AN EVALUATION OF ALTERNATE STAINLESS STEEL FINISHING TECHNIQUES FOR LIQUID CELL OPTICAL MIRRORS USED IN THE MID-INFRARED

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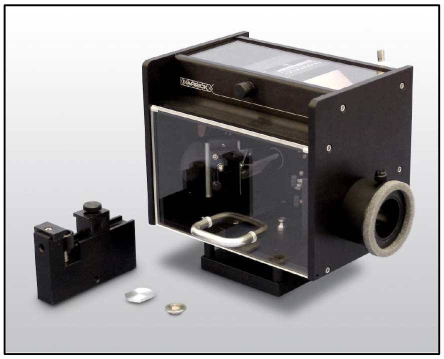

Figure 1. The Seagull™ Variable Angle Reflectance Accessory.

Element	Percent by Weight (max.)
Carbon	0.03
Manganese	2.00
Silicon	0.75
Chromium	16.00 18.00
Nickel	10.00 14.00
Molybdenum	2.00 3.00
Phosphorus	0.045
Sulfur	0.030
Nitrogen	0.10
Iron	Balance (approx. 65.5)

Table 1. Chemical composition of 316L stainless steel[2,3].

ABSTRACT

A previous report[1] demonstrated the feasibility of a new liquid cell capable of both variable angle internal and external reflection spectroscopy. One of the drawbacks in the external reflection mode was poor optical throughput caused by the diffuse, bead-blasted surface of the cell bottom. In this work, alternate techniques for producing reflective finishes are evaluated. The 316L stainless steel alloy is chosen as the prospective cell body material best suited for ready availability, low cost, and chemical inertness. This is a low-carbon variant of 316 stainless steel with additional advantages: higher machinability, greater corrosion resistance at weld points, and reduced tendency to crack after welding. There are no significant cost differences between 316 and 316L stainless steels. Rectangular test samples are made with the following finishes (after machining): none; bead-blasting; electropolishing; grinding and electropolishing; and grinding and optical polishing. Diamond turning, another common mirror finishing technique, cannot be done on stainless steel. Likewise, as the liquid cell is likely to have input/output flow ports, plating, which is subject to delamination and inefficient coating in taps and narrow diameters, is not evaluated. A variable angle external reflection accessory, installed in an FTIR spectrometer, is used to measure the samples for reflectivity in the mid-infrared region at angles of 5° to 85°. Conclusions are made on the best performance and cost tradeoffs.

EXPERIMENTAL

All spectra were taken with a Thermo/Nicolet Nexus™ 670 FTIR spectrometer equipped with a DTGS detector and a standard KBr mid-IR beamsplitter and using Thermo/Nicolet Omnic™ Version 6.1 software. All spectra were run at 4000 to 400 cm^{-1}, using a velocity of 0.6329

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Finish	Avg. Refl (%R)	Samp/Samp St. Dev. (%R)	Same** Samp St. Dev. (%R)	Wave-length St. Dev. (%R)	Angle St. Dev. (%R)	Slope (%R/cm^{-1} x 10^{-3})	Corr. Coeff.
Machine	29.5	7.7	0.2	4.5	14.6	-3.25	-0.988
Machine, Bead Blast	31.6	1.1	0.1	11.0	16.9	-7.68	-0.903
Machine, Electropolish	79.5	6.2	0.4	5.9	4.0	-4.54	-0.982
Machine, Grind, Electropolish	54.4	1.6	0.2	12.8	12.1	-9.45	-0.936
Machine, Grind, Optical Polish (Optics Masters)	85.7	0.4	0.2	3.6	2.5	-2.80	-0.996
Optical Polish (Manasota Optics)	85.9	---*	---*	3.6	2.4	-2.74	-0.995

Table 2. Reflectivity properties of various finishes. *Only one sample was available. **Runs with same sample at 45°.

