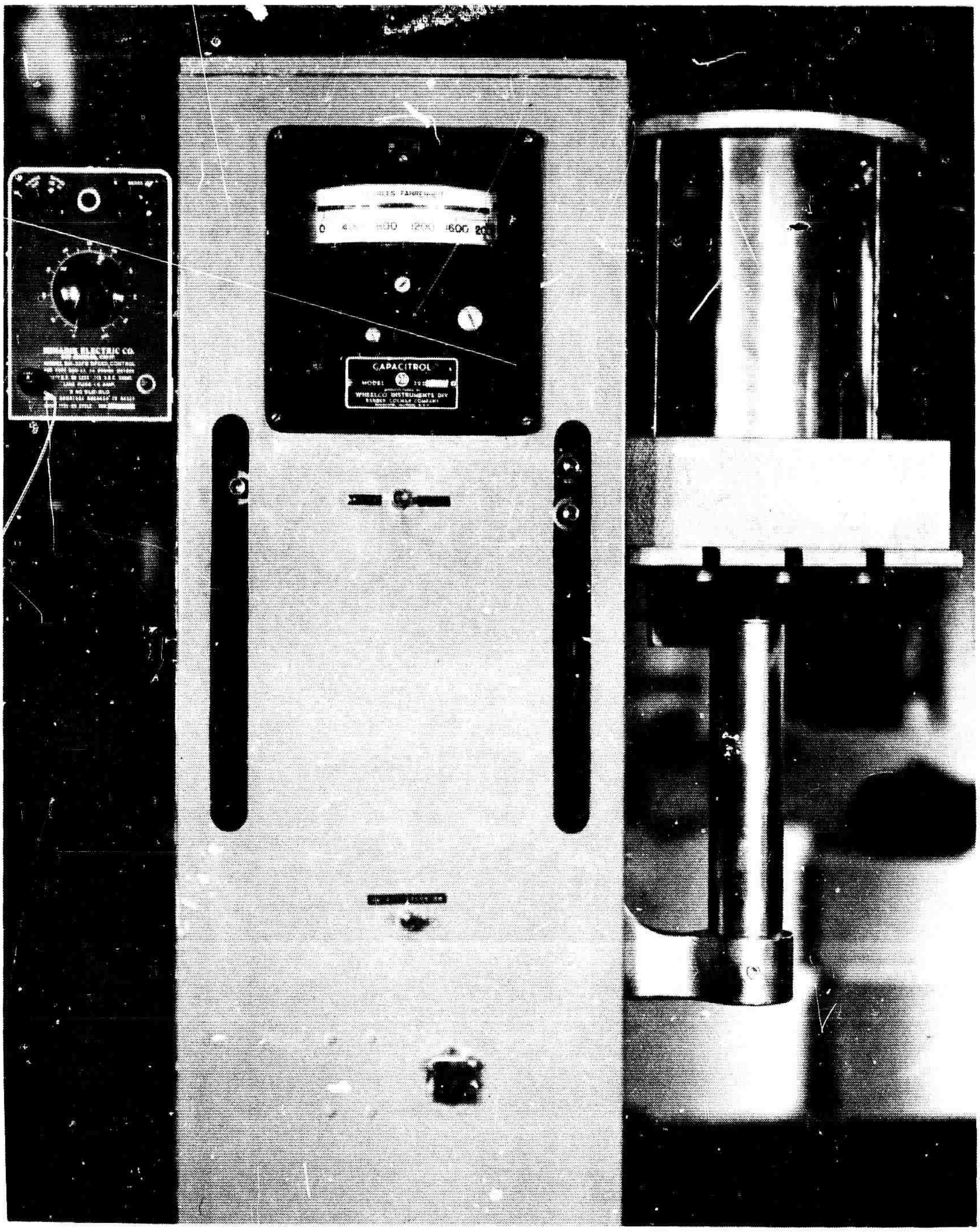


Figure 10. Vertical Casting Unit

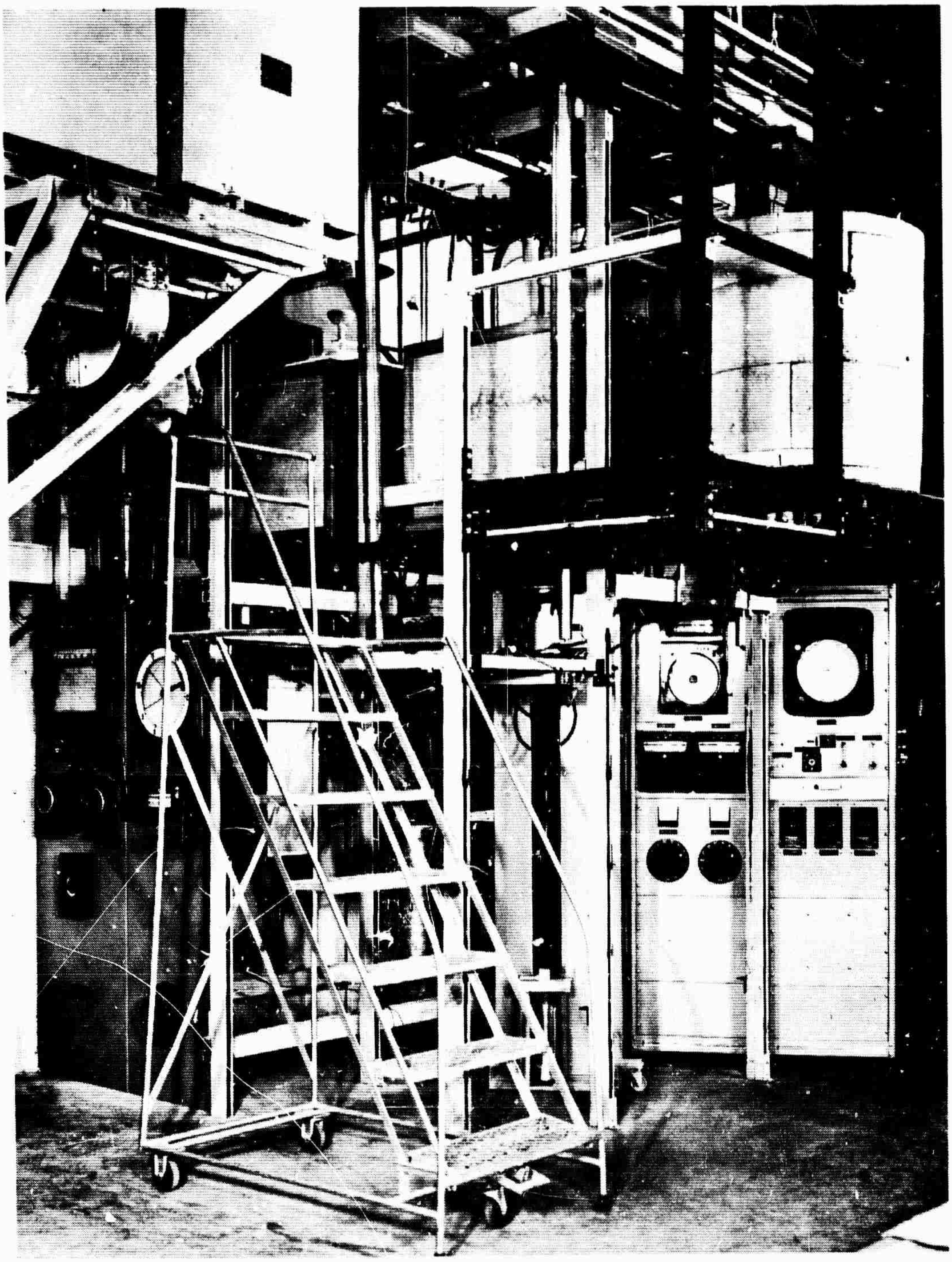


Figure 11. View of All-Ceramic Melter with Casing Removed to Show Refractory Insulation

The system has been designed so that it may be easily modified to maintain a neutral atmosphere over a platinum crucible lining. A schematic diagram of one such possible arrangement is shown in Figure 12. This approach will be tried if the all-ceramic method proves to be unsuccessful.

