Application Note

SEAGULL™ VARIABLE ANGLE REFLECTANCE ACCESSORY NO. 21130

An Evaluation of Different Finishing Techniques for the Fabrication of Mid-Infrared Liquid Cell Optical Mirrors From Hastelloy™ B-3 Alloy

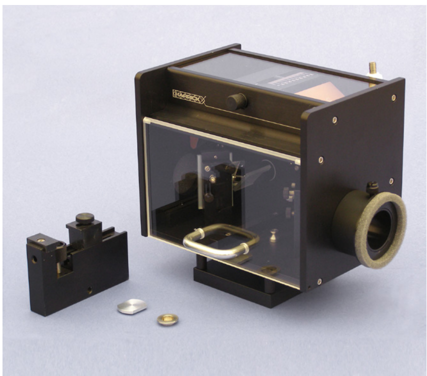

Figure 1. The Seagull™ Variable Angle Reflectance Accessory.

Element	Percent by Weight (maximum)
Nickel	10-14
Molybdenum	2-3
Chromium	16-18
Iron	~65.5
Cobalt	---
Tungsten	---
Manganese	2.00
Aluminum	---
Titanium	---
Silicon	0.75
Carbon	0.03
Phosphorus	0.045
Sulfur	0.030
Nitrogen	0.10

Table 1. Chemical compositions of Hastelloy™ B-3 and 316L stainless steel.

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POSTER PAPER NO. 570-7P; SESSION 570 – NEW PRODUCTS AT PITTCON 2013; PRESENTED AT THE 2013 PITTSBURGH CONFERENCE IN PHILADELPHIA, PENNSYLVANIA; MONDAY, MARCH 18, 2013

ABSTRACT

A previous study[1] evaluated different finishing techniques on 316L stainless steel for optimum performance in the mid-infrared. The purpose of that study was to investigate which method would be the best for producing mirrors intended for use in liquid cells.[2] The results indicated that conventional optical polishing was the best in terms of reflectance, flat response, and reproducibility. One drawback of 316L stainless steel, however, is that, with its relatively high iron content, diamond lathe turning, a typical machining procedure to produce mirrors for the mid-infrared, could not be used.[3] One corrosion resistant material which does not have this drawback has been chosen for further investigation in this study. Hastelloy™ B-3, produced by Haynes International, Inc., is a nickel alloy with a relatively low iron content. Rectangular test samples are made with the following finishes: none (raw material), bead blasting, carbide turning, diamond turning, and optical polishing. Three test samples for each finish are analyzed. The results are compared with those previously obtained for the optically polished 316L stainless steel. 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°, at 20° increments.

Finish	Avg. Refl (%R)	Samp/Samp St. Dev. (%R)	Wave-length St. Dev. (%R)	Angle St. Dev. (%R)	Slope (%R/cm^{-1} x 10^{-3})	Corr. Coeff.
Raw Material	54.9	18.4	10.1	13.3	-7.29	-0.957
Bead-Blast	43.4	1.7	10.3	13.2	-7.56	-0.936
Carbide Lathe-Turned	75.7	4.6	7.7	5.0	-5.89	-0.991
Diamond Lathe-Turned	67.7	6.2	8.6	4.9	-6.93	-0.997
Optical Polish	83.6	0.4	3.2	2.9	-2.30	-0.987
Optical Polish (316L stainless)	85.7	0.4	3.6	2.5	-2.80	-0.996

Table 2. Reflectivity properties of various finishes.