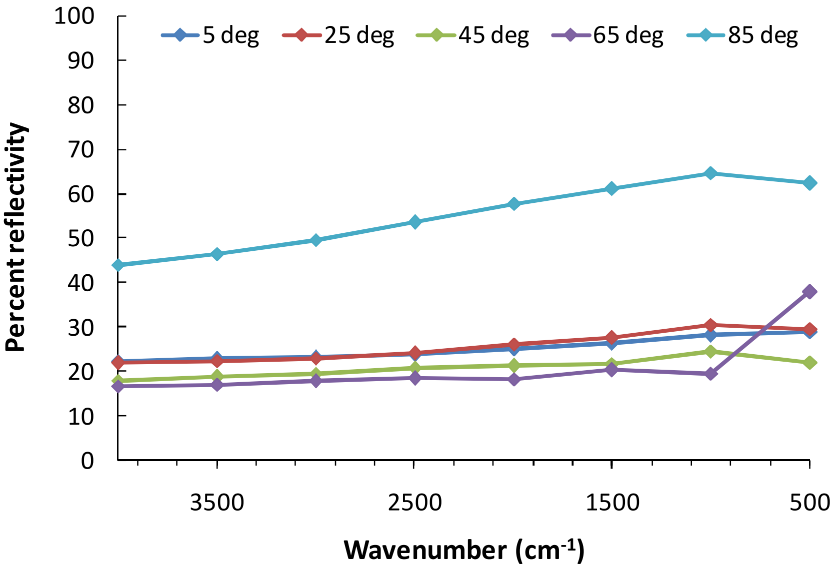

Figure 2. Machine finish reflectivity at various angles.

cm s^{-1}, Happ-Genzel apodization, Mertz phase correction, and no zero filling. The aperture was set to 100 (fully open). The gain was set to 1. The resolution was set to 16 cm^{-1} to allow faster data acquisition. Spectra were derived from 64 co-added scans. A background spectrum was taken prior to each sample spectrum using a front surface aluminum on glass mirror (Harrick Order No. MOP-115; Harrick Part No. 144-305) with dimensions of 3.2 x 50 x 25 mm. The purge inputs of the spectrometer and the installed accessory were connected to filtered air (water and carbon dioxide removed) produced by a Parker Balston Model 75-62 FT-IR Purge Gas Generator at 40 SCFH.

The Harrick Seagull™ variable angle reflection accessory (SEA-NI8) (see Figure 1) was installed in the sample compartment of the FTIR spectrometer. The standard external reflection sample holder was used. This accessory allows the reflection angle to be changed from 5° to 85°. The Seagull™ was first aligned at 45° using the front surface aluminum mirror. Then data were collected from 5° to 85° at 20° increments.

Samples consisted of test pieces machined from 316L stainless steel (Order No. 9195K11; McMaster-Carr). This grade of 316 stainless steel, with lower carbon content, is less subject to corrosion and to fault formation at welds. The chemical composition of this material is given in Table 1. Each piece was machined to the following dimensions: 3.1 mm thick x 50 mm wide x 25 mm long. Three test pieces for each finish type were prepared and one reading at each angle for each piece was made.

An additional set of tests was conducted on the first sample of each type to determine the effect on variability by simply removing and reinstalling the sample and readjusting the angle of incidence. Although all samples were run at an angle of incidence of 45°, the angle was moved away from 45° by about 10°, then returned to 45° for the next run. In this manner, the sample and instrumental manipulations described in the previous paragraph were duplicated in order to determine the variability which resulted from such manipulations alone. This variability could then be compared with that obtained by the regular analysis in order to shed further light on sample to sample variations.

The five finish types were: machining; machining/bead-blasting; machining/electro-polishing; machining/surface grinding/electro-polishing; and machining/surface grinding/optical polishing. The specification for the machining operation was at least 32 µin RMS surface roughness. The specification for the subsequent surface grinding operation was at least 16 µin RMS surface roughness. The specification for optical polishing was 80/50 scratch/dig.

