Eliminating Platinum Inclusions in Laser Glass

Damaging platinum inclusions in Nova laser glass can be eliminated by new glass-production techniques. The improved glass will replace the glass disks now in the laser amplifiers and will permit long-term, damage-free operation at full power.

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The large, neodymium-doped phosphate-glass disks in the Nova laser amplifiers have been damaged because of microscopic, metallic-platinum inclusions. These inclusions come from the platinum crucibles used to melt the glass in the production process. The inclusions have a median size of only about 10 μm, roughly equivalent to the size of a white blood cell. Although extremely small, they vaporize upon exposure to the powerful pulses of laser light and produce macroscopic fracture sites within the glass (see Fig. 1). These fracture sites vary in size from a few hundred micrometres to several millimetres.

Damage to the laser glass has been found to occur at laser-light fluences significantly below the Nova design limit. Much of the glass will have to be replaced if Nova is to be operated without damage at its design limit. To prevent further damage to the laser glass, we have chosen to limit the output of the laser to about 50% of the design capability (even though Nova has already demonstrated it can meet its full specified performance). We will stay at this level until the amplifier disks can be replaced with the inclusion-free glass that is now being developed. Replacement is estimated to take about 18 months. Fortunately, since we have about 24 months of experiments that require only 50% power, we will be able to use Nova fully while the glass is being replaced.

Damage sites caused by the laser irradiation of platinum inclusions have been observed in the neodymium-doped phosphate-glass components of both the Nova laser and its predecessor, Novette. The glass disks contain platinum particles with sizes ranging from less than 5 μm (the detection limit) up to about 100 μm, with a mean of about 10 to 20 μm, and varying in concentration from roughly 15 per litre to as many as 1000 per litre.

The inclusions cause fractures in the glass when they are irradiated by laser pulses of 1-ns duration above a threshold value of about 2.5 to 3.0 J/cm^2. We have found, on the basis of experiments and computer modeling, that this damage threshold corresponds to vaporization of the platinum particles as they are heated to temperatures above the normal boiling point of platinum. Results from both models and experiments show that the damage threshold

should be independent of particle size (for inclusions larger than 1.0 μm) and should increase as approximately the square root of the laser pulse length.

One of the most disturbing observations associated with the platinum damage is that it continues to grow with repeated irradiations. Therefore, for all practical purposes, all except very small platinum inclusions ($\lesssim$1 μm) must be eliminated from the glass before it can be used in a high-power laser system.

Historical Perspective

Platinum-inclusion damage in laser and optical glasses has been a recurrent problem throughout the development of high-power lasers. In particular, platinum damage in silicate laser glass has been well known for many years. In some laser systems, platinum inclusions were not a problem. Our previous high-power laser, Shiva, used a neodymium-doped silicate glass, and we observed no damage in that system. However, results from current studies of the Nova glass indicate that Shiva operated just below the damage threshold for platinum.

We chose phosphate-based glass for use in Nova for a number of physics performance reasons, but we also expected it to reduce the risk of problems with platinum inclusions. Our expectation was based on the fact that the solubility of platinum is much greater in phosphate glasses than in silicates.

When phosphate glass was selected for use in Nova, we tested five 15 × 15 × 2-cm samples with 1-ns, 1.064-μm pulses at 12 J/cm^2, using a 6-cm-diameter beam produced by an arm of the Argus laser. These experiments indicated that the glass typically might contain one small (<10-μm) inclusion in each 10 cm^3. Our estimate of inclusion density was correct for all but a few of Nova's disks. However, these qualification studies did not correctly predict the large sizes (some exceeding 250 μm) of some of the inclusions that have been found in glass supplied for Nova, nor did they predict the growth of the damage around the inclusions with repeated irradiations.

Extent of Nova Glass Damage

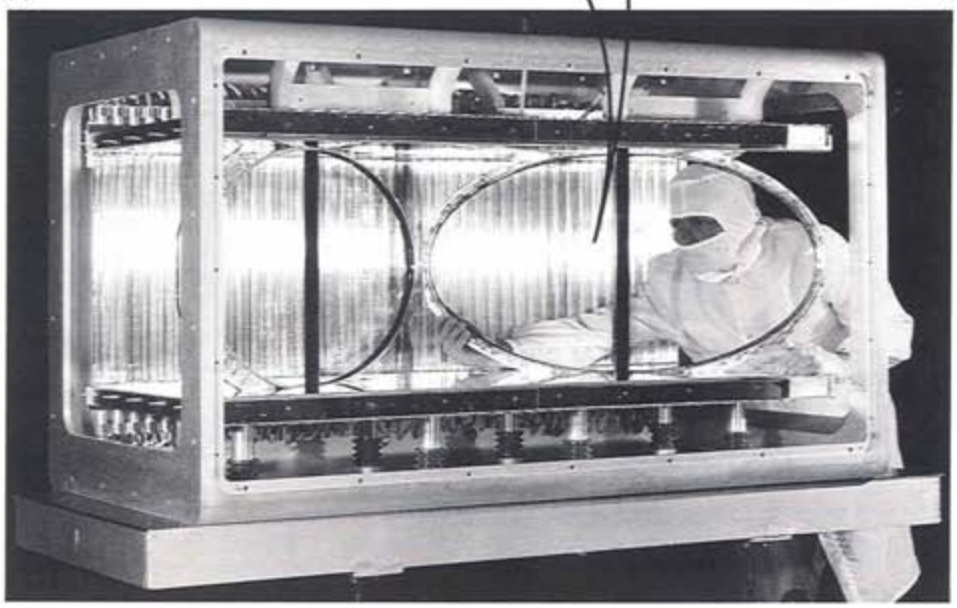
The output stage of a beam line of the Nova laser contains groups of neodymium-doped glass amplifiers with apertures of 20.8, 31.5 and 46 cm (see Fig. 2). Each group of amplifiers is followed by a vacuum spatial filter. A laser pulse passing along the beam line is amplified at one beam diameter to the desired pulse energy and then focused into a spatial filter that removes small-scale spatial ripple and enlarges the beam diameter to match the next group of amplifiers. A spatial

Fig. 1

A typical neodymium-doped, phosphate-glass, half elliptical disk from a large 46-cm amplifier in Nova contains about 7 litres of glass; two half-disks are joined together to form a full ellipse. (a) The damage spots in the glass are produced by microscopic inclusions of platinum. During our inspection of the disks in Nova, we found between 100 and 10 000 damage sites in each disk. (b) One of the 46-cm amplifiers, showing the location of the glass disks; the disks are set at Brewster's angle within the amplifier box.