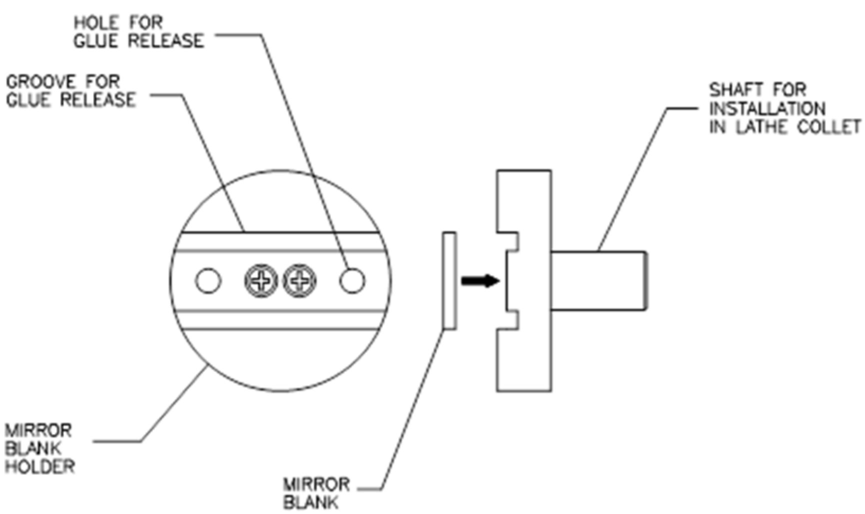

Figure 2. Mirror support assembly.

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 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

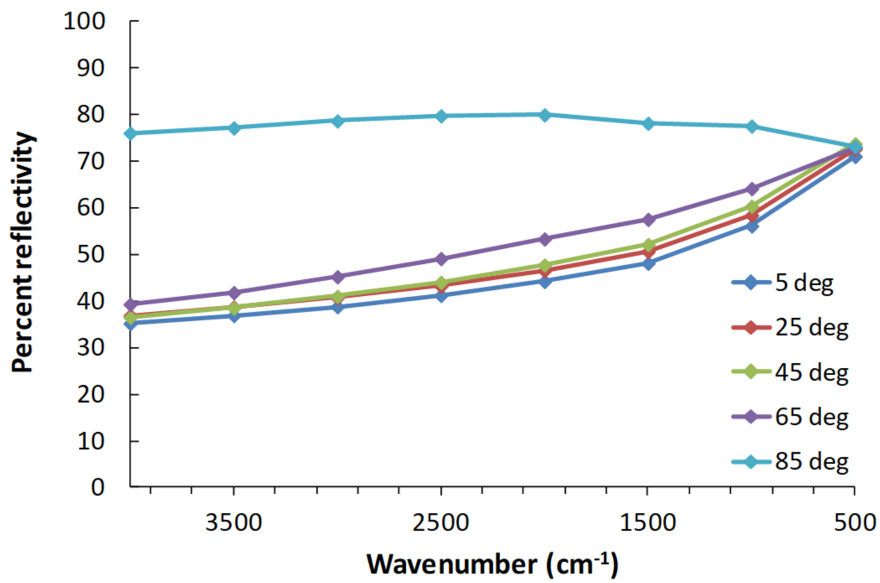

Figure 3. Raw material finish reflectivity at various angles.

