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# Mid-infrared volume diffraction gratings in IG2 chalcogenide glass: fabrication, characterization and theoretical verification

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# ABSTRACT

Ultrafast laser inscription (ULI) has previously been employed to fabricate volume diffraction gratings in chalcogenide glasses, which operate in transmission mode in the mid-infrared spectral region. Prior gratings were manufactured for applications in astrophotonics, at wavelengths around 2.5  $\mu$ m. Rugged volume gratings also have potential use in remote atmospheric sensing and molecular spectroscopy; for these applications, longer wavelength operation is required to coincide with atmospheric transparency windows (3–5  $\mu$ m) and intense ro-vibrational molecular absorption bands. We report on ULI gratings inscribed in IG2 chalcogenide glass, enabling access to the full 3–5  $\mu$ m window. High-resolution broadband spectral characterization of fabricated gratings was performed using a Fourier transform spectrometer. The zeroth order transmission was characterized to derive the diffraction efficiency into higher orders, up to the fourth orders in the case of gratings optimized for first order diffraction at 3  $\mu$ m. The outcomes imply that ULI in IG2 is well suited for the fabrication of volume gratings in the mid infrared, providing the impact of the ULI fabrication parameters on the grating properties are well understood. To develop this understanding, grating modeling was conducted. Parameters studied include grating thickness, refractive index modification, and aspect ratio of the modulation achieved by ULI. Knowledge of the contribution and sensitivity of these parameters was used to inform the design of a 4.3  $\mu$ m grating expected to achieve > 95% first order efficiency. We will also present the characterization of these latest mid-infrared diffraction gratings in IG2.

Keywords: Grating, chalcogenide, mid-infrared, volume diffraction grating, ultrafast laser inscription, transmission grating.

# 1. INTRODUCTION

Diffraction gratings are a key component of broadband spectroscopic optical systems, allowing differentiation of closely-spaced spectral features, and coverage over a broad spectral range. Typically, ruled reflection gratings are used in these applications, as they are spectrally broadband and allow coverage from visible through to near- and mid-infrared spectral regions.

Volume transmission gratings are formed by introducing a periodic refractive index modification within the volume of a bulk material. Volume transmission gratings have some advantages over traditional ruled reflection gratings. One key feature is the polarization dependence, which affects the grating response to a far lesser extent in the transmission grating, as the diffraction in both linear polarization states 'sees' approximately the same refractive index modulation profile. A second advantage is the ability to 'unfold' the instrument beam path, enabling more compact and complex optical systems to be realized. Lastly, volume gratings intrinsically offer a high level of robustness owing to a grating structure embedded into the bulk of an optical material.

Typical volume transmission gratings have been demonstrated in the visible and near-infrared spectral regions to date, generally for astronomical spectroscopy applications.<sup>1</sup> These gratings are typically volume holographic gratings, and are created by changing the local refractive index of a photosensitive dichromated gelatin layer by illumination with an

Optical Components and Materials XV, edited by Shibin Jiang, Michel J. F. Digonnet, Proc. of SPIE Vol. 10528, 105280R © 2018 SPIE · CCC code: 0277-786X/18/\$18 · doi: 10.1117/12.2288357 interference pattern.<sup>2</sup> The transmission characteristics of this material dictate it cannot be used for wavelengths greater than approx. 2.5  $\mu$ m. Gratings suitable for peak diffraction efficiency at similar wavelengths include those fabricated in gallium lanthanum sulphide (GLS) chalcogenide glass; these were previously demonstrated to have diffraction efficiencies close to 90% when an anti-reflection coating was applied.<sup>3</sup> GLS has transmission across 2–8  $\mu$ m,<sup>4</sup> and so could potentially be used to produce gratings with first order diffraction across this entire spectral region.

The purpose of this work was to identify a material that could be used to produce gratings suitable for atmospheric molecular spectroscopy, in the atmospheric windows at 3–5 and 8–12  $\mu$ m. Given the success of grating inscription using GLS, an alternative commercial chalcogenide glass was investigated<sup>5</sup> that has transmission across the full range of both atmospheric windows. The material chosen was IG2, which is fabricated by Vitron, and exhibits approx. 70% transmission across 1.5–12  $\mu$ m.<sup>6</sup>

This paper describes work undertaken to demonstrate volume transmission gratings with peak first order diffraction in the 3–5  $\mu$ m window. Fabrication and characterization of a grating with peak diffraction wavelength at 3  $\mu$ m is described. Detailed theoretical modelling predicts the refractive index modification and periodic form of the measured grating structure. Using the obtained parameters, a grating with peak diffraction efficiency at 4.3  $\mu$ m, specifically for measurement of CO<sub>2</sub> absorption at this wavelength, was predicted and fabricated. Modeled expectations and measured transmission spectra are presented. This is the first demonstration of a mid-infrared volume transmission grating with peak diffraction efficiency at this wavelength, and which covers the entire 3–5  $\mu$ m window with measured diffraction efficiency greater than 27% across the full band.