(a)

(b)



filter at the output of the 46-cm amplifiers further cleans the beam and increases its diameter to 74 cm, the diameter maintained over the final path to the target chamber.

As a result of this series of amplifications and beam expansions, the laser fluence (i.e., the energy per unit area) varies with position along the chain (see the sawtooth curves in Fig. 2), with the largest fluence occurring at the output of the final 46-cm amplifier. The number of glass disks that must be replaced in Nova can be determined by comparing the measured damage thresholds with the average fluences at various positions along the beam line. Such a comparison is made in Fig. 2, in which the shaded bands indicate the damage thresholds for 1- and 5-ns-long laser pulses. It is clear that all of the disks in the 46-cm amplifiers and most of those in the 31.5-cm amplifiers will need to be replaced with the inclusion-free glass. As an added margin of safety, we will also probably replace the laser glass in the last 20.8-cm amplifier.

Solving the Inclusion Problem

In May 1985, a group of LLNL chemists, material scientists, physicists, and engineers were assembled to address the problem of eliminating platinum inclusions in phosphate laser glass. The group had five major goals:

- To assess the damage to Nova and to determine an operating range for which no further damage would occur while improved glass was being developed.
- To measure the damage thresholds of selected platinum inclusions and to determine the growth rate of the damage around the inclusions.
- To develop a quantitative model for the damage caused by laser heating of the platinum inclusions.
- To develop a reliable quality-assurance technique for inspecting large volumes of glass for microscopic

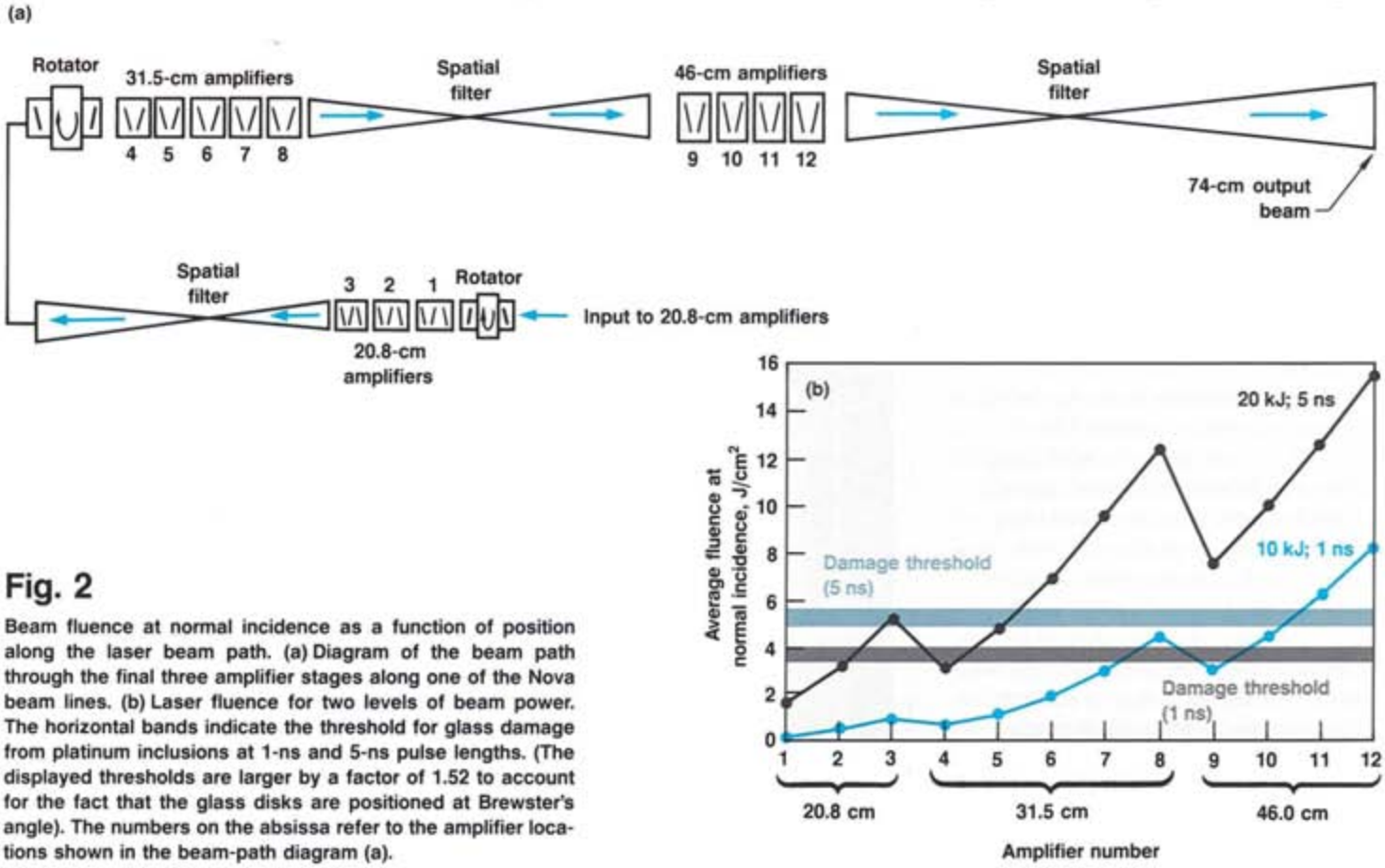


Fig. 2

Beam fluence at normal incidence as a function of position along the laser beam path. (a) Diagram of the beam path through the final three amplifier stages along one of the Nova beam lines. (b) Laser fluence for two levels of beam power. The horizontal bands indicate the threshold for glass damage from platinum inclusions at 1-ns and 5-ns pulse lengths. (The displayed thresholds are larger by a factor of 1.52 to account for the fact that the glass disks are positioned at Brewster's angle). The numbers on the absissa refer to the amplifier locations shown in the beam-path diagram (a).

platinum inclusions (see the article on p. 24).