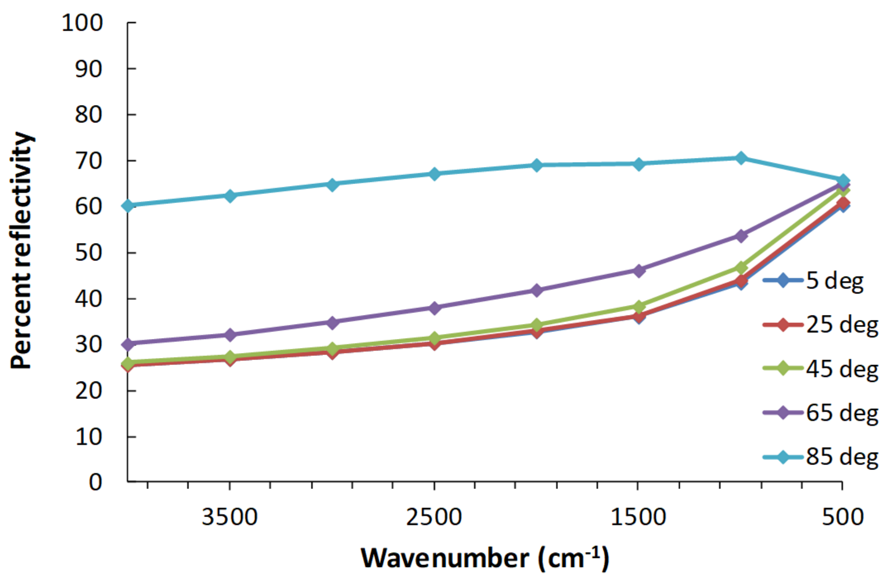

Figure 4. Bead blast finish reflectivity at various angles.

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 Hastelloy™ B-3 alloy (Haynes International, Inc.; Windsor, CT; www.haynesintl.com). This corrosion-resistant nickel alloy has a much lower iron content than 316L stainless steel, as seen in Table 1.

Hastelloy™ B-3 then has the potential to be finished using diamond tooling.[4] The raw material had a thickness of approximately 3.2 mm. Each piece was cut into a rectangle 50 mm wide by 25 mm long. Three test pieces for each finish type were prepared and one reading at each angle for each piece was made.

The five finish types were: none (raw material), bead blasting, tungsten carbide lathe turning,[7] diamond lathe turning, and optical polishing. Bead blasting was done in-house using a special cabinet (Model 3824; Cyclone Manufacturing; Dowagiac, MI; www.cycloneblasters.com) operated at 75 psi compressed air pressure. Glass beads from the same company of 60 to 100 grit (254 to 122 μm diameter) were used in the cabinet. Lathe turning was done in-house using specially designed holders (see Figure 2)

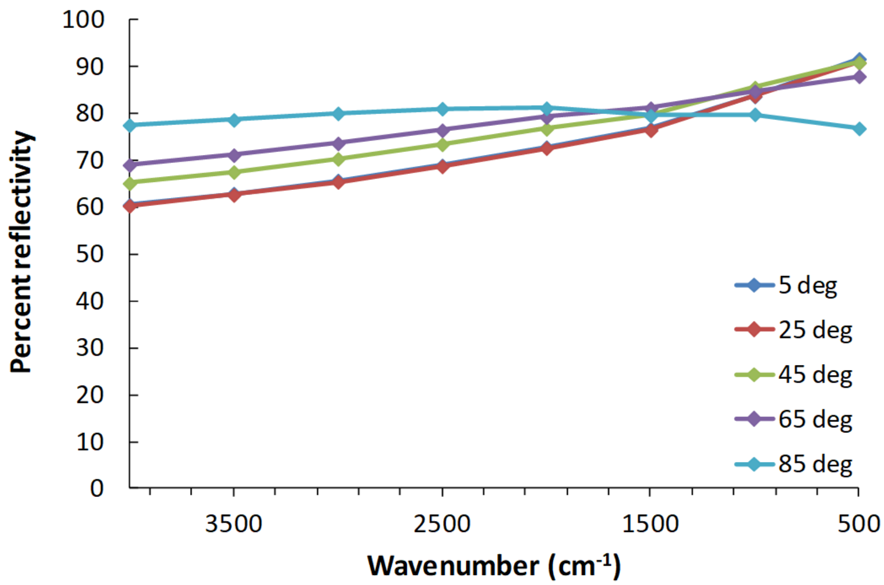

Figure 5. Tungsten carbide lathe turned finish reflectivity at various angles.

