Surface-texture evolution of different chemical-vapor-deposited zinc sulfide flats polished with various magnetorheological fluids

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ABSTRACT

The macro-structure of chemical-vapor-deposited (CVD) zinc sulfide (ZnS) substrates is characterized by cone-like structures that start growing at the early stages of deposition. As deposition progresses, these cones grow larger and reach centimeter size in height and millimeter size in width. It is challenging to polish out these features from the top layer, particularly for the magnetorheological finishing (MRF) process. A conventional MR fluid tends to leave submillimeter surface artifacts on the finished surface, which is a direct result of the cone-like structure.

Here we describe the MRF process of polishing four CVD ZnS substrates, manufactured by four different vendors, with conventional MR fluid at pH 10 and zirconia-coated–CI (carbonyl iron) MR fluids at pH 4, 5, and 6. We report on the surface-texture evolution of the substrates as they were MRF polished with the different fluids. We show that performances of the zirconia-coated–CI MR fluid at pH 4 are significantly higher than that of the same fluid at

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pH levels of 5 and 6 and moderately higher than that of a conventional MR fluid at pH 10. An improvement in surface-texture variability from part to part was also observed with the pH 4 MR fluid.

Keywords: MRF, CVD ZnS, polishing, pebbles, surface artifacts

1. Introduction

Zinc sulfide (ZnS) is an IR optical material widely used for applications such as IR windows, domes and optical lenses [1]. It is industrially produced by the chemical-vapor-deposition (CVD) technique to reach a dense, milky yellow–orange color, with ~70% transmission in the mid-long-wave IR region [1]. Its inner structure consists of cone-like structures that grow larger as deposition takes place, up to a thickness of a few centimeters in size [2,3]. These cone-like structures manifest on the top of the deposited surface as “pebbles” [2] or “hillocks” [4] and are often called “alligator skin” [5].

The importance of good surface finishing of an optical component is well understood. Lack of good finishing results in the scattering and absorption of light, leading to low optical performance [1,6]. Polishing out the pebble structure from a CVD ZnS substrate to a relatively smooth [<3 -nm root mean square (rms)] surface is quite challenging, especially for an undestructive polishing process [7] (that leaves no plastic deformation and does not destroy the crystallographic array of the top finished layer), such as the magnetorheological finishing (MRF) technique.

MRF is a deterministic, sub-aperture polishing process that is capable of polishing flats, spheres, and aspheric shapes [8]. It uses an MR fluid composed of micron-size carbonyl iron (CI)

powder, water, stabilizing additives, and abrasives (usually ceria or nanodiamonds). When exposed to a magnetic field, the fluid stiffens (as the CI particles align with the magnetic field) and functions as a polishing pad with a layer of nonmagnetic abrasive particles on the top layer. This fluid is kept at relatively high pH (10 or higher) to suppress corrosion of the iron particles. By suppressing corrosion, a conventional MR fluid can last more than three weeks.

Kozhinova *et al.* [2] and Hallock *et al.* [9] demonstrated an improvement in surface artifacts on CVD ZnS and ZnSe, respectively, during MRF by using acidic (pH ~4.5) MR fluids and soft CI particles; however, the MR fluids used in their work did not provide consistent and repeatable results. The two main challenges they faced were a (a) material-removal-rate performance that varied among CVD ZnS substrates manufactured by different vendors (0.5 to 1.5 $\mu\text{m}/\text{min}$) and (b) rapid corrosion of the CI particles in the MR fluid.

There is no consensus of deposition parameters for CVD ZnS [4,10]. Different manufacturers select different deposition conditions (such as deposition temperature, pressure, and vapor velocity). Therefore it is reasonable to assume that physical properties of the final product (such as color, average grain size, and crystallographic orientation volume fraction) are different among vendors, and might lead to variations in polishing results among different parts.

In 2008 a novel zirconia sol-gel coating process of CI particles to protect them from corrosion was invented at the University of Rochester [11]. These coated particles were successfully mixed to a MR fluid at pH 8 to perform a MRF experiment on several optical glasses and ceramics [12]. In 2013 [13] we reported on a MRF experiment using MR fluids based on the same zirconia-coated-CI particles at pH levels of 4, 5, and 6. In that experiment we used single-crystal ZnS planes to investigate the role of material-removal-rate anisotropy of polycrystalline (pc) CVD ZnS during MRF. The results showed a relatively uniform removal rate

(0.06 $\mu\text{m}/\text{min}$) for single-crystal planes when using MR fluid at pH 4. Our conclusions predicted an improvement in surface artifacts (the emergence of pebbles on the surface) when using this type of fluid for MRF polishing of a CVD ZnS substrate.