Machining to 32 µin (0.80 µm) RMS surface roughness was done in-house. Bead-blasting was done in-house using a special cabinet (Model 3824; Cyclone Manufacturing; Dowagiac, MI;

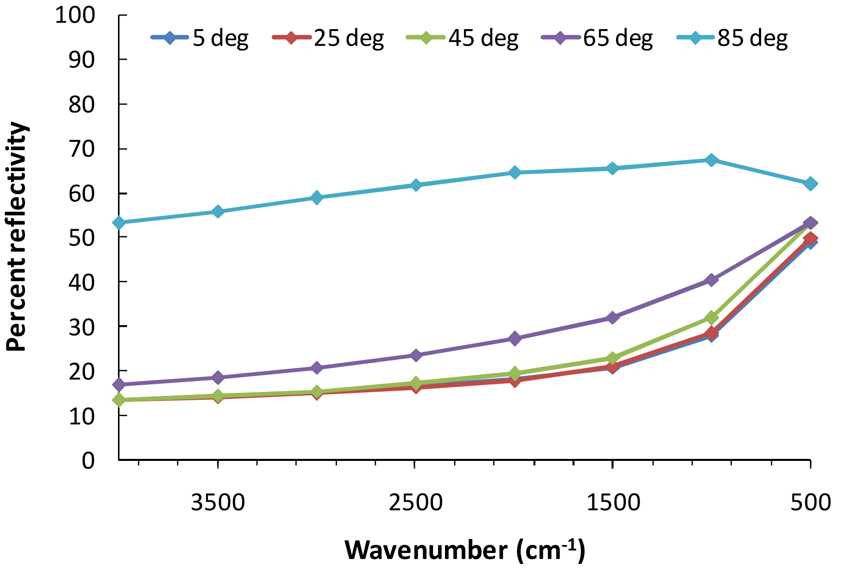

Figure 3. Machine/bead blast finish reflectivity at various angles.

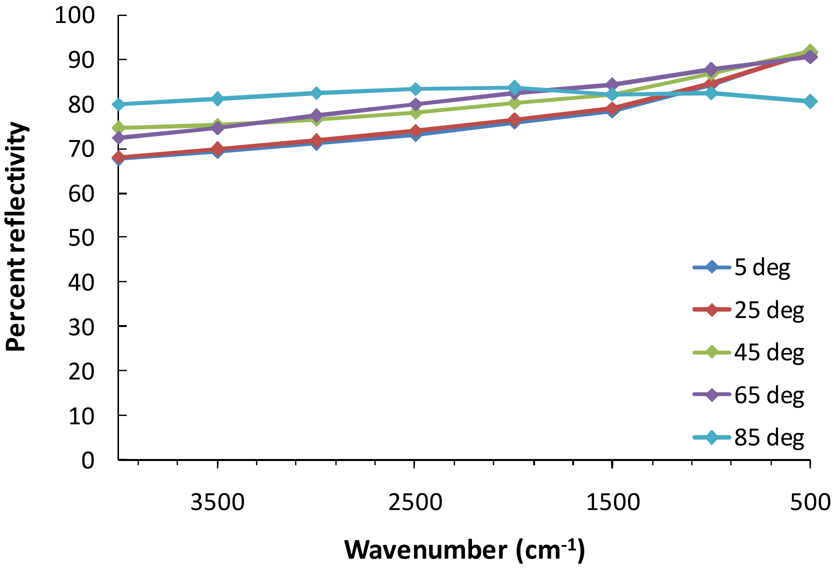

Figure 4. Machine/electropolish finish reflectivity at various angles.