# 2. FABRICATION OF DIFFRACTION GRATING

Ultrafast laser inscription of volume diffraction gratings, including specific laser inscription parameters, has been described in detail elsewhere.<sup>5</sup> The diffraction gratings were fabricated in 25 mm diameter, 1 mm thick IG2 discs. The gratings were 6 mm square, central to the disc face. The grating structure was inscribed within the material, starting approx. 100 µm from the front surface. The grating extended within the material to a specific optical thickness, which was physically formed of a number of sequentially-inscribed grating layers positioned above one another. The number of layers was determined prior to fabrication by experimental observation of the dependence of peak diffraction wavelength on the number of inscribed layers for a number of near-infrared gratings, described elsewhere. This grating thickness determines the peak diffraction wavelength, corroborated by the model described later, and values are given later for the individual gratings. Compared to grating fabrication in GLS,<sup>5</sup> the pulse energy required for grating fabrication in IG2 was lower; the pulse energy used here was 13 nJ. A picture of a fabricated grating is shown in Figure 1(a).

#### 3. MEASUREMENT OF GRATING RESPONSE

The gratings were measured using a Bruker Vertex 80v Fourier Transform Spectrometer (FTS), in transmission mode. Measuring the zeroth order transmission of the grating indicates the diffracted power into the first, second and higher diffraction orders. A rotation stage was installed in the FTS sample compartment to allow the grating to be rotated within the characterization beam, and the zeroth order transmission for various angles of incidence (AOI) was measured. Prior to measurement, the Allan variance of the instrument was obtained using the grating measurement settings, to ensure sufficient averaging to reduce instrument noise effects. All spectra were collected with the instrument under vacuum to reduce the effect of water absorption lines around 3 µm on the collected spectra.

The zeroth order transmission spectra for various AOI are shown in Figure 1(b), truncated to show only the first order diffraction features. The spline (dashed) is for illustrative purposes only, and indicates the superblaze of the first order diffraction, which peaks at 2.8  $\mu$ m. The peak diffraction wavelength is at 3  $\mu$ m, and first order diffraction occurs across the entire 3–5  $\mu$ m spectral window. The 0° measurement (black line) is shown to indicate the transmission of bulk IG2.



Figure 1. (a) Photograph of a fabricated IG2 grating structure, obtained under near-infrared illumination. (b) Zeroth order transmission spectra of IG2 transmission grating designed for peak diffraction wavelength at 3 µm. Plots are truncated to show only the first diffraction order dips.

Likewise, the spectra of the second and third diffraction orders were also collected, as part of the same measurement step; these truncated spectra are shown in the modelling section below.

## 4. MODELLING OF GRATING STRUCTURE

Modelling of the grating structure considered three key parameters of the inscribed grating lines. The first, as indicated above, was the thickness of the overall grating structure within the IG2 material. The second was the refractive index modification ( $\Delta$ n) induced on laser inscription. The final parameter is the 'aspect ratio' (AR): the fraction of one grating period that is modified. The figure below shows the physical interpretation of these parameters.



Figure 2. Definition of model parameters. Green dashed cross-section in upper image has the refractive index profile shown in green in the lower part of the figure.

The parameters were observed to have the following effect on the modeled spectral response:

- Increasing the thickness of the grating increased the wavelength of peak first order diffraction, and also reduced the full width at half maximum (FWHM) of the spectral response at each AOI.
- Increasing the refractive index modification shifts the peak first order diffraction towards longer wavelengths.
- Modification of the AR changed the spectral shape of the observed diffraction. The superblaze (dashed line, Figure 1), narrows around the peak diffraction wavelength as the AR is increased, for AR ≤ 0.5. At the same time, the relative peak intensities of the higher order diffraction peaks varied, as a result of the shifting superblaze curve.

Optimization of these parameters was by least squares difference evaluation of the collected data compared with the model output. In addition, care was taken to maintain the peak first order diffraction dip at  $2.8 \pm 0.1 \mu m$ .

Figure 3 shows the output produced by optimizing the model parameters as described above. The model parameters used were:

• Thickness = 128 µm. This is well known from the fabrication stage, so was kept constant.



Figure 3. Measured (solid) and modeled (dashed) data for first (a), second (b) and third (c) order diffraction of a grating designed for peak first order diffraction at 3 µm.

One final model step was required to produce these spectra. Looking at the higher diffraction orders, it is observed that the diffraction dip is broadened to a doublet (e.g.,  $35^{\circ}$  green spectrum in Figure 3(b),  $55^{\circ}$  blue spectrum in Figure 3(c)). This broadening is due to the slightly convergent beam used for characterization in the FTS. By measuring the angle required to produce two peaks with the spacing of this doublet, the convergence of the characterization beam can be estimated to be AOI  $\pm 1.35^{\circ}$  in air. To produce the spectrum for each AOI, five discrete modeled spectra were obtained, for AOI  $- 1.35^{\circ}$ , AOI  $- 0.67^{\circ}$ , AOI, AOI  $+ 0.67^{\circ}$ , AOI  $+ 1.35^{\circ}$ . The average of these five spectra was taken, and the resulting spectra normalized to the 0° spectrum shown in Figure 1(b) above, for direct comparison between the measured and modeled data.

