Platinum Inclusions in Laser Glasses

THEIR ORIGIN AND PREVENTION

For some years it has been known that a few particles of platinum may be found in all optical glasses which have been melted in systems containing platinum. These have never been observed to affect the performance of even the most critical of lenses but in 1963 it was discovered at the American Optical Corporation that in laser rods operated at high peak powers they may absorb so much energy as to cause explosive fracture.

The particles, as described by Richard F. Woodcock in a report (1) of investigations in the Central Research Laboratory of the American Optical Corporation at Southbridge, Massachusetts, are generally lacy and irregular in shape with rounded globular contours. They may be so small as only just to be resolved by the optical microscope—about 2 μm across—and exceptionally they may be as large as 500 μm. Even the smallest particle may cause damage.

Possible Origins

Several possible sources of the particles were considered in the investigation and were rejected. They are of the wrong shape to have been torn off by abrasion and no evidence was found to support a fanciful suggestion that they might be the debris from grain boundary attack which it was thought might also detach protruding grains "forced up by grain growth". Moreover, the possibility that they might be precipitated from solution of platinum in the glass was ruled out by tests which indicated that the solubility of platinum in laser glass is less than one part in 10^{10}. It was established that neither the size nor the shape of the particles is affected by long heat-treatment at temperatures up to the softening point of the glass.

The most probable explanation is that they are derived from platinum oxide, PtO_2, formed by oxidation of the free surfaces of the platinum crucibles, platinum heating elements or thermocouples, and are conveyed as vapour to the molten glass. The evidence for this is that platinum particles were not found in glasses which had been melted in platinum in an atmosphere of pure nitrogen, even after they had been kept molten for 90 hours at 2,450°C. Moreover, in an experiment in which a stream of oxygen was passed through a horizontal tube furnace over a sheet of platinum and then over a ceramic crucible containing molten glass, both side by side, particles of platinum were found in the glass. The investigators, however, were not able to establish the mechanism by which the lacy particles of metallic platinum were formed in the molten glass from the gaseous oxide in the atmosphere above. On the other hand, they showed that satisfactory laser glass, free from particles of metallic platinum, was obtained from glass melted in platinum crucibles when oxygen was rigidly excluded and an atmosphere of pure nitrogen was maintained in the furnace.

Methods of Prevention

In applying these findings to production, there was rightly concern over the possibility that platinum crucibles might be contaminated by such elements as antimony and zinc, reduced from the melt, but there was no consistent evidence of such damage. In the event, it was found that it was possible fairly simply to modify an existing 50 lb. platinum-lined extruder furnace so as to maintain over the melt an atmosphere of nitrogen containing about 1 per cent of oxygen. Several satisfactory bars of laser glass were made in this way; but it is indicated that further work to determine the optimum oxygen content—and to control it—is desired.

Concurrently work was carried out to develop an all-ceramic melter. Some progress is reported but the best melts showed various degrees of striae.

This report provides strong evidence that the platinum particles do not derive from attack by the molten glass on the platinum crucibles but from decomposition of PtO_2 vapour in the atmosphere above the surface. And this PtO_2 vapour almost certainly will originate through the reaction of oxygen with the hot outside surface of the crucible and with the even hotter platinum heating elements. Hence the question arises as to whether it might be possible to protect the glass melt from contact with PtO_2 vapour simply by provision of a reasonably close-fitting cover or lid. It would seem well worth a trial.

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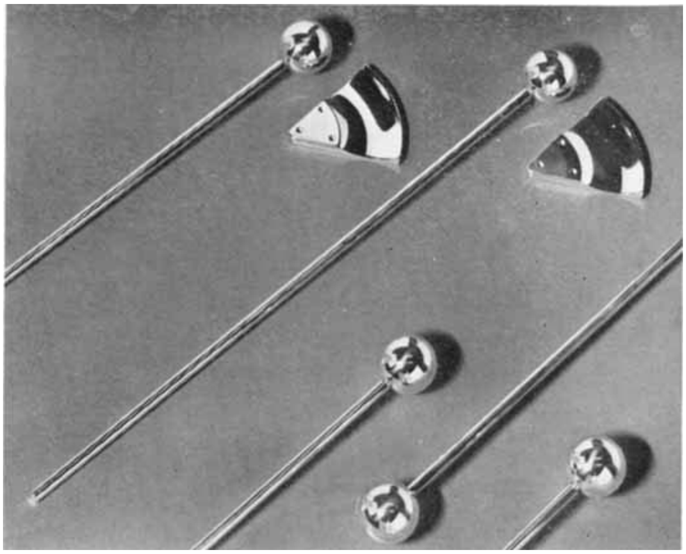
Reference

1 Preparation of Platinum-free Laser Glass, *U.S. Rept. AD692,504*, 1969, (Aug.), 48 pp.

Rhodium Plated Langmuir Probes for Sounding Rockets



Rhodium-plated spherical Langmuir probes for the measurement of electron densities at high altitudes. The sector plates are used for calibration purposes

At the Radio and Space Research Station, Slough, rocket payloads are built for investigation of the production and density of ionisation in the D-region of the ionosphere at altitudes between 65 and 100 kilometres. Several different experiments are combined in each rocket flight, and one of these is a spherical Langmuir probe for measurement of electron density.

The probe consists simply of a conducting sphere, to which a programmed voltage is applied. The current to the probe, due to collection of electrons from the ionosphere, is measured and used to find the electron density as a function of height. It is essential that these probes have a very uniform surface contact potential; also spurious signals, due to photoemission of electrons from the surface by the sun's radiation, must be made insignificant. For these reasons it is necessary for the probes to be rhodium plated and to be kept uncontaminated until they are exposed to the upper atmosphere.

Radio and Space Research Station Langmuir probes plated by Johnson Matthey Chemicals Limited have already been flown successfully on twelve flights of British "Petrel" research rockets and a similar number are to be flown in 1970–71. The plating work is again by Johnson Matthey.