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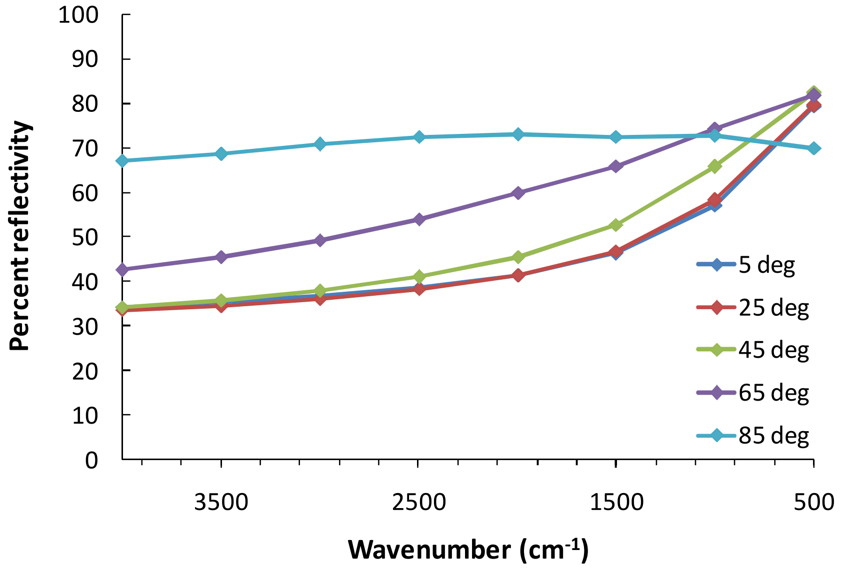

Figure 5. Machine/grind/electropolish finish reflectivity at various angles.

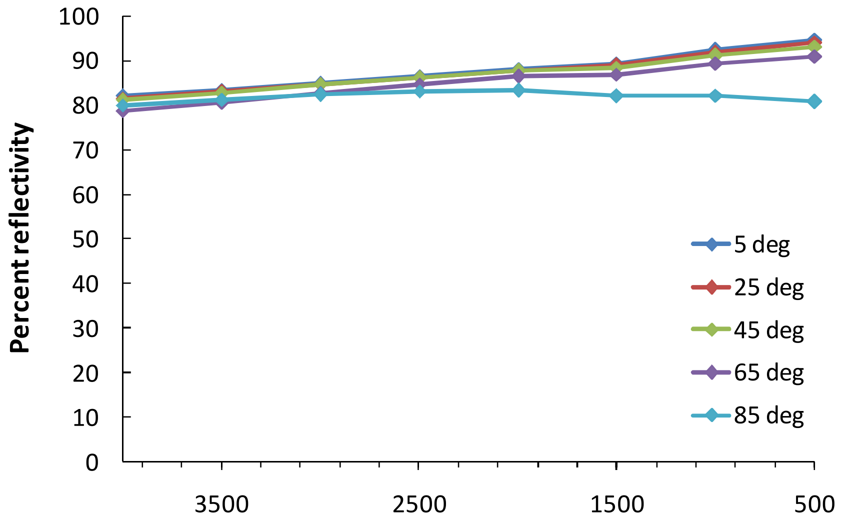

Figure 6. Machine/grind/optical polish finish reflectivity at various angles.

www.cycloneblasters.com) operated at 75 psi compressed air pressure. Glass beads from the same company of 60 to 100 grit (254 to 122 µ diameter) were used in the cabinet. Surface grinding to 16□ µin (0.40 µm) RMS surface roughness was done by KAF Manufacturing (Stamford, CT; www.kaf.com). Optical polishing to 80/50 scratch/dig was done by Optics Masters (Poway, CA; www.opticsmasters.net). Electropolishing was done by Electrobright (Macungie, PA; www.electrobright.com). One optical polish sample, without premachining or pregrinding, was produced by Manasota Optics (Sarasota, FL; www.manasotaoptics.com).

RESULTS AND DISCUSSION

Reflectivities for each of the five finishes are shown in Figures 2 through 6, which plot the average reflectivities of three samples vs. wavelength for 5° to 85° at 20° increments. Figure 7 shows the curves for all finishes with the results averaged across all angles. Various overall reflectivity properties are summarized in Table 2.

Comparing, in Table 2, the sample-to-sample standard deviations (± 0.4 to ± 7.7 %R) with the lower results obtained from simple sample/angle manipulation (± 0.1 to ± 0.4 %R) indicates that the former set of values is a real indication of variability between samples of the same finish.