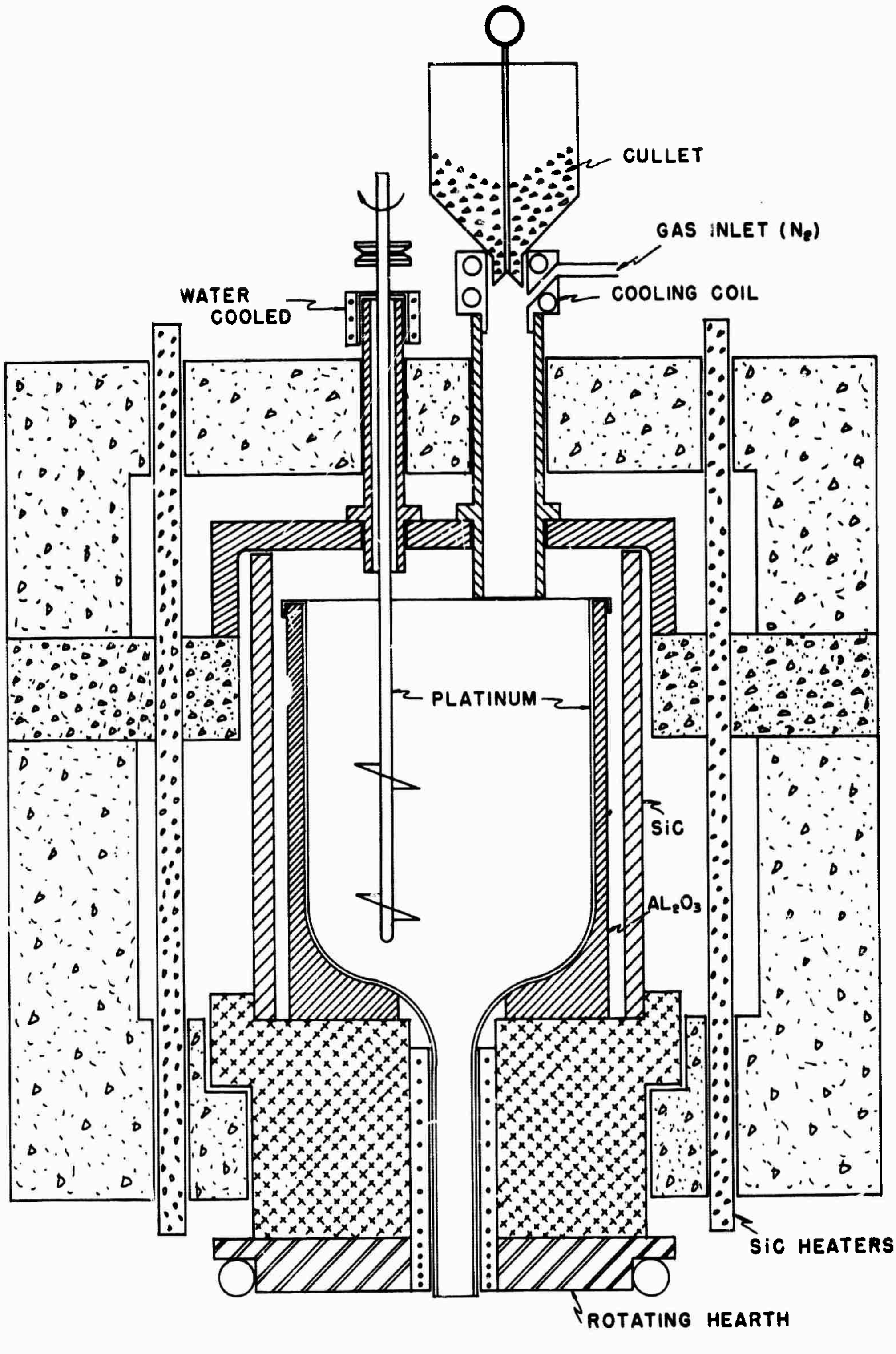


Figure 12. Schematic Diagram of a Possible System for Melting in Platinum in an Inert Atmosphere

4. EVALUATION OF LASER GLASS

As a part of the overall effort to make platinum-free laser glass available, other optical glass manufacturers were contacted and solicited to prepare such glasses to meet the specifications developed by American Optical. From the experience of the Systems Group of the American Optical Research Division in the design, construction, and operation of laser devices, a set of specifications was drawn up which represented a reasonable goal for laser glass that would be capable of high performance in high energy laser systems. These specifications are presented in Appendix A.

