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SURFACE ANALYSIS OF POLISHED FUSED-SILICA
LASER LENSES BY ION-SCATTERING SPECTROMETRY

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SURFACE ANALYSIS OF POLISHED FUSED-SILICA LASER LENSES BY ION-SCATTERING SPECTROMETRY

INTRODUCTION

New advances in high-powered glass lasers, particularly the NOVA system, have resulted in a need for lenses having higher damage threshold values than those now available. It is currently thought that surface contaminants on the lenses are responsible for initiating part of the damage. These contaminants are apparently introduced during the final polishing stages. In this study, we used ion-scattering spectrometry (ISS) to identify contaminants arising through the use of different polishing techniques.

Five lenses were studied, each having undergone different polishing procedures. The first lens was not polished after receiving it from the manufacturer (#381). Ion microprobe data were available for this lens, and they were compared to ISS results. The second lens had been polished with rouge, a polishing compound no longer in use (#796). This sample served as a further check on the ISS results. The third lens was studied as received from the manufacturer--with no handling or cleaning (#802). The final two lenses had both been polished using high-purity ceria, cerium oxide (#800 and #801). The difference between these two was that #800 was polished using a nylon lap, and #801 was polished using pitch as a lap. The 800-series lenses were all made from the same batch, and constituted the major part of the investigation.

EXPERIMENTAL

The lenses used in this study were fused silica, 5 cm (2 in.) in diameter and 1 cm (3/8 in.) thick. A special holder constructed of aluminum was installed in the ISS vacuum chamber to allow easy insertion of the lenses (Fig. 1).

The base pressure of the system was in the 10^{-9} -Torr range. The chamber was back-filled with a noble gas to the 10^{-5} -Torr range. ^4He , ^{20}Ne , and ^{40}Ar were used in this study as the source gases for the ion gun.

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Figure 1. A lens mounted on the aluminum holder in the analysis position of the ISS.

Two differently focused ion beams were used: (1) a beam about 0.3 mm in diameter with 1 nA ($0.35 \mu\text{A}/\text{cm}^2$) current, and (2) a beam about 1 mm in diameter with 40 nA ($1.3 \mu\text{A}/\text{cm}^2$) current. It took over 24 hours for the latter beam to profile through the polished layer on one of the lenses. If we assume the layer to be 50-nm thick, this works out to a sputtering rate of about 35 pm/min ($0.35 \text{ \AA}/\text{min}$).

Because the lenses were insulators, it was necessary to use an incandescent tungsten filament, located above the sample, to flood the surface of the lens with electrons. This neutralized any local charged regions resulting from the ion bombardment. Figure 2 shows the effect of varying the amount of charge neutralization on an ISS spectrum. Figure 2(a) shows no charge neutralization. The peaks were reproducible, and their location was directly proportional to the incident beam energy. As the neutralization was increased, the high energy peaks merged together [Figures 2(b)-(e)]. All this occurred within 10 mA of the filament current value of 1.2 A. Figure 2(e) shows three scans taken within a twenty-minute period, with all conditions held constant for each. The neutralization current was apparently near a threshold value, with minor fluctuations causing great differences in the resulting spectra. With an immeasurable increase in filament current, the yield of scattered ions dropped by a factor of about 25 [Figure 2(f)]. With further increases in filament current, the broad peaks were transferred into the peaks characteristic of oxygen and silicon [Figures 2(g,h)]. Any further increase in filament current had no effect on the spectrum, thus suggesting a saturation level had been reached. It should be noted that, in Figure 2(h), the peak location for silicon was below the theoretical value of $E/E_0 = 0.607$ where E is the reflected ion beam energy and E_0 is the incident energy. Thus, some adjustment of the filament current to a value just below saturation was needed to position the silicon peak properly.

Once the silicon and oxygen peaks were positioned properly, the next step was to identify the contaminant peaks. With $^4\text{He}^+$ as the probe ion, elements of mass less than that of chromium (52 amu) were identified with little ambiguity. The peaks for elements of higher mass are poorly resolved, and could be assigned to a range of elements at best. Improved mass resolution was obtained by using a larger mass ion, $^{20}\text{Ne}^+$, but some low-concentration contaminants that showed up under $^4\text{He}^+$ bombardment were undetectable when $^{20}\text{Ne}^+$ ions were used. This may be due to a different

Figure 2. Effect of sample charging on ISS spectra using 1.5 keV $^4\text{He}^+$ ions on fused quartz: (a) no charge neutralization; (b) charge neutralization filament current of 1.2 A; (c) slight increase over 1.2 A of filament current; (d) additional small increase in filament current; (e) at threshold value of charge neutralization; (f) slightly above threshold value of filament current; (g) filament current of 1.22 A; and (h) at saturation value for the charge neutralization filament current.

neutralization rate for the incident neon ions. Further adjustments of the beam parameters (energy, current density, etc.) might lead to detection of the contaminants. No contaminants were detected when $^{40}\text{Ar}^+$ ions were used.

