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CHARACTERIZATION OF A NEODYMIUM GLASS ROD DAMAGED IN
THE PLASMA-X LASER

R. Vandervoort, Editor

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CHARACTERIZATION OF A NEODYMIUM GLASS ROD DAMAGED IN THE PLASMA-X LASER

INTRODUCTION

A Nd glass rod was severely damaged during operation of the Plasma-X laser. The rod was manufactured by Owens-Illinois* and is designated ED-2 laser glass. The nominal composition of this type of glass, in weight percent, is: $\text{SiO}_2 = 66.7$, $\text{Li}_2\text{O} = 15.0$, $\text{CaO} = 10.2$, $\text{Al}_2\text{O}_3 = 4.7$, $\text{Nd}_2\text{O}_3 = 3.1$, and $\text{CeO}_2 = 0.5$. The rod (406) was inspected by the Optics Lab before use. Size, straightness, parallelism and flatness of faces, and optical properties were all within specification. The entrance and exit surfaces were inclined at Brewsters angle to the rod axis.

The rod was used in the stage 2 amplifier section of the Plasma-X laser. Normal input was 5 mJ in 50 ps. Final output was about 1 J, and back reflections from the target put 1-J pulses into the rod. Peak power density was approximately 1 GW/cm^2 . The system was subjected to hundreds of pulses. Visual examination of the rod after termination of the experiment showed that both the entrance and exit faces had sustained damage. There was also some internal damage.

The rod was sectioned in the optics fabrication facility as shown in Fig. 1. Analyses were made for Nd content as a function of radial and longitudinal position, crystalline impurities, and volatile impurities. Other sections were used to characterize the surface and internal damage.

*Reference to a company or product name does not imply approval or recommendation of the product by the University of California or the U.S. Atomic Energy Commission to the exclusion of others that may be suitable.

Fig. 1. Sectioned laser rod.

- (1) Damage - scanning electron microscopy
- (2) Damage - light microscopy
- (3) Nd content (radial) - microprobe
- (4) Nd content (longitudinal) - microprobe
- (5) Damage - light microscopy
- (6) Nd content (longitudinal) - microprobe
- (7) Damage - light microscopy
- (8) Nd content (longitudinal) - microprobe
- (9) Nd content (radial) - microprobe
- (10) Damage - light microscopy
- (11) Damage - scanning electron microscopy
- (12) Crystalline impurities - x-ray diffraction
- (13) Volatile impurities - thermal outgassing
- (14) Not used
- (15) Damage - light microscopy
- (16) Not used
- (17) Crystalline impurities - x-ray diffraction
- (18) Nd content (radial) - microprobe
- (19) Volatile impurities - thermal outgassing
- (20) Damage - light microscopy
- (21) Crystalline impurities - x-ray diffraction

NEODYMIUM CONTENT

J. Johnson and J. Wittmayer

The electron probe microanalyzer was used to examine six specimens from a damaged Nd-glass laser rod. The information obtained included verification of the manufacturer's nominal Nd concentration and variations of Nd concentration as a function of specimen radius and length.

The use and description of electron probe microanalyzers is amply discussed in the literature. For the present work we used a Materials Analysis Company Model 400S electron probe microanalyzer interfaced with a Digital Equipment Corporation PDP-8 computer. The major functions of the probe are under program control of the computer: the computer positions the spectrometers, controls the operation of the x-ray detection and signal processing equipment, prepares a data tape compatible with Octopus input to the CDC-6600 computer, and checks and maintains the proper operation of the probe.

In making a quantitative analysis with the electron probe microanalyzer, the intensity of a particular characteristic x-ray line emitted by an element in the unknown specimen is measured and then compared with the intensity of that emitted from a pure-element reference standard under the same conditions. This relative intensity is corrected for background contributions from the other elements present, instrument dead time, mass attenuation effects, secondary and tertiary fluorescence, atomic number effects, and x-ray take-off angle. Quantitative analysis for up to nine elements per specimen can be made simultaneously with the present data reduction computer program called "Microanalysis."

Point count analyses were performed on all specimens while traversing the specimens under the electron beam in 100- μ m increments. Between 40 and 50 analyses were made per traverse, and three traverses were made on each specimen. Table 1 summarizes the data for Nd concentrations.

Figures 2 and 3 illustrate variations in Nd concentration vs distance in microns for both "best" and "worst" case distribution for radial and longitudinal tracings.