Most of the firms contacted expressed interest, but commented that some of the specifications would not be easy to meet, particularly the combination of freedom from platinum inclusions, high homogeneity and low absorption. They stated that these criteria could be met separately with some effort but glass that would satisfy all of the requirements would be a severe test of glass making skill.

Laser glasses from several manufacturers were evaluated, but the only glass that approached the specifications was some made for American Optical by the Jenaer Glaswerk, Schott and Gen., of Mainz, Germany.

The results of the evaluation of this glass are as follows:

(a) platinum particles
No platinum particles were observed. The glass was said to have been melted in a large ceramic crucible.

(b) absorption
The absorption at 1 μ was 0.6%/cm.

(c) homogeneity
The refractive index difference was approximately $\mp 5 \times 10^{-6}$.

(d) non-metallic inclusions
There were bubbles present in quantities larger than allowable under the specifications, but not enough to present a serious problem.

(e) lifetime
The fluorescent lifetime was 540 μsec.

(f) doping
The neodymium concentration was a nominal 5% by weight.

The glass exhibited more susceptibility to solarization than is generally encountered in laser glasses. A new specification is being prepared to define the safe limit for solarization.

The most serious deficiencies of the Schott glass from the point of view of maximum performance in high energy laser systems were the relatively high absorption and the solarization. These conclusions have been communicated to Schott, and they are currently working on an improved glass.

APPENDIX A

Specifications for Laser Glass for High Energy Systems

(a) Platinum Particles

The glass shall contain no metallic inclusions and in particular no platinum inclusions. This requires, of course, that the glass be prepared in a system that contains no platinum in contact with the glass.

(b) Absorption

The maximum absorption shall not be greater than 0.2% per centimeter at 1.06 μ. The measurement of a sample at least 70 mm long and 25 mm in diameter is recommended. The use of the General Electric Hardy Spectrophotometer is recommended, and the value at 1.00 μ can be accepted for the 1.06 μ value as the neodymium absorption spectrum is essentially flat in this region.

(c) Homogeneity

The glass shall contain no striae visible to the unaided eye, and the difference in refractive index($\Delta n_{6328\,\text{Å}}$) across a one inch section shall be no greater than $\pm .75 \times 10^{-6}$. The method of measurement shall be the use of a single-pass interferometer of the Mach-Zehnder configuration with a helium-neon gas laser as a light source (λ = 6328 Å). This is approximately equal to 2½ fringes across a 25 mm diameter rod one meter long when it is examined through its long axis in the interferometer. It is understood, of course, that this measurement can be carried out only on glass that has been given the most careful annealing.

(d) Non-metallic Inclusion

Bubbles and stones are undesirable. The total number of bubbles and stones of a mean diameter of 0.1 mm shall not exceed 10 per pound. The maximum size bubble or stone shall not exceed 0.5 mm in mean diameter and in this size only one bubble or stone per pound is permissible.

(e) Lifetime

The composition of the glass should be such that it produces a fluorescent lifetime (decay time) of at least 500 microseconds. The method of measurement is described in Appendix I of Semi-Annual Technical Report Number One (Contract Nonr-4656(00).

(f) Doping

The glass should contain 5% to 6% neodymium oxide by weight.

REFERENCES

1. W. R. Prindle, G. A. Granitsas, and C. G. Silverberg, "Preparation of Platinum-Free Laser Glass", Semi-annual Technical Report Number One, Contract Nonr-4656(00), January 1965.

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13. ABSTRACT

It has been demonstrated that the oxidation of platinum followed by the subsequent reduction of the oxide particles to the metal is a major source of inclusions in laser glasses melted in systems containing platinum. These glasses can be melted in platinum without inclusions if an inert atmosphere such as nitrogen or argon replaces oxygen in the system. Laser glasses prepared in this manner have given excellent performance in high energy laser devices.

In a parallel but different approach to the elimination of platinum inclusions an all-ceramic melting system has been designed and constructed. This system, which utilizes special refractories and stirring techniques, is undergoing preliminary tests.

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Laser Glass						
Nd:Doped Glass						
Metallurgy						
Platinum						

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