In general, the characteristics that are sought for the stainless steel mirrors are high reflectivity; low sample-to-sample and angle variability; and a flat response of reflectivity vs. wavelength. The results presented in Table 2 and Figures 2 through 7 indicate that the optical polish is superior to all of the others in these categories.

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(The negative slopes indicated in Table 2 are due to the fact that, in general, the reflectivities increase with decreasing wavenumbers.) It would be this finish that would be chosen for use as a mirror in liquid cells for the mid-infrared. Although the machine/electropolish finish appears, as second best, a viable alternative, it is probably too sensitive to the underlying machine finish to be a reliable production alternative. Hence, the most expensive process, optical polishing, is the only practical choice.

A comparison of the data from the machine/electropolish finish with that of the machine/grind/electropolish finish indicates that the former is superior and that the grinding process may actually have been detrimental. The fact that the one optical polish sample produced by Manasota Optics, in which no pregrinding was done, was almost identical to the optical polish samples produced by Optics Masters, which had pregrinding, indicates that pregrinding can be eliminated in future optical polishing.

The figures and Table 2 do show some interesting additional characteristics. High reflectivity jumps at high angles are shown for the machine, machine/bead-blast, and machine/grind/electropolish finishes. The bead-blast finish shows very good sample to sample reproducibility but very poor reflectivity. (This bead-blast finish was used in the previous study.[1]) Electropolishing does produce an improvement in reflectivity as seen by a comparison between the machine and machine/electropolish finishes.

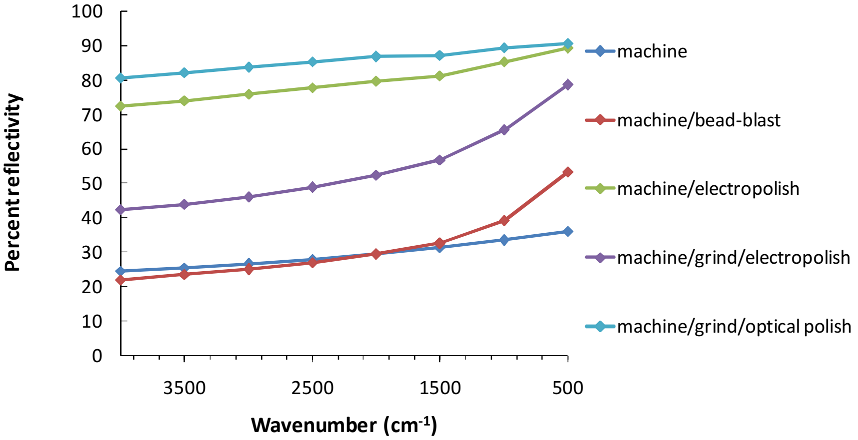

Figure 7. Reflectivity of various finishes (average from 5° to 85°).

With any stainless steel, including 316L, there is a high iron content (65%). See Table 1. This fact precludes the use of the economic diamond turning process, typically used on aluminum and other non-ferrous metals to produce mid-infrared mirrors.[2] An alternate to stainless steel for a corrosion resistant machinable metal alloy is Hastelloy (Haynes International; Kokomo, IN). Although there are several diffent grades of this material, only one, Hastelloy B-3, has a sufficiently low iron content (1.5%). See Table 3.

Future work will determine if Hastelloy B-3 is diamond-turnable. If it is, the reflectivity results will be compared with the optical polishing 316L sample results presented in this report.

Element	Percent by Weight (max. unless otherwise stated)
Nickel	65 (minimum)
Molybdenum	28.5 (nominal)
Chromium	1.5 (nominal)
Iron	1.5 (nominal)
Cobalt	3
Tungsten	3
Manganese	3
Aluminum	0.5
Titanium	0.2
Silicon	0.1
Carbon	0.01

Table 3. Chemical composition of Hastelloy B-3.[5]

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