Here we present the surface-texture evolution of several differently grown CVD ZnS substrates that were MRF polished with four MR fluids at pH levels of 10, 6, 5, and 4. Our goal was twofold: to check (1) if a decrease in MR fluid pH improves the surface artifacts of a CVD ZnS surface during MRF polishing; and (2) if MR fluid pH is capable of dealing with part-to-part variations in the surface texture among CVD ZnS materials deposited by different vendors.

We must emphasize that our intent is not to identify which vendor's ZnS material may be preferable for a MRF application, but rather to establish how chemomechanical parameters of the MR fluid affect CVD grown ZnS.

2. Materials and Methods

2.1. Polycrystalline CVD ZnS Substrates

Four CVD ZnS substrates were purchased from four different vendors. One is an elemental CVD ZnS substrate purchased from China; the other three are forward-looking–infrared (FLIR) CVD ZnS substrates purchased from China and the U.S. The difference between the two types lays in the chemical reaction of the precursor gases (for more information, refer to Refs. 1 and 6). We will refer to them as substrates A (FLIR, U.S.), B (FLIR, U.S.), C (FLIR, China), and D (elemental, China). Each sample measured 40 mm in diameter and 5 mm in thickness. The samples were pre-polished in-house on pitch with diamond abrasives (as described in Ref. 2) to a peak-to-valley (p–v) flatness of 1λ to 2λ and an areal roughness of less than 27-nm p–v and 2-nm rms.

2.2. X-Ray Diffraction

To determine the relative portion of crystallite orientations within the samples, we performed an x-ray diffraction (XRD) analysis using a general-purpose x-ray diffractometer (Philips X'Pert, MPD system). A Cu radiation ($\lambda_{\text{Cu}} = 1.5418 \text{ \AA}$) was used to produce the x-ray diffraction pattern in a 2θ angle range of 10° to 70° with step intervals of $2\theta = 0.03^\circ$. The diffraction data were analyzed using X'Pert High Score software. The reference database for cubic ZnS was taken from the Joint Committee for Powder Diffraction Standards (JCPDS) file by the International Centre for Diffraction Data, Newtown Square, PA. We used JCPDS files 65-0309, 65-5476, and 65-1691.

2.3. MRF Spotting Experiment

MRF spotting experiments were performed on a research platform referred to as the “spot-taking machine” (STM). It has features similar to a conventional MRF machine; however, it is free to move only in the vertical z direction. Consequently, it is capable of performing only single spots on the surface. The MR fluids used here were conventional diamond fluid (D11) at pH 10, purchased from QED Technologies [14] and zirconia-coated–CI-based [11] MR fluids at pH levels of ~ 4 , ~ 5 , and 6, developed in our laboratory. The initial fluid composition of the zirconia-coated–CI fluid, before the addition of any acid, is given in Table 1.

For the experiment with the conventional fluid, each CVD ZnS substrate was spotted once for 60 s. For the experiment with the zirconia-coated–CI fluids, each substrate was spotted once at pH 6.00 ± 0.0 , once at pH 5.12 ± 0.0 , and once at pH 4.22 ± 0.1 . Each spot lasted 10 min as a result of the lower removal rate of 0.06 to $0.16 \text{ }\mu\text{m/min}$. The zirconia-coated–CI MR fluid was

first loaded on the STM at pH 6.00 ± 0.0 . After spotting the substrates, the fluid pH was lowered to 5.12 using ~4 ml of 8 M nitric acid for another round of spotting, which was followed by additional reduction in pH to 4.22 using ~5 ml of 8 M nitric acid. Because of issues with pH stability during the spotting experiment at pH 4.22, an additional 8 M of nitric acid was continuously added to maintain the fluid pH level at ~4.20. Further reduction of the fluid's pH was impossible since corrosion signs of the fluid were observed (such as rapid increase of pH and rust-like odor, most likely caused by failure of the zirconia protective layer).