- To work with the glass suppliers to quantify the mechanisms for platinum contamination and to develop manufacturing processes to eliminate or minimize it.

All five of these tasks have been completed, and we are in the process of procuring the replacement laser glass for Nova.

Assessment of Glass Damage

All of the beam lines on Nova have had tens of shots that produced energies in some sections of the laser greater than the thresholds for damage by platinum inclusions. Consequently, one of our first tasks was to assess the damage level in the present Nova system and to determine the operating power for which no further damage would occur with the existing glass.

To assess the extent of the damage in the existing amplifier disks, we began a microscopic examination of selected disks from the various beam lines. We minimized interference with normal laser operations by examining a small but statistically representative number of all Nova glass disks. These disks were removed from the beam line and taken to an inspection station in a clean room. This inspection stand permits both back and side illumination of the disk with a focused beam from a high-intensity lamp. Large damage sites or inclusions are easily located by the light they scatter. Each damage site was then marked and examined with a microscope to determine its size. Using this method, a trained observer can locate most platinum inclusions or damage sites greater than 10 μm. Because of the characteristic fracture patterns about the damage sites, we can easily distinguish among damage sites, bubbles, and undamaged platinum inclusions. Most damage sites are distinguished by a series of radial fractures that form a circular pattern perpendicular to the beam direction (see Fig. 3).

Although the microscopic examination distinguishes among damage sites, bubbles, and undamaged inclusions, it cannot identify the chemical composition of the inclusion. To verify that the inclusions were indeed platinum, we cored cylindrical samples of several of the damage sites and then fractured the core through the inclusion area. Using scanning electron microscopy and electron-probe microanalysis, we found a high concentration of platinum in the damage sites. In some cases, we also found a pure platinum kernel remaining from the initial inclusion.

Figure 4 displays the results from examination of several of the 46-cm

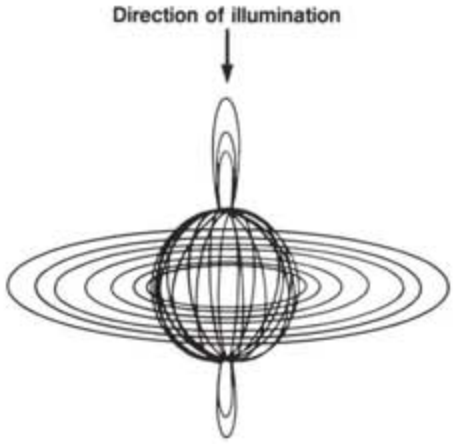


Fig. 3

Schematic representation of the morphology of glass damage caused by a platinum inclusion. There is damage in a small lobe in either direction along the beam, but it is not as extensive as the radial pattern.

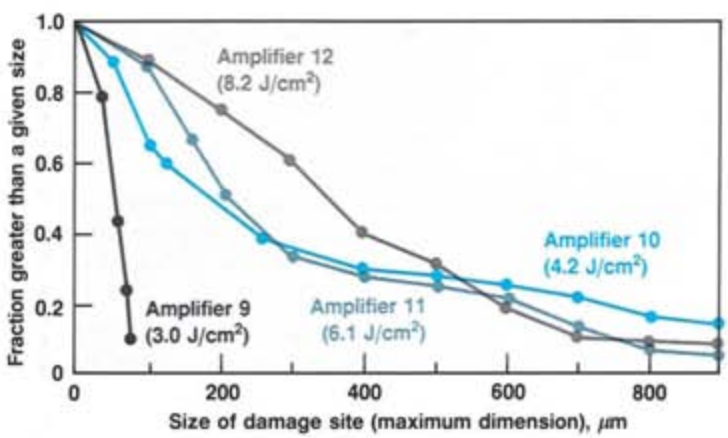


Fig. 4

Normalized plot of the fraction of damage sites (in the 46-cm disks) that exceed the dimension shown on the abscissa. The amplifier numbers correspond to the locations shown in Fig. 2a, and the average fluence is given in parentheses. Many of the damage sites are greater than 500 μm in diameter in the amplifier disks exposed to fluences greater than the 3.5-J/cm^2 (1-ns) damage threshold, i.e., ampifier disks 10, 11, and 12.

laser disks. The data have been normalized and plotted to show the fraction of damage sites greater than a particular size. (We did this because the actual number of sites in a nominal 7-litre laser disk varied from about 100 to as many as 10 000.) As the data show, there is a large spread in the sizes of the damage sites. Furthermore, we observed that the median damage size increases nearly linearly with increasing laser fluence. We believe this linear relationship may be due only to the fact that the laser disks have seen a limited number of shots. It is not clear what the functional relationship will be once the damage growth has stabilized.

These data provide an assessment only of damage that has occurred to the laser amplifier disks over the course of a relatively small number of shots, i.e., about 100 shots vs the expected 10 000 shots over the life of Nova. However, it appears that all amplifier disks that see fluences (at Brewster's angle) greater than 3.5 J/cm^2 (1 ns) will experience an unacceptable level of damage and must be replaced.