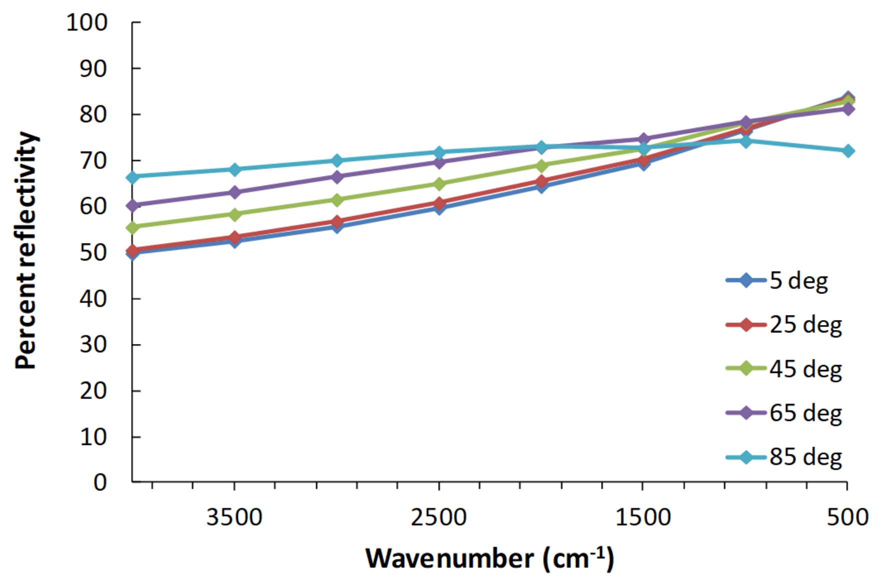

Figure 6. Diamond lathe turned finish reflectivity at various angles.

to which the sample rectangles were temporarily attached using Loctite 380 adhesive (Henkel Corporation; Rocky Hill, CT; www.henkel.com). Following the lathe operations, the samples were removed by soaking in acetone (Klean-Strip™ Product #GAC18; W. M. Barr & Co., Inc.; Memphis, TN; www.kleanstrip.com). Tungsten carbide and diamond tooling were obtained from McMaster-Carr (P/N 3367A345 for tungsten carbide and P/N's 3316A44 and 3316A34 for diamond). The visual finishes on the tungsten carbide turned lathe parts were estimated to be 16 µin. (0.40 µm) RMS surface roughness or better. The visual finishes on the diamond turned lathe parts degraded. For the first sample it was estimated to be 16 µin. (0.40 µm) RMS surface roughness. For the second it was estimated to be 32 µin. (0.80 µm) RMS surface roughness. And for the third it was estimated to be 63-125 µin. (1.6-3.2 µm) RMS surface roughness. Optical polishing to an 80/50 scratch/dig specification was done by Optics Masters (Poway, CA; www.opticsmasters.net). (In the previous study,[1] it was found that precision grinding prior to optical polishing was unnecessary, so this step was omitted in the current work.)

RESULTS AND DISCUSSION

Reflectivities for each of the five

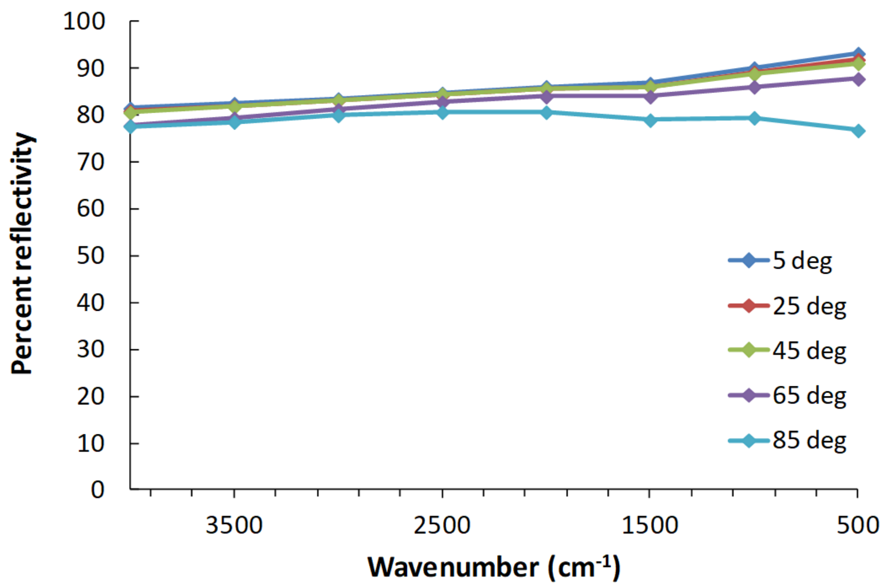

Figure 7. Optical polish finish reflectivity at various angles.