Because of water evaporation from the MR fluids during the experiment, any addition of acid had a negligible effect on the CI particles' concentration in it. The fluid moisture percentage when the experiment was over was 20.42%, less than 1 wt% difference from what it was at the beginning. We will refer to the MR fluids at pH 4.22 as pH 4, at 5.12 as pH 5, and at 6.00 as pH 6.

The machine settings were a ribbon height of 1.4 to 1.6 mm; a penetration depth of 0.2 mm; a wheel speed of 200 rpm; a pump speed of 110 rpm; and an electric current of 15 A. When the experiment was finished, each substrate had four spots on its surface, one at each pH.

During the experiment, we paid special attention to removing at least $0.5 \mu\text{m}$ at the deepest point of removal. This was based on Kozhinova *et al.* [2] observations, which stated that pebbles start to appear on pre-polished surfaces of CVD ZnS once a minimum of $0.5 \mu\text{m}$ of material has been removed with MRF.

2.4. MR Fluid Rheology

MR fluid off-line viscosity was measured using a Brookfield cone/plate rheometer [15]. At each pH, ~0.5 ml of fluid was extracted directly from the mixing vessel of the STM and

placed on the viscometer plate. The fluid went through a time test in which the shear rate went from 0 1/s to 200 1/s for 30 s and then went back down to 0 1/s for another 30 s. This helped to minimize the transient behavior of the fluid. Following the time test, we measured the viscosity of the fluid as function of shear rate from 40 to 1000 1/s. All measurements were repeated three times.

2.5. Metrology

To evaluate the emergence of pebbles on the surface inside the spots, we used a Zygo NewView5000 white-light interferometer with a $1\times$ Mirau objective and a $0.8\times$ zoom [16]. The low magnification provides a large field of view that is more suitable for observing submillimeter features, such as pebbles. The spots were masked along the inner edge, and a cylinder shape was removed from the remaining masked surface. The areal rms roughness of the masked area was recorded and plotted along with ten manually drawn lineout profiles in the direction of the MRF ribbon. We also analyzed a power spectral density (PSD) in the x direction using the “average X PSD” function in the accompanying software MetroPro. To do that, we masked a rectangular area ($2\text{ mm} \times 1\text{ mm}$) around the depth of deepest penetration (ddp) of each spot. This analysis also provided us with information about the degree of waviness of the spotted surfaces.

3. Results

3.1. X-Ray Diffraction of CVD ZnS Substrates

Table 2 shows the XRD results of relative intensity of crystallite orientation within the samples. For all samples, the peaks were normalized to the highest peak; i.e., for samples A, C,

and D, the intensity peaks were normalized to the (111) peak, whereas for sample B, the peaks were normalized to the (311) peak. From Table 2 we see that the relative intensity rating is different among the four samples. For example, sample A's XRD results show that the (111) plane has the highest relative intensity score followed by plane (200) and then plane (311). For sample B, the ranking from high to low for the first three peaks is (311) followed by (111) and (200). This inconsistency in the order of diffracting planes is also true for samples C and D.

3.2. MR Fluids' Off-Line Viscosity and Material-Removal Rate

Figure 1 shows the off-line viscosity of all fluids as a function of shear rate. The viscosity of the zirconia-coated-CI fluid is pH dependent. It decreases as pH decreases, although the CI particle concentration has not changed (80 to 81 vol %). The fluid's off-line viscosities at ~800 (1/s)—a shear rate corresponding to that experienced by an MR fluid when ejected from the STM nozzle [12]—are ~60 cP for the conventional MR fluid and ~194, ~109, and ~47 cP for pH 6, 5, and 4, respectively, for the zirconia-coated-CI MR fluids. The MR fluid at pH 4 has the lowest viscosity of all fluids.

Table 3 presents the material peak-removal rates (pr) of the different fluids. The material removal rate of the zirconia-coated-CI MR fluid is significantly lower than that of the conventional MR fluid. This is expected because our MR fluids had no added abrasives, in contrast to the conventional MR fluid. However, pr results for CVD ZnS at pH 6 are similar to what were previously published by Shafrir *et al.* [12] when working with this type of zirconia-coated-CI particles in their MR fluid at pH 8. The zirconia-coated-CI fluid at pH 4 has the lowest pr of 0.06 $\mu\text{m}/\text{min}$. Note that the zirconia-coated-CI MR fluid has no additive

abrasives in it besides possible free nanozirconia particles that are co-generated during the coating process [11,17], while the conventional MR fluid comes with nanodiamond abrasives in it.