Measurements of Damage Initiation and Growth

We measured threshold fluences for fracture initiation in glass containing platinum inclusions and repeatedly irradiated the inclusions to determine the rate of damage growth. Undamaged glass samples containing platinum inclusions were cut from a 20.8-cm phosphate-glass amplifier disk. The major dimensions of the inclusions ranged from 4 to 75 μm. Selected samples with inclusions were placed in the center of a laser beam and irradiated at normal incidence with 1.064-μm pulses having durations of 1.3, 9, or 50 ns. For each shot, the fluence at the beam center was measured to within $\pm 7\%$.

We subjected each inclusion to a sequence of irradiations, usually beginning at a fluence level near 1 J/cm^2. After each irradiation, we inspected the inclusion with an optical microscope. If there was no apparent alteration in the inclusion, we irradiated it again at a higher fluence. Once damage began, the fluence in 1.3-ns experiments was increased to 5 to 10 J/cm^2, a range typical of the highest fluences anticipated in Nova. The inclusion was repeatedly irradiated at these fluences until the size of the damaged volume either stabilized or reached a dimension larger than 250 μm. In experiments with either 9- or 50-ns pulses, the fluences used to observe evolution of damage were selected to be 2 to 2.5 times the threshold for initiation of damage.

The damage threshold was defined as the appearance of one or more fractures extending a few micrometres into the glass surrounding the inclusion. These initial fractures had shapes that were peculiar to individual inclusions. After a few irradiations at fluences above threshold, the damage usually evolved into a circular, planar fracture oriented perpendicular to the incident beam. This damage morphology is the same as that observed on Nova. We could not determine by optical microscopy whether the damage expanded along the direction of the beam.

In most of the experiments, repeated irradiation of the inclusion caused damage growth to sizes larger than 250 μm. Our data for the largest inclusion we studied (major dimension 75 μm) illustrate this damage growth. As Fig. 5 shows, the inclusion survived three irradiations at fluences up to 2.4 J/cm^2 and was first damaged at 3 J/cm^2, during the fourth shot. Damage grew rapidly during the next two shots at higher fluence but ceased when the fluence was dropped below 2.5 J/cm^2. Four additional irradiations at higher fluences caused further damage growth, to a size much larger than 250 μm.

We found damage thresholds to be independent of platinum inclusion size at all three test pulse durations. This is because only the surfaces of the particles are heated during the

laser pulse. The characteristic length for heat flow into a platinum particle is only 0.15 μm in 1 ns and 1.1 μm in 50 ns. Since all inclusions tested had dimensions exceeding 4 μm, only the illuminated surfaces of the particles were rapidly heated to the vaporization temperature.

Thermal Modeling of Damage

Our experiments and calculations show that laser light is absorbed by the opaque platinum, causing a thin skin of platinum on the front surface of the inclusion to vaporize. This produces a shock wave that propagates through the inclusion and is transmitted to the glass. Glass, being a brittle material, fractures because of the stresses caused by the shock wave and the vaporized platinum. These fractures propagate until the pressure of the vaporized platinum decreases below that required for further crack propagation.

Platinum damage in laser glass was first modeled in 1970 by Hopper and Uhlmann,[1] who used analytic expressions for the heat transport and calculated temperatures exceeding 10 000 K when fluence levels were 20 J/cm^2 over a 30-ns laser period. They theorized that very small inclusions were safe at these pulse lengths because heat losses to the glass kept the temperatures and hence the stresses within allowable bounds. However, they argued that for sizes above 0.1 μm, stresses exceeded the strength of the glass, and it could fracture. Their thermal modeling suggested that damage would be dependent upon particle size because the surface-to-volume ratio of the inclusion decreased as the radius increased. Therefore, the larger inclusions had more thermal capacity and hence lower average temperatures and stresses.

In our thermal model, we represent the physical system with a sphere of platinum surrounded by an annulus of glass (Fig. 6a). The model represents what could be a typical inclusion embedded in laser glass (Fig. 6b). In the model, laser light is absorbed on the upper surface of the platinum sphere with a cosine distribution to account for the curvature of the surface.