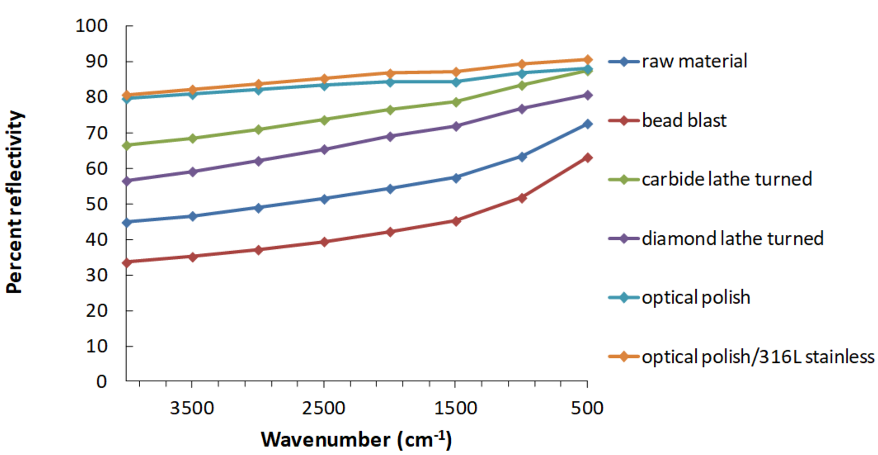

Figure 8. Reflectivity of various finishes (average from 5° to 85°)

finishes are shown in Figures 3 through 7, which plot the average reflectivities of three samples vs. wavelength for 5° to 85° at 20° increments. Figure 8 shows the curves for all finishes with the results averaged across all angles. Various overall reflectivity properties are summarized in Table 2. The previous results[1] for the optically polished 316L stainless steel are also shown in Figure 8 and Table 2.

In general, the characteristics that are sought for the Hastelloy™ 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 3 through 8 indicate that the optical polish is superior to all of the others in these categories. (The negative slopes indicated in Table 2 are due to the fact that, in general, the reflectivities increase with decreasing wavenumbers.) Hence, the most expensive process, optical polishing, is the only practical choice. The optically polished Hastelloy™ samples were nearly as good as the optically polished 316L stainless steel samples, as indicated in Figure 8 and Table 2. In general, the reflectivity increases with wavelength and angle for all finishes. The intended application[2] would require good reflectivity throughout the mid-infrared wavelength range and at the lower angles, say

5° to 10°, and the optical polish finish fulfills both of these requirements.

Due to the added expense of the Hastelloy™ alloy in both material and machining, the 316L material would be preferred under the majority of circumstances where the added inertness of Hastelloy™ was not required.

As indicated by the figures and Table 2, the diamond turned samples were inferior to those produced by tungsten carbide turning. In fact, reflectivities declined in going from the first to the third sample produced. Two diamond tools were broken in the process of producing the three samples and it is believed that this was due in large part to an incompatibility of the two materials. Evidently there is a sufficient amount of iron in Hastelloy™ B-3 to cause problems with diamond turning.

The figures and Table 2 do show some additional interesting characteristics. High reflectivity jumps at high angles are shown for the raw material and bead-blast finishes. The bead-blast finish shows very good sample-to-sample reproducibility but very poor reflectivity. (A similar result was obtained for 316L stainless steel in the previous study.[1])

Future work will focus on the practical aspects of liquid cell construction using the optical polishing technique.

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