The data above shows good agreement between measured and modeled spectra across all diffraction orders. The measured peak first order diffraction is implied by the drop in zeroth order transmission at this wavelength, assuming no other diffraction orders propagate at this wavelength and angle. For this 3  $\mu$ m grating, the zeroth order transmission drops from the normalized background level of 70% to 26%, implying first order diffraction efficiency of 44%.

There is a near-constant offset in normalized transmission between modeled and measured spectra, which implies an overestimate of the grating diffraction efficiency across all orders. This offset is 11% across the first order diffraction spectra, and reduces to 8% for second order, and 3% for third order. The physical interpretation of this error is complex, due to the wide range of contributing factors. These include, but are not limited to: errors in the representation of the modeled FTS beam via five-spectra average; lack of knowledge of the uniformity of the convergent beam, including the assumption that the angle of convergence is not wavelength-dependent; assumption that the refractive index

modification observed by the characterization beam is the same at all wavelengths; and potential mismatch in alignment of the grating layers caused by the nominal positioning accuracy of the air-bearing stage system, which is  $0.2 \mu m$ . It is likely that all factors contribute to some extent to the discrepancy between modeled and measured data, and further fabrication and characterization system characterization is required to reduce these errors.

The ideal diffraction efficiency of the grating is masked to some extent by the errors introduced by the characterization method. To predict the ideal performance of the grating, a single AOI spectrum was modeled, to simulate a perfectly collimated input characterization beam, and is shown in Figure 4. This modeled spectrum indicates that the peak first order diffraction efficiency is close to 70%, and shows the expected sinc-squared spectral profile expected for a top-hat refractive index modulation (this is smoothed in Figure 3(a), above). If an anti-reflection coating were applied to such a grating structure, the peak diffraction efficiency is anticipated to be > 95%.



Figure 4. Modeled spectra for a single angle of incidence (collimated beam), showing close to 70% first order diffraction efficiency at the peak diffraction wavelength.

#### 5. 4.3 MICRON GRATING

The ultimate aim of this work was to create a grating suitable for spectroscopy of atmospheric CO<sub>2</sub> at 4.3  $\mu$ m, similar to the method previously presented for methane at 7.8  $\mu$ m.<sup>7</sup> Thus a grating with central wavelength centered at 4.3  $\mu$ m was modeled and fabricated, based on the knowledge gained from the 3.0  $\mu$ m grating fabrication and modelling procedure.

Waveguide modelling followed the same procedure outlined above. The  $\Delta n = 0.031$  and AR = 0.175 parameters were maintained from the 3 µm grating model, and the thickness was adjusted to predict the thickness required for peak first order diffraction at 4.3 µm. Based on the refractive index of IG2 at 4.3 µm, n = 2.5123, the thickness required was predicted to be 192 µm. This was input to the model, and the discrete angle scan repeated as described above. This model also implements the  $\pm 1.35^{\circ}$  converging AOI to account for the widening of the spectral response observed at short wavelength. The data is shown in Figure 5(a) below.



Figure 5. Modeled (a) and measured (b) data for a grating designed for peak first order diffraction at 4.3 µm.

The fabricated grating spectral response is shown in Figure 5(b), for various AOI as before. In general, there is good agreement between the form of the measured and modeled data, with broader FWHM at the short wavelength part of the spectrum, narrowing toward longer wavelength. The superblaze shows similar form in both cases; the model predicts slightly longer peak first order diffraction wavelength ( $4.5 \mu m$ ) than the data shows ( $4.2 \mu m$ ). The reason for this is likely due to the change in refractive index modification with increasing depth within the IG2 substrate; for deeper inscription planes, the IG2 causes more aberration to the inscription beam, and thus the extent of refractive index modification is likely to be smaller further inside the material. This would account for the peak diffraction wavelength occurring at slightly shorter wavelength, as the average refractive index modification is smaller in this case. Model definition showed that reducing the refractive index modification leads to peak first order diffraction at shorter wavelength.

#### 6. CONCLUSION

We have demonstrated transmission diffraction gratings fabricated by ultrafast laser inscription in IG2 chalcogenide glass. Detailed modeling of the grating structure indicated the fabrication parameters used for inscription, and predicted the grating thickness required to fabricate gratings suitable for peak diffraction at longer wavelength. This was corroborated by experimental verification. The measured peak diffraction efficiency was 44% for a 3 µm grating, and 34% for a 4.3 µm grating.

Modeling of the grating spectral response also indicated characteristics of the measurement system that mask the true diffraction efficiency of the grating. For the 3  $\mu$ m grating, it is expected that measurement using a truly collimated beam would indicate diffraction efficiencies close to 70%; application of an anti-reflection coating would therefore increase the diffraction efficiency at the design wavelength to > 95%. This predicted performance would enable use of these gratings as efficient diffraction components for atmospheric spectroscopy and astrophotonics applications.

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