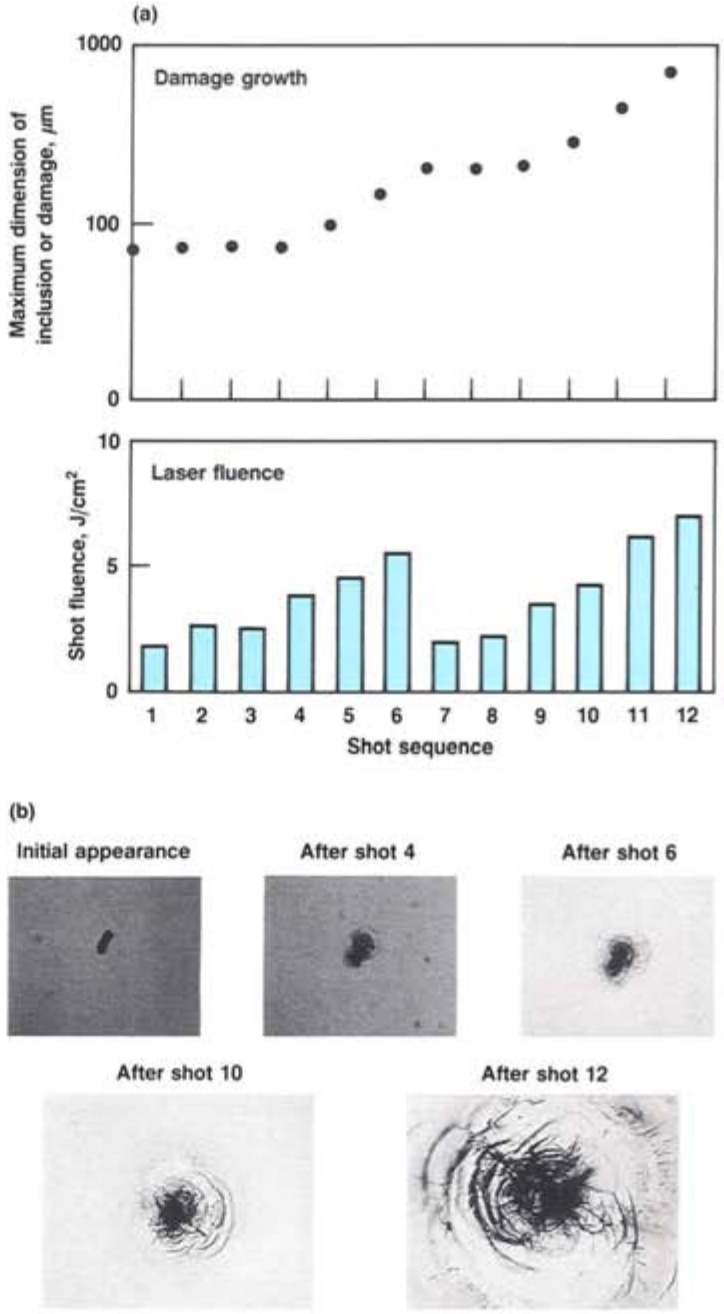
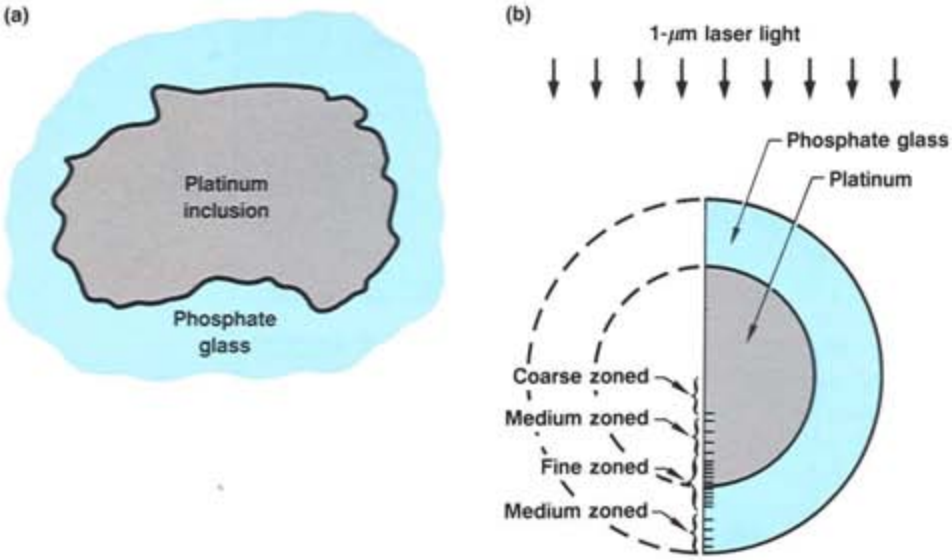


Fig. 5

Evolution of damage surrounding a bar-shaped inclusion with initial major dimensions of 75 μm (one of the largest we observed). (a) Graphs showing damage growth and laser fluence over the 12-shot sequence. Damage began during shot 4 at 3.8 J/cm^2 (normal incidence) and grew rapidly during each shot at fluences greater than 4 J/cm^2. (b) Photographs of the area surrounding the inclusion clearly show the growth of the damage.

Fig. 6

Thermal model used to calculate the effect of laser heating on a platinum inclusion in phosphate glass. (a) Conceptual diagram of what could be a typical inclusion and (b) our spherical model representative of this inclusion. The calculational zones are finest at the site of greatest transient response.



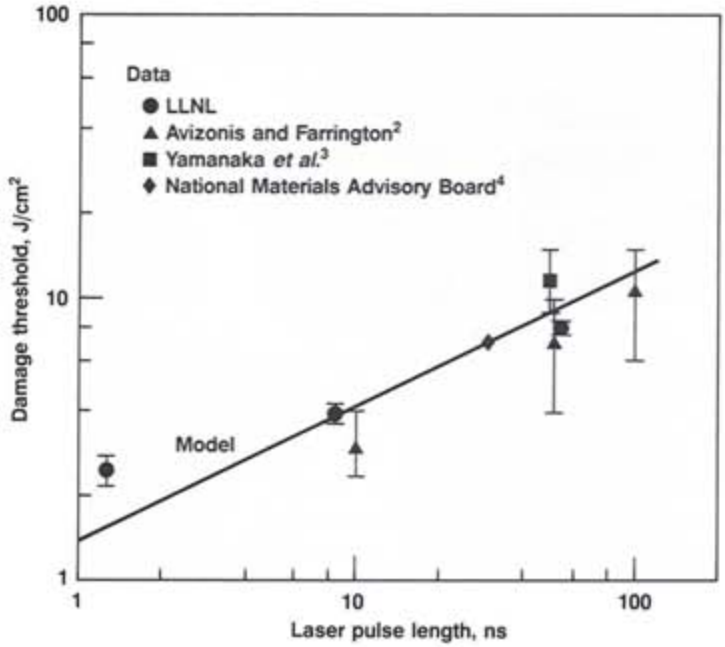


Fig. 7

Effect of pulse length on damage threshold fluences. The data points represent measurements made by LLNL researchers and by experimenters elsewhere; the solid line represents the results of LLNL model calculations. Agreement between our calculation and the experiments is quite good.

Literature values for the spectral and thermal properties of platinum and glass are used in the model. The surface of the platinum is allowed to radiate to a 300-K (room temperature) surrounding medium. No absorption is permitted in the glass, but heat is conducted between the glass and the platinum with negligible contact resistance. The equation of state for platinum above the boiling point is that used in the LASNEX code.