Note that quantitative microprobe analysis is based on 100% composition. Since it is impossible for electron beam microanalyzers to detect x rays from lithium, it was necessary to accept the nominal Li_2O value provided by the manufacturer as a working value of the lithium concentration for the computer program. Deviations from this value would tend to increase the relative error of the absolute value of the Nd analysis, but would not substantially affect the relationship of one Nd analysis to another in the same specimen.

Table 1. Neodymium content in ED-2 laser glass.

Specimen ^a	Traverse	Nd (wt%)	Average Nd (wt%)
3 radial	1	3.0579 ± 0.0553	3.1131 ± 0.0638
3 radial	2	3.1256 ± 0.0781	
3 radial	3	3.1536 ± 0.0997	
4 long.	1	3.3682 ± 0.3419	3.2451 ± 0.1960
4 long.	2	3.0442 ± 0.0862	
4 long.	3	3.3106 ± 0.2741	
6 long.	1	3.3106 ± 0.2741	3.1131 ± 0.0981
6 long.	2	3.0047 ± 0.0853	
6 long.	3	3.0648 ± 0.0742	
8 long.	1	3.0387 ± 0.0784	3.1111 ± 0.0871
8 long.	2	3.0916 ± 0.0640	
8 long.	3	3.2022 ± 0.1550	
9 radial	1	3.2000 ± 0.1635	3.1799 ± 0.0944
9 radial	2	3.2172 ± 0.1676	
9 radial	3	3.1153 ± 0.0813	
18 radial	1	3.3392 ± 0.3072	3.2508 ± 0.1596
18 radial	2	3.3056 ± 0.2678	
18 radial	3	3.1136 ± 0.0601	

^aSee Fig. 1 for specimen location.

Fig. 2. Distribution of Nd in a glass laser rod in the radial direction.

Fig. 3. Distribution of Nd in a glass laser rod in the longitudinal direction.

CRYSTALLINE IMPURITIES

G. Smith

Three semicircular disks, cut from a Nd-doped glass rod, were examined on a powder diffractometer to detect any crystalline regions greater than about 1000 Å. Such regions constitute an inhomogeneity which might lower the radiation stability of the material. All results were negative: no sharp x-ray diffraction maxima were observed; only the broad haloes, characteristic of a glassy or amorphous material, were detected. The lower limit of detection is estimated to be about 0.5 vol% and may be even less since the glass matrix contributes no interfering lines.

In future work the extremely small-angle scattering region could be investigated. In this region, inhomogeneities in the size range 20 to 1000 Å show up because of density differences rather than crystalline diffraction effects, as in the higher angle region. The amorphous haloes could also be studied to see how the glass network is modified by various dopants and to provide an analytical tool to determine the amount of doping.

VOLATILE IMPURITIES

R. Bedford

Two samples of neodymium glass from a damaged laser rod were analyzed for evolution of volatile constituents while they were heated from room temperature to 1100°C. The experiments were made to determine if dissolved gases in the rods could be involved in the damage mechanism.

The samples which weighed about 250 mg each were cleaned, crushed, and weighed before analysis. Samples 13 and 19 were taken from the entrance end and the center portion of the rod respectively. No damage effects were detected in either sample by microscopic examination. The material was spread in a thin layer on platinum trays in a platinum crucible. The analysis was done in a vacuum furnace by heating at about 10°C per minute and recording the system pressure. The composition of gas was monitored continuously with a quadrupole mass spectrometer scanning from mass 1 to 50. Several times during each run scans were made from mass 10 to 150. The pressure records and spectra were compared to background runs made under the same conditions without a sample in the crucible.

The only gas detected evolving from the samples was CO₂. Carbon dioxide was released from both samples over the temperature range 350° to 450°C. The maximum rate of evolution occurred at 400°C, where the pressure was 33 μPa, or about twice background for 22, and 53 μPa, or about 4 times background for 13. It is estimated that 0.02 ± 0.01 wt% of CO₂ was evolved from 22 and 0.04 ± 0.02 wt% CO₂ from 13 in this temperature interval. There were no differences in gas evolution between the samples other than the somewhat greater CO₂ release from 13 at temperatures from 350° to 450°C. The intensity at mass 44 (CO₂⁺) was higher than background at all temperatures above 350°C for both samples, indicating a continuous, slow release of CO₂ as they were heated. The dissimilarity between the two samples could be related to a difference in distribution of carbonates in the glass rod.