3.3. Surface Texture and Artifacts After MRF Polishing

The surface texture inside the MRF spots is mostly composed of submillimeter features. Figure 2 shows the PSD for CVD ZnS surfaces spotted with a conventional MR fluid at pH 10 and zirconia-coated–CI MR fluids at pH 6, 5, and 4. For all (four) vendors, results show that surface texture and waviness are higher at pH 6, somewhat lower at pH 5, but significantly lower at pH 4. When compared with the conventional MR fluid at pH 10, the use of the zirconia-coated–CI MR fluid at pH 4 is comparable. For two of the ZnS materials [Figure 2(b) and 2(c)], the zirconia-coated–CI MR fluid leads to lower PSD than the conventional MR fluid.

The areal rms roughness and the average lineout profiles taken within the spots are presented in Figure 3(a) and 3(b), respectively. For all vendors, when polishing with the zirconia-coated–CI MR fluids, both areal (a 2-mm $\times$ 4-mm “D”-shaped) rms roughness and average lineout profiles (3 to 4 mm long) decrease with a reduction in MR fluid pH and viscosity. Moreover, the zirconia-coated–CI MR fluid at pH 4 shows lower rms roughness than the conventional MR fluid. Interestingly, variation in roughness from sample to sample is minimal when polishing with this type of fluid.

It is important to mention that roughness measurements were taken at a low magnification, i.e., at low resolution; therefore, it is suitable to relate to the data as it represents surface texture (at length scales 0.05 to 1 mm) rather than surface microroughness, which is not given here.

4. Discussion

X-ray diffraction results for the tested substrates show that the portions of the common crystallite orientations within each sample vary from sample to sample. McCloy *et al.* [18] showed that the crystallographic structure and the crystallite orientations of CVD ZnS material vary along the growth direction during deposition. From their powder XRD tests, the portion of the 200 and 400 orientations in the mandrel area is higher than that in the free surface. On the contrary, the portion of the 220 orientation increases as deposition takes place. Therefore we can conclude that the sample crystallographic properties depend on the physical location of the cut (top, middle, or mandrel side) and the desired dimensions of the cut (thin or thick). Unless one can control the cut position when purchasing a CVD ZnS substrate, variations in the crystallographic properties are a given fact. In addition, as mentioned in the Sec. 1, there is no consensus on deposition settings for CVD ZnS. This likely affects the relative portions of the crystallite orientations within the deposited material and adds to the variations in properties among samples purchased from different manufacturers. We believe that this nonuniformity in crystallographic properties within the material and among materials manufactured by numerous vendors is the source for anisotropy in the material removal rate during MRF polishing, which leads to surface artifacts of CVD-grown materials, such as ZnS.

In the MRF spotting experiments, we observed that the zirconia-coated–CI MR fluids' viscosity is pH dependent. Viscosity significantly decreased with a decrease in pH because the acid increased the zeta potential of the zirconia-coated–CI particles and helped disperse the fluid as pH decreased [13]. The drop in viscosity also influenced the prr which decreased consequently. The material prr of the conventional MR fluid is significantly higher than that of

the zirconia-coated–CI MR fluids, even though it has relatively low viscosity. Since all fluids have similar CI particles concentration, this clearly was a result of the nano-diamond abrasives within the conventional MR fluid.

From the PSD results we see that MRF with zirconia-coated–CI MR fluid at pH 4 achieved a remarkable improvement in surface texture (lower spatial frequency range) and surface microroughness (higher spatial frequency range) than the fluids at pH 5 and pH 6. This is true for all four CVD ZnS substrates. The performance of this fluid at pH 4 is slightly better than the conventional MR fluid for substrates manufactured by vendors B and C but comparable for substrates manufactured by vendors A and D. Clearly, a lower value of PSD indicates a surface with lower texture. Therefore, we can say that pebbles' emergence on the surface of CVD ZnS substrates can be reduced when using zirconia-coated–CI MR fluid at pH 4.