We performed the modeling on a Cray computer, using TOPAZ, a finite-element, heat-conduction code, along with the MAZE and ORION codes to prepare the input and process the output. The results of our modeling are shown in Fig. 7, where the damage threshold is plotted against laser pulse length. Figure 7 also shows our experimental measurements (black dots) of damage thresholds at the three pulse lengths, 1.3, 9, and 50 ns; the damage thresholds were 2.5 ± 0.3, 4 ± 0.4, and 8 ± 0.8 J/cm^2, respectively.

The solid line that represents our model in Fig. 7 has a slope of about 1/2, which corresponds to approximately a square-root dependence of damage threshold on laser pulse length. The slight departure of the theory from the data at the shortest pulse length (1.3 ns) may reflect the limited crack propagation per shot in this region. In other words, it takes a number of shots before any damage can be physically observed. Results from our model compare well with other experimental data, which are also shown in Fig. 7.

Figure 8 shows the thermal profiles that are predicted by our model for a platinum inclusion in glass irradiated with a 1-ns pulse of 1.064-μm laser light at a fluence of 3.7 J/cm^2. The thermal profiles are for two time periods, 1 ns (immediately at the end of the pulse) and 10 ns (9 ns after the end of the pulse). The profile at 10 ns shows the cooling that occurs at the inclusion surface, largely as a result of thermal conduction into the platinum. There is negligible heat loss into the surrounding glass over this time period. Peak temperatures at the end of the pulse are high (greater than 10 000 K), suggesting that significant vaporization of platinum is occurring at the inclusion surface (the normal boiling point of platinum is about 4100 K).

Table 1 lists the calculated maximum platinum surface temperatures and resulting pressures of vaporized platinum for laser shots at various fluences. We have no doubt that the pressures are high enough to propagate fractures. This, in conjunction with our experimental results, suggests that damage will occur if the temperature of the platinum exceeds its boiling point. The experimental data in Fig. 7 show a damage limit of about 2.5 J/cm^2 for a 1-ns pulse and 4 J/cm^2 for a 10-ns pulse. This agrees fairly well with our calculated values.

We are also developing a model for the glass fracturing induced by the heating of the platinum particle. We have not yet developed it sufficiently to predict crack growth. However, crack growth can be conservatively estimated by multiplying the time during which platinum is in a vaporized state by the speed of sound in glass. This estimation method gives reasonable values of crack growth for fluences close to those of available experiments (see Table 1).

The key conclusion of our thermal analysis is that damage will occur if the platinum temperature exceeds platinum's boiling point, thus producing high vapor pressure of platinum. In addition, we found that, for pulse lengths of interest for Nova experiments, the damage is independent of inclusion size (above about 1 μm) and the threshold for damage initiation is lower for the shorter laser pulses.

Glass-Melting Technology

As part of our effort to eliminate platinum inclusions in the

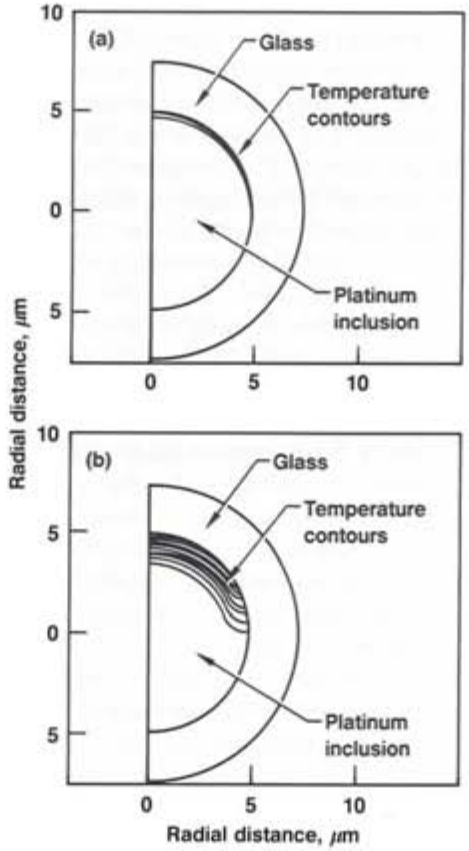


Fig. 8

Calculated temperature contours for a 5-μm-radius platinum inclusion surrounded by phosphate glass and illuminated by a 1-ns pulse of 1.064-μm laser light at an incident fluence of 3.7 J/cm^2. (a) Calculation at 1 ns (i.e., immediately after the pulse) showing the strong surface heating of the particle; the temperature contours range from a minimum of 1500 K to a maximum of 11 000 K. (b) Calculation at 10 ns (9 ns after the pulse); the temperatures have cooled considerably (minimum of about 570 K, maximum of 2700 K) and the heat has penetrated 1 to 2 μm into the particle.

Table 1 **Calculated characteristics of damage to glass caused by metallic platinum inclusions. The predicted fluence damage threshold is 1.4 J/cm^2 for a 1-ns laser pulse length.**

Incident fluence, J/cm^2	Absorbed fluence, J/cm^2	Pulse length, ns	Maximum temperature, K	Maximum pressure, GPa	Crack propagation per shot, μm
37	10	1	1.3×10^5	280	500
18	5	1	7.6×10^4	150	150
3.7	1	1	1.5×10^4	50	8.4
1.8	0.5	1	6.5×10^3	$\ll 50$	3.5
1.5	0.4	1	5.3×10^3	$\ll 50$	1.5
3.7	1	10	4.5×10^3	—	—

phosphate glass used in the Nova laser, we are working with the two companies that are supplying the glass (Hoya Optics, Inc., and Schott Glass Technologies, Inc.). Our efforts are directed toward understanding the formation mechanism of platinum inclusions in phosphate glass and developing process conditions to eliminate it.