As described above, the procedure for interpreting ISS spectra is to align the peak for some element known to be present in the target with its theoretical E/E_0 value. Then the identities of the elements giving rise to the remaining peaks may be determined from the E/E_0 values at which they occur. Unfortunately, this scheme is not infallible. Peaks from other elements known to be present do not invariably occur at their theoretical E/E_0 values. For example, the ISS was calibrated for $^4\text{He}^+$ ions scattered from tantalum (181 amu); the beam was then directed at a gold target (197 amu). The peak for gold fell at $E/E_0 = 0.96$, while its theoretical value is 0.932. Both samples were at the same distance from the ion gun (thus ensuring the same scattering angle for the detected ions), and were under the same conditions of charge neutralization.

To minimize the problem of the shifted peaks, some samples of known elements were placed on the top of each lens. Comparison of the peak locations for these known elements with the peak locations for the unknown contaminants allowed the identity of the contaminant to be narrowed down to a small number of possible elements.

RESULTS

LENS #381 (UNPOLISHED AFTER RECEIVING)

The main purpose in examining this lens with ISS was to determine what information was obtainable. With the charging phenomenon, it was not known if even the major constituent elements could be identified. Once the charge neutralization system was properly operating, the characteristic peaks for silicon and oxygen were clearly resolved, along with several unknown peaks. Figure 3 shows a typical $^4\text{He}^+$ spectrum for #381. The unknown peaks were identified as magnesium, and potassium or calcium (or both). Scans previous to this one also showed fluorine and sodium, which were cleaned off quickly under ion bombardment. The fluorine was not found on initial scans of the lens, but appeared following ion bombardment of a piece of aluminum foil and a

Figure 3. A spectrum of lens #381 (unpolished after receiving) using 1.5 keV ^4He ions.

bake-out of the chamber. The aluminum foil was found to have a large amount of fluorine on its surface. Either ion sputtering or the bake-out must have caused the fluorine to re-deposit onto the lens. No fluorine was detected in any of the subsequent studies of other lenses.

Comparison of ISS and ion-microprobe data showed that, with the exception of aluminum, both techniques had identified the same elements as major contaminants on the surface. The ISS is unable to distinguish aluminum on a quartz lens due to the large size of the silicon peak.

LENS #796 (POLISHED WITH ROUGE)

This lens was used to test if any impurities due to the polishing compound could be detected. In particular, since the compound was rouge (iron oxide), there should be an iron contaminant. Figure 4(a) shows a $^4\text{He}^+$ spectrum for #796. The iron contaminant was easily detected, along with potassium or calcium, and two high mass elements.

Since the opposite side of the lens had been coated with gold, it was possible that the highest energy peak resulted from gold that had migrated from the coated side. To check this possibility, a gold foil was laid on top of the uncoated side of the lens, and the spectrum was calibrated such that the $^4\text{He}^+$ on gold peak fell at its theoretical value of $E/E_0 = 0.932$ [Fig. 4(b)]. A scan was then taken on the lens, which showed the element to have a larger mass than that of gold. Scans done with $^{20}\text{Ne}^+$ (also calibrated with the gold foil) demonstrated the element to be more massive than gold (Fig. 5). The $^4\text{He}^+$ peak location corresponds to a range of elements from lead (207 amu) to lawrencium (260 amu). The $^{20}\text{Ne}^+$ spectrum narrows this down to an element between radon (222 amu) to thorium (232 amu). No peaks were seen when $^{40}\text{Ar}^+$ was used.

The remaining unknown peak on the $^4\text{He}^+$ spectrum could be due to elements from tin (119 amu) to cerium (140 amu). The $^{20}\text{Ne}^+$ spectrum gave a range from tin (119 amu) to iodine (127 amu).

All of the contaminants were eventually sputtered off the lens [Fig. 4(c)], with iron remaining the longest, indicating that the contaminants were contained in the polished region.

Figure 4. Spectra of lens #796 (polished with rouge) using 1.5 keV ^4He ions: (a) lens alone; (b) with adjacent gold foil; and (c) sputter-cleaned.

Figure 5. Spectra of lens #796 (polished with rouge) using ^{20}Ne ions and calibrated with gold foil.

LENS #802 (NOT POLISHED AFTER RECEIVING)

Since all of the lenses had potassium or calcium as a contaminant, we decided to examine a lens which was not polished nor handled in any way after being received from the manufacturer. Lens #802 arrived in a sealed plastic container, and was opened just prior to loading into the ISS chamber. No cleaning was done, and plastic gloves were used to handle it. Figure 6(a) is a $^4\text{He}^+$ spectrum for this lens, and shows large potassium or calcium contamination. In addition, there are two higher mass peaks. $^{20}\text{Ne}^+$ analysis [Fig. 6(b)] indicates the higher mass peak is in a range from cesium (133 amu) to praseodymium (141 amu).

The other peak is in a range from cobalt (59 amu) to zinc (65 amu), based on the $^4\text{He}^+$ spectrum. The corresponding $^{20}\text{Ne}^+$ peak was undetectable.