Root-mean-square (rms) data collected from a masked area inside the spots and as multiple lineouts (given in Figure 3) support the results obtained from PSD analysis. Reduction in surface texture is observed as the zirconia-coated–CI fluid pH and viscosity drop down. Moreover, from both areal [Figure 3(a)] and lineout [Figure 3(b)] results, we clearly see the ability of this fluid at pH 4 to maintain similar performances among all four tested substrates. We observe therefore that among CVD ZnS substrates deposited by different manufacturers, variations in areal and lineout rms roughness from part to part are minimal when using zirconia-coated–CI MR fluid at pH 4.

5. Conclusions

We have demonstrated that CVD ZnS substrates manufactured by different vendors show variations in the portions of crystallite orientations among each other. This raises challenges

when one wishes to get consistent performance during MRF polishing of such differently grown material. Our results show that when several CVD ZnS substrates are polished by MRF with a zirconia-coated–CI MR fluid at pH 4, both pebble emergence and part-to-part variations in surface texture are minimized. The performance of the zirconia-coated–CI MR fluid at pH 4 was better than those of MR fluids with pH levels of 5 and 6. On the other hand, for some ZnS materials, the zirconia-coated–CI MR fluid at pH 4 produced diminished features (at a scale length of 0.05 to 1 mm) as compared to the conventional MR fluid. Of course, the zirconia-coated–CI MR fluid at low pH has a particularly low material prr, especially at pH 4, which is expected because of the absence of any abrasives. We believe that adding some type of abrasive, such as ceria or nanodiamonds, will likely boost the overall prr of the zirconia-coated–CI MR fluid and improve its efficiency.

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FIGURE CAPTIONS

Fig. 1. Viscosity versus shear-rate plot for conventional MR fluid at pH 10 (squares) and zirconia-coated–CI MR fluids at pH 4 (circles), pH 5 (triangles), and 6 pH (diamonds).

Fig. 2. Average PSD along the x direction for conventional MR fluid (solid line), zirconia-coated–CI MR fluid at pH 6 (dashed line), pH 5 (dashed–dotted line), and pH 4 (dotted line) for Samples A–D, respectively.

Fig. 3. Macroroughness (rms) versus fluid pH for conventional MR fluid at pH 10 and zirconia-coated–CI MR fluid at pH 6, pH 5, and pH 4. (a) Areal rms roughness of a 2-mm × 4-mm “D”-shaped area; (b) average rms roughness taken from ten lineout profiles (3- to 4 mm-long lines). Horizontal lines are used to guide the eye, identifying the maximum and minimum values.

Table 1

Initial zirconia-coated–CI MR fluid composition before adjusting pH with 8-M nitric acid.

The fluid pH is ~6.0.

Material	ρ (g/ml ₃)	Volume (ml)	M (g)	Volume (%)	Wt (%)
Zirconia-coated–CI powder	6.72	384.80	2583.93	38.60	80.67
Polyethylenimine (PEI) solution	1.10	69.68	76.65	6.99	2.39
H ₂ O	1.00	542.36	542.36	54.41	16.93
Total	—	996.84	3202.94	100.00	99.99

Table 2

Relative intensity of CVD ZnS substrates from four vendors with respect to diffraction angle (2θ) and crystallographic plane. The shaded cells represent the highest peak that was used to normalize the rest of the data.

Approximate diffracting angle $2\theta(^{\circ})$	Diffracting planes at 2θ	Relative intensity within sample (%)			
		FLIR		Elemental	
		Sample A JCPDS 65-0309	Sample B JCPDS 65-5476	Sample C JCPDS 65-1691	Sample D JCPDS 65-5476
28.5	111	100	70.2	100	100
33.1	200	84.3	43.8	48.0	2.14
47.5	220	25.3	15.5	23.9	22.2
56.3	311	69.3	100	59.8	13.4
59.1	222	2.1	0.9	1.6	1.7
69.5	400	48.4	28.5	33.1	1.2

Table 3

Peak removal rate in $\mu\text{m}/\text{min}$ of the different MR fluids and their pH level.

MR fluid type	MR fluid pH	Peak removal rate ($\mu\text{m}/\text{min}$)
Conventional fluid (with diamond abrasives)	10	3.50
	6	0.16
Zirconia-coated CI (without abrasives)	5	0.10
	4	0.06

Figure 1-Revised

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Figure 2-Revised

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Figure 3-Revised