Preparing the Laser Glass

The laser glass currently in use in Nova was prepared in a two-step, batch-melting process. In the first step of this process, a cullet glass is made by melting the raw starting materials. This cullet then serves as the feedstock for the final melting of the optical-quality glass. The cullet is usually melted in a relatively inert, refractory crucible. The molten cullet is poured onto a cold surface where it fractures into smaller pieces as a result of the thermal stresses. The characteristics of the cullet are far different from those of the final product; the cullet is generally full of bubbles and striae and possibly contains small particles of undissolved component material.

The fractured glass that is produced from the melted cullet is used in the second and final melting. This step is carried out in a platinum crucible of about 40-litre capacity. The use of platinum vessels has long been the standard in the glass industry because platinum is erosion resistant and essentially insoluble in the glass matrix. During the final step, the molten glass is mechanically stirred to homogenize the glass and to remove any striae produced by volatilization. It is also treated to remove bubbles. At the end of the final melt, the glass is cast into a mold and annealed.

Chemistry of Platinum

Even though platinum is largely inert to most molten glasses, it still causes a low level of particulate contamination, generally at a level of less than a few parts per million. Except for the glass used in high-power lasers, this platinum contamination generally does not affect optical performance.

The sources of platinum-particle contamination in glass have been the subject of several previous studies.[5,6] It is generally agreed that the particles are produced by abrasion or wear, by direct dissolution of the platinum crucible and subsequent precipitation of the dissolved platinum, or by vapor-phase transport via a volatile oxide (PtO_2) and redeposition as metallic platinum. In principle, abrasion and wear can be eliminated by care in processing and have usually been ruled out as a source of contamination.

Platinum is an unusual metal in that it forms a volatile oxide (PtO_2) when heated above about 700°C in an oxygen-containing atmosphere.[7,8] The PtO_2 vapor pressure in equilibrium with metallic platinum (at 0.1 MPa O_2) is about 10^5 times greater than that of the platinum metal. At typical melting temperatures, PtO_2 is stable only in gas phase; upon contact with a solid or liquid surface, it redeposits platinum metal.[8]

Therefore, in oxidizing systems, it is possible to transport platinum to the molten glass via PtO_2 and then redeposit it as platinum metal. This mechanism is further confirmed by experiments in which the glass is melted in a ceramic crucible that is physically separated (but in close proximity) to platinum metal.[5] When the melt is carried out in oxygen, platinum particles are found in the glass. On the other hand, when an inert gas is used (e.g., nitrogen), no platinum particles are observed. It would appear, therefore, that one could eliminate platinum-particle formation by preparing glass in an inert environment. Unfortunately, the oxygen released by the decomposition of oxide components of the glass may be sufficient to produce platinum transport and subsequent inclusion formation.[5]

A related study[6] of silicate laser glass reports that platinum solubility increases with oxygen content of the gas during the melt. This study

proposes that platinum dissolution (by the glass) and later precipitation may be the mechanism causing inclusion formation in melts carried out in inert or reducing environments.

Unlike silicate glasses, phosphate glasses are known to have a much higher solubility limit for platinum (several orders of magnitude higher); that is, phosphate glasses can hold much more platinum in solution before it precipitates as metal particles. It was generally thought, therefore, that the platinum would remain in solution in the glass and thus that platinum-inclusion formation would not be a problem in phosphate glasses.

Key Process Variables

A number of variables affect the formation of metallic-platinum inclusions in phosphate glasses. The most important are the oxygen content of the process gas, the melt temperature and temperature uniformity, the processing time, and the platinum solubility in the glass. Experiments were undertaken at both Hoya Optics, Inc., and Schott Glass Technologies, Inc., to examine the effects of these variables on the density of platinum inclusions in their glasses.

Both the Hoya and Schott scientists observed a dramatic reduction of inclusions in the phosphate glass when the oxygen content of the processing gas was increased. This result is illustrated in Fig. 9, in which platinum-inclusion count is plotted as a function of oxygen partial pressure in the process gas. These data are from 0.5-litre test melts of glass carried out at Hoya; a similar trend was observed at Schott for their particular glass composition. Hoya also found that use of a proprietary additive further reduced the inclusion count under the most oxidizing conditions (also shown in Fig. 9). The experiments at Schott and Hoya clearly demonstrate that the oxidizing conditions promote the dissolution of platinum particles in the melt.

Platinum dissolution appears to be a means of eliminating platinum inclusions, but there are several limitations to this method. First, of course, is the solubility limit of platinum in the glass. Fortunately, for phosphate glasses, the solubility limit is quite high (>1000 ppm) under normal processing conditions. Therefore, the glass continues to hold the ionic platinum even while cooling down, and precipitation at that point generally has not been a problem.

A greater concern than the solubility limit is the absorption of 400-nm-wavelength light by the ionic platinum dissolved in the glass. Absorption of light of this wavelength reduces the pumping efficiency of the xenon flashlamps and could affect the laser's performance. We have found that as long as the absorbance at 400-nm remains below 0.2 cm^{-1}, laser performance is not affected. This absorption level corresponds to an ionic-platinum concentration of about 130 ppm.

Schott has found that the processing temperature also

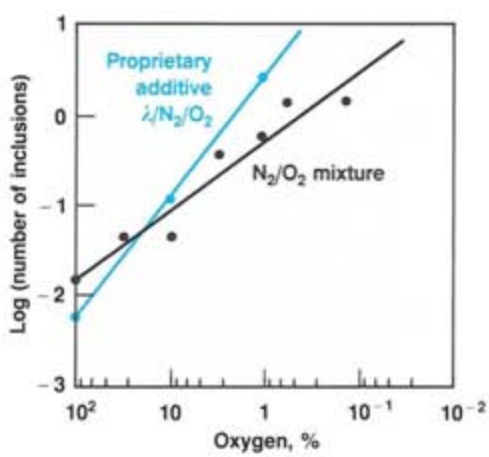


Fig. 9

Relation between oxygen partial pressure in processing gases and measured density of platinum inclusions. The results (data points) are for half-litre melts carried out at about 1100°C. The black line shows the results for an oxygen-nitrogen mixture. The blue line shows the effect of adding a proprietary additive developed by Hoya Optics, Inc., to the process gas.

dramatically affects the inclusion density in their glass (see Fig. 10). Raising the processing temperature from 1100 to 1450°C produces about a 10^3 to 10^4 drop in inclusion density. These results are for a series of half-litre melts in the presence of oxidizing process gas. For this same series of melts, Schott also measured the absorption at 400 nm and found that it remains below the 0.2-cm^{-1} specification.

