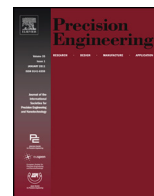




Contents lists available at [ScienceDirect](http://www.sciencedirect.com)

Precision Engineering

journal homepage: www.elsevier.com/locate/precision



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

Sivan Salzman^{a,b,*}, Henry J. Romanofsky^a, Stephen D. Jacobs^{a,b,c,d},
John C. Lambropoulos^{a,b,e}

^a Laboratory for Laser Energetics, University of Rochester, 250 East River Road, Rochester, NY 14623, United States

^b Materials Science Program, University of Rochester, Rochester, NY 14623, United States

^c The Institute of Optics, University of Rochester, Rochester, NY 14623, United States

^d Department of Chemical Engineering, University of Rochester, Rochester, NY 14623, United States

^e Department of Mechanical Engineering, University of Rochester, Rochester, NY 14623, United States

ARTICLE INFO

Article history:

Received 1 June 2015

Received in revised form 15 July 2015

Accepted 28 July 2015

Available online xxx

Keywords:

MRF

CVD ZnS

Polishing

Pebbles

Surface artifacts

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-Cl (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-Cl MR fluid at pH 4 are significantly higher than that of the same fluid at 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.

© 2015 Elsevier Inc. All rights reserved.

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,

* Corresponding author at: University of Rochester, Laboratory for Laser Energetics, 250 East River Road, Rochester, NY 14623, United States. Tel.: +1 5852733729.
E-mail address: ssal@le.rochester.edu (S. Salzman).

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

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. [1,6]). 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 $\mu\text{m}/\text{min}$. 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.

Download English Version:

<https://daneshyari.com/en/article/7180716>

Download Persian Version:

<https://daneshyari.com/article/7180716>

[Daneshyari.com](https://daneshyari.com)