LENS #800 (POLISHED WITH 99.9% PURE CERIA ON A NYLON LAP)

Lens #800 and lens #801 had both been polished with high purity ceria (ceum) after receipt from the manufacturer. The difference between the two was the material used as a lap during the polishing: pitch for lens #801, and nylon for #800.

Figure 7(a) is a $^4\text{He}^+$ spectrum taken near the edge of lens #800. Scans at several spots across the diameter of the lens showed that the magnesium contaminant was present only near the edge of the lens. This contaminant was probably introduced during handling. As for the other lenses, there is a potassium or calcium contaminant, and cerium is present from the polishing compound. The peak at $E/E_0 = 0.775$ is attributable to one or more elements in the range from chromium (52 amu) to nickel (59 amu). This contaminant varied in concentration across the lens; in some spots it was the major surface contaminant. Once sputtered off the lens, another peak was revealed at about $E/E_0 = 0.83$ [Figure 7(b)]. This peak was very weak in intensity, and often appeared as a shoulder on the left of the cerium peak [Figure 7(c)]. Thus, it is difficult to assign a narrow range of elements to it. Assigning it a range from $E/E_0 = 0.825$ to 0.850 corresponds to a range of elements from germanium (73 amu) to rubidium (86 amu). The cerium contaminant is clearly seen in the $^{20}\text{Ne}^+$ spectrum, but none of the other contaminants appear.

Figure 6. Spectra of lens #802 (as received from manufacturer): (a) using ${}^4\text{He}^+$; and (b) using ${}^{20}\text{Ne}^+$.

Figure 7. Spectra of lens #800 (polished with 99.9% pure ceria on a nylon lap) using $^4\text{He}^+$: (a) a spectrum near the lens edge; (b) the peak at $E/E_0 = 0.775$ is sputtered away; and (c) the $E/E_0 = 0.830$ peak as a shoulder on the cerium peak.

LENS #801 (POLISHED WITH 99.9% PURE CERIA ON A PITCH LAP)

A $^4\text{He}^+$ spectrum for #801 is shown in Fig. 8. A peak corresponding to cerium is present, as is the potassium or calcium peak. In addition, there is a rather large contaminant located at about $E/E_0 = 0.810$. The elements which could cause this peak are zinc (65 amu), gallium (70 amu), or germanium (73 amu). Copper (64 amu) was ruled out when a comparison was made with the peak location for copper from a standard copper sample. The $^{20}\text{Ne}^+$ spectrum showed the cerium contaminant, but no other elements were detected.

CONCLUSIONS

This study revealed both the benefits and the problems of using ISS for studying contamination of lens surfaces. In comparison with ion-microprobe analysis, both techniques detected essentially the same contaminants. Yet, ISS has an advantage in that insulating samples can be studied more easily. The ion microprobe required metallic screens and masks over the lens to avoid charge build-up. Several different screens were used to determine what elemental signals were due to the screen itself. In ISS, the charging problem was eliminated through use of the charge neutralization filament. It is also possible with ISS, using small diameter beams, to determine if there is any correlation between damaged regions (blisters and craters) and changes in the relative distribution of contaminants across the surface of the lens.

The disadvantage of using ISS is that accurate elemental identification of the contaminants may well prove impossible. If the ISS were combined with another surface analysis tool, such as Auger or SIMS, then this problem would be eliminated. If used alone, the best one could hope for in some circumstances is a range of possible elements contributing to an ISS peak.

Even so, there is information to be gained with ISS. Cerium was found on those lenses polished by cerium oxide, lenses #800 and #801. Lens #802, as received from the manufacturer, had a contaminant located near cerium, and can probably be identified as cerium, since cerium oxide is a common commercial polishing compound. Lens #802 also revealed the source of potassium (a contaminant found on all lenses studied) to be the manufacturer. Potassium and calcium are common additives to glass, yet ISS did not reveal them in the

Figure 8. Spectrum of lens #801 (polished with 99.9% pure ceria on a pitch lap) using $^4\text{He}^+$.

bulk, suggesting that they were introduced during polishing or handling of the lenses. All three lenses had additional contaminants, also present only in the polished region. Lens #800, polished on a nylon lap, had more contaminants than the others. Examination of a piece of the nylon material with ISS revealed carbon, oxygen, sodium, chlorine, and cerium. The cerium was expected since the nylon lap was used to hold the cerium oxide polishing compound. The sodium and chlorine were present only on the outer surface, probably resulting from handling. But none of the other contaminants found on lens #800 appear to be due to the nylon lap. This would suggest that either the contaminants were present on the lens when received, were introduced during handling, or were present in the cerium oxide powder (although it should have shown up in the powder remaining in the nylon).

If this study were to be continued in the future, the following changes might prove beneficial:

- 1) Examine each lens with ISS and with some other surface analytical technique, to pinpoint elemental identities;
- 2) Examine each lens direct from the manufacturer prior to performing further polishing procedures; and
- 3) Examine all of the polishing materials used (laps and powders) to check on contamination in the laboratory.