The effect of processing time on inclusion reduction or absorption at 400 nm is much less dramatic than the effect of temperature. The fact that time has less effect than temperature is not surprising; the rate of dissolution is governed by a typical Arrhenius relationship that depends linearly on time and exponentially on $-1/T$, where T is temperature. Changes in process temperature, therefore, produce a much larger effect than corresponding changes in processing time.

In parallel with the dissolution experiments by the glass vendors, we have also developed a simple one-dimensional model of the process by which the platinum inclusions dissolve in the glass. Using this model, we have calculated that the dissolution time for 10- to 20-μm metallic platinum particles is about 2 to 10 hours at 1100°C (Fig. 11). This calculated result agrees well with experiments by the glass companies. One of the conclusions from our calculations is that the dissolution time is proportional to the square of the particle size. Therefore, trying to dissolve very large particles (100 μm diameter) is not possible with the current processing method. Fortunately, inclusions greater than 20 μm diameter are rare and should not present a problem.

Using results from these and other experiments, both Hoya and Schott have been able to modify their melting processes to eliminate most if not all platinum inclusions. In brief, both companies have sought to control the platinum problem by eliminating outside sources of platinum contamination and by adjusting process conditions (e.g., oxygen partial pressure, temperature, and time) to place and keep all platinum in solution.

Fig. 10

Measured inclusion density as a function of the temperature at which phosphate glass is melted (the experimental temperature range was 1100 to 1450°C). These experiments were carried out by Schott Glass Technologies Inc.

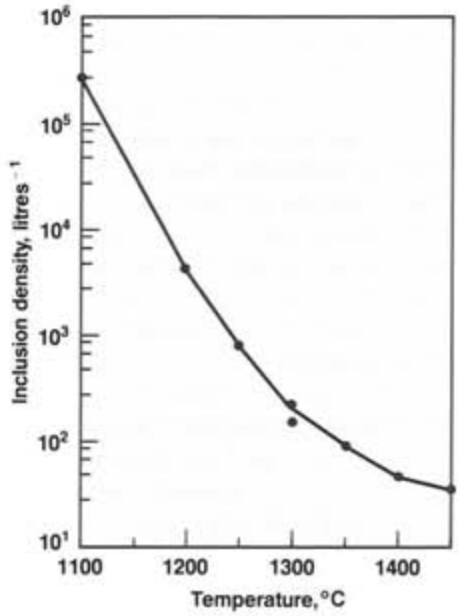
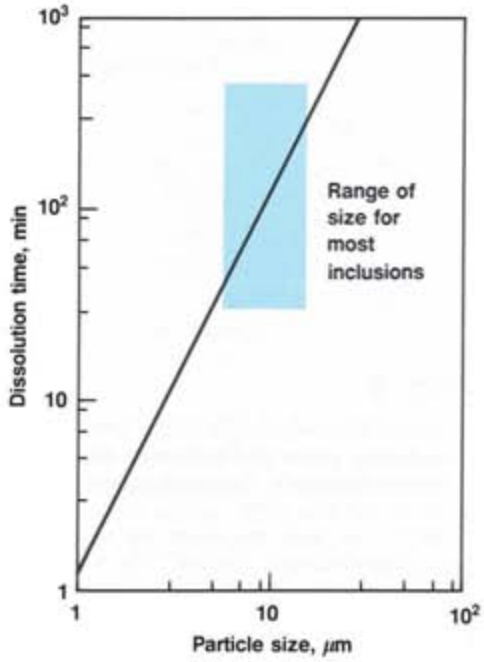


Fig. 11

Calculated dissolution times for platinum inclusions in glass as a function of particle size. Most inclusions are about 10 to 20 μm in diameter and are estimated to take about 2 to 10 hours to dissolve. We obtained these results with a simple, one-dimensional model we developed to simulate platinum particle dissolution.



Replacement of Nova Laser Glass

Considerable progress has been made toward the production of inclusion-free glass. In early December 1985, we tested seven large pieces of glass with a total volume of about 17 to 18 litres (equivalent to about three

large laser disks) and found an average of about 0.2 inclusions per litre of glass; four pieces of glass were completely platinum free. For comparison, the glass disks now in Nova have platinum-inclusion concentrations of between 10 and 1000 per litre; thus the new glass represents about a 100-fold or greater improvement. This would yield large laser disks with an average of only 1 to 2 particles each. (The 31.5-cm and 46.0-cm disks each contain about 7 litres of glass). Using both Nova and our new rapid-scan inspection tool (see the article on p. 24), we tested the samples for damage at two to four times the fluence of the damage threshold.

Hoya and Schott have both embarked on programs to construct new melters embodying the results of their experiments in platinum-particle reduction and including enhancements which should further reduce platinum-particle content—the goal, of course, being none. We anticipate initiating, in mid-1986, pilot runs of about 20 large disks produced by each vendor using the new melting facilities. At that time, we will request a price quotation on production quantities of the glass. Our plan is to obtain from each company replacement glass for the disks it originally supplied. We estimate the replacement will be complete approximately 18 months after the start of production melting.

Key Words: computer code—MAZE, ORION, TOPAZ; glass—laser, neodymium, phosphate, platinum inclusion, siliate; laser—amplifier, damage, glass, Nova.

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