Both samples began to release CO₂ in irregular bursts after reaching 920°C. This continued as the temperature increased to 1100°C and during the 30-min hold time at that temperature. This outgassing behavior is typical of thick samples of moderately viscous liquids such as glass. Apparently the viscosity of the glass is low enough for it to be drawn into droplets by surface tension at 920°C. After the experiments the glass residue was in the form of one or two small drops on each tray.

The base pressure during the intermittent CO₂ release was about 19 μPa. Typical bursts raised the pressure to 33 μPa and the largest raised the pressure to 70 μPa momentarily. All detectable peaks were monitored and the only ones with irregular intensities were at masses 28 and 44. The changes in intensity for mass 28 were much smaller than for mass 44, and they probably were due only to CO⁺ from fragmentation of CO₂.

Results from this work showed that the glass rod contains significant amounts of residual carbonates or trapped CO_2 . It is possible that these impurities might lower the threshold for damage. Laser experiments with carbonate free glass are recommended to test this possibility.

DAMAGE

R. Vandervoort, W. Steele, and S. DiGiallonardo

Surface damage on the entrance and exit faces was studied using the scanning electron microscope. A usable magnification from 15 to 15,000 X plus good depth of focus made the SEM a suitable instrument for this work. Bulk damage was observed with a conventional optical microscope using transmitted light.

There were no significant differences between the number of damage sites or types of damage found on the entrance and exit surfaces. This result was not unexpected since the surface damage thresholds are the same for laser rods with the Brewster angle geometry. Both surfaces exhibited localized zones of microspalling, melting, blow holes from gas evolution, and feathered tracks of dielectric breakdown (Fig. 4). Only one type of bulk damage was found. This consisted of a uniform filamentary damage track associated with one or more larger zones of cracking or melting. Examples of internal damage are illustrated in Fig. 5.

The filamentary damage is characteristic of failure by self-focusing of the laser beam. When self-focusing occurs, electrons from ionized impurity atoms and small inclusions can be accelerated under the applied optical field until atoms of the glass matrix are ionized. Then a concentrated electron avalanche occurs, and the glass sustains damage in the zone of the intense flux of electrons. The large damaged region surrounding the filamentary track probably results from internal fracturing and/or melting due to localized heating at minute inhomogeneities in the glass. These inhomogeneities, which absorb energy, are probably smaller than 1 μm , since none were observed optically at magnifications up to 1000 X.

The concentration of damage was much higher at the surfaces of the laser rod than in the bulk. Detailed examination of the damage surfaces revealed two potential causes for this difference in behavior. First, a number of inclusions were found embedded in the surface of the glass (Fig. 6). Second, many small holes were observed in regions that had melted (Fig. 4c).

Inclusions in the surface could be centers for damage since the particles would undoubtedly absorb large amounts of energy during operation of the laser. Thermal stresses from localized rapid heating could generate shock waves causing microspall. The origin of these inclusions is uncertain, but they are probably polishing compounds used to prepare the ends of the laser rod. The holes in zones that had undergone melting indicated evolution of some volatile specie in the glass. Two likely candidates are CO_2 or water vapor. The CO_2 could either come from residual carbonates, if used in the manufacture of the glass, or from melting the glass in the presence of CO_2 . The H_2O could be absorbed from the air or from the slurry used to polish the ends of the rod. Carbon dioxide is the most likely specie since the outgassing experiments showed that CO_2 was evolved from the glass when it was heated.

50 X

(4a)

1000 X

(4b)

5000 X

(4c)

1000 X

(4d)

Fig. 4. Surface damage of an ED-2 glass rod caused by a laser beam. (a) Microspalling (b) Melting (c) Holes from gas evolution (d) Dielectric breakdown.

100 X

(5a)

100 X

(5b)

Fig. 5. Internal damage in an ED-2 glass rod caused by a laser beam. Photomicrographs are from section 7 (see Fig. 1). The entrance surface is not shown; it is to the left of the figures.

1000 X

Fig. 6. Surface inclusions on ED-2 laser glass.

Damage related to surface inclusions could be eliminated by careful inspection of the ends of the laser rods before use. Rods with surface inclusions could be discarded or chemical or ion polishing could be used to remove the particles. Vacuum outgassing at room temperature could remove absorbed water from the surface layers of the glass if it were found that outgassing raised the threshold for damage of surfaces free from inclusions and other imperfections such as scratches. However, this process might have to be repeated at periodic intervals since water from air could be reabsorbed. Carbonate free glass should be used to eliminate damage from evolution of CO₂ gas.

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