Simple method for antireflection coating ZnSe in the 20 μm wavelength range

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We present a simple, inexpensive, and effective method of applying antireflection coatings to zinc selenide windows designed to operate in the thermal infrared wavelength region.

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1. Introduction

Sensitive infrared spectrometers and radiometers, which operate in the thermal infrared spectral region, require that their detectors be cooled to cryogenic temperatures, typically through the use of liquid nitrogen or closed cycle coolers. In these systems the detector is placed in a vacuum vessel, which requires that the entrance optical window of this dewar assembly provide a good vacuum seal. The window material must have sufficient mechanical strength to withstand the pressure gradient across it, be able to form a vacuum tight seal with the dewar assembly, withstand a range of environmental settings (e.g., temperature and moisture), have high optical transparency in the spectral region of interest, and, ideally, be of low cost.

In our application, an infrared radiometer operating at $\sim 20\,\mu$m,[1,2], zinc selenide (ZnSe) meets nearly all the above requirements, however, it suffers from large reflective surface losses due to its high refractive index, $n_{\mathrm{ZnSe}} = 2.46$, which results in a transmission of ~60% in the 10–20 μm range. We have therefore developed a simple, yet effective method for antireflection coating ZnSe that raises its transmission to over 90% in this spectral range.

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2. Theory

When an electromagnetic wave encounters a boundary between two media having different electrical properties, a fraction of the wave will be reflected. Electromagnetic theory requires that the tangential components of the electric and magnetic fields are continuous across the boundary from which Fresnel's equations, which describe the amplitude reflection, r, and transmission, t, coefficients of the incident wave at the boundary, can be derived (see any standard optics textbook [3]). For radiation normally incident on a boundary separating two media of refractive index n_1 and n_2, then r and t can be expressed as

$$r = \frac{n_2 - n_1}{n_2 + n_1}, \qquad t = \frac{2n_1}{n_2 + n_1}. \tag{1}$$

In the case of ZnSe–air/vacuum boundaries, assuming no absorption losses and normally incident light, the reflected wave amplitude at the air–ZnSe interface is ~0.4. An identical fraction of the transmitted wave amplitude is reflected from the outgoing ZnSe–vacuum interface.

The theory of interference shows that if the ZnSe is coated with a thin film of material of refractive index n_{AR}, whose optical thickness is equal to an odd multiple of $\lambda_0/4$, then the reflected power will be zero for normally incident light if $n_{\mathrm{AR}} = \sqrt{n_{\mathrm{ZnSe}} n_{\mathrm{Air}}}$ [3].

In the case of ZnSe we require a material with $n_{AR} = \sqrt{2.46} \approx 1.57$.

Polypropylene (PP), which has a refractive index of $n = 1.48$, is reasonably close to the required value of 1.57 and has the major advantage of being readily available in a large variety of thicknesses. The minimum, and thus optimal, thickness antireflection PP coating required for a ZnSe window designed to operate at a wavelength centered at 20 μm becomes $d \approx 20\,\mu\text{m}/(4 \times 1.48) = 3.38\,\mu\text{m}$.

3. Applying Antireflection Coating

Having identified the optimal thickness of the antireflection coating, the challenge is to form a robust bond of the PP to both surfaces of the ZnSe. An additional concern in our application is that the interior coating is inside a sealed vacuum dewar attached to a Stirling cycle cooler. Any outgassing from contaminants in the PP, or air trapped under the coating, will lead to a loss of vacuum and failure of the cooler to reach its operating temperature.

PP sheets of thickness closest to the optimum value (3.3 μm) were first cleaned by plasma ion etching and then washed with isopropyl alcohol. Circular disks of PP, the same size as the ZnSe windows, were cut out from the PP sheet (first placed on a few sheets of copy paper), using a punch with a slight twisting motion. The resulting thin pieces of PP were then floated into position on the ZnSe window, and excess isopropyl alcohol was carefully removed with a tissue. When dry, the process was repeated for the other side of the window. The resulting PP/ZnSe/PP sandwich was then placed in a vacuum oven press assembly and heated to a temperature of 150 °C close to the melting point of PP (165 °C), while applying pressure and under vacuum.

Figure 1 provides details of the vacuum oven press. Two steel disks, 125 mm in diameter and 12 mm thick, are clamped together using threaded rods and nuts. A layer of silicone rubber foam (~4 mm thick) followed by a layer of silicone rubber sheet (~1.5 mm thick) are used to remove the effects of any blemishes on the steel surface and apply an even force across the faces of the windows. The innermost layer is a teflon sheet (75 μm thick), which holds the PP against the ZnSe, while not sticking to the PP at the operating temperature of the press. A torque of 10 Nm is applied to each of the six nuts that form the press. The completed unit is then mounted on a copper heating plate (diameter 127 mm, thickness 23 mm), wrapped in insulation (aluminium foil and fiberglass), and placed in a vacuum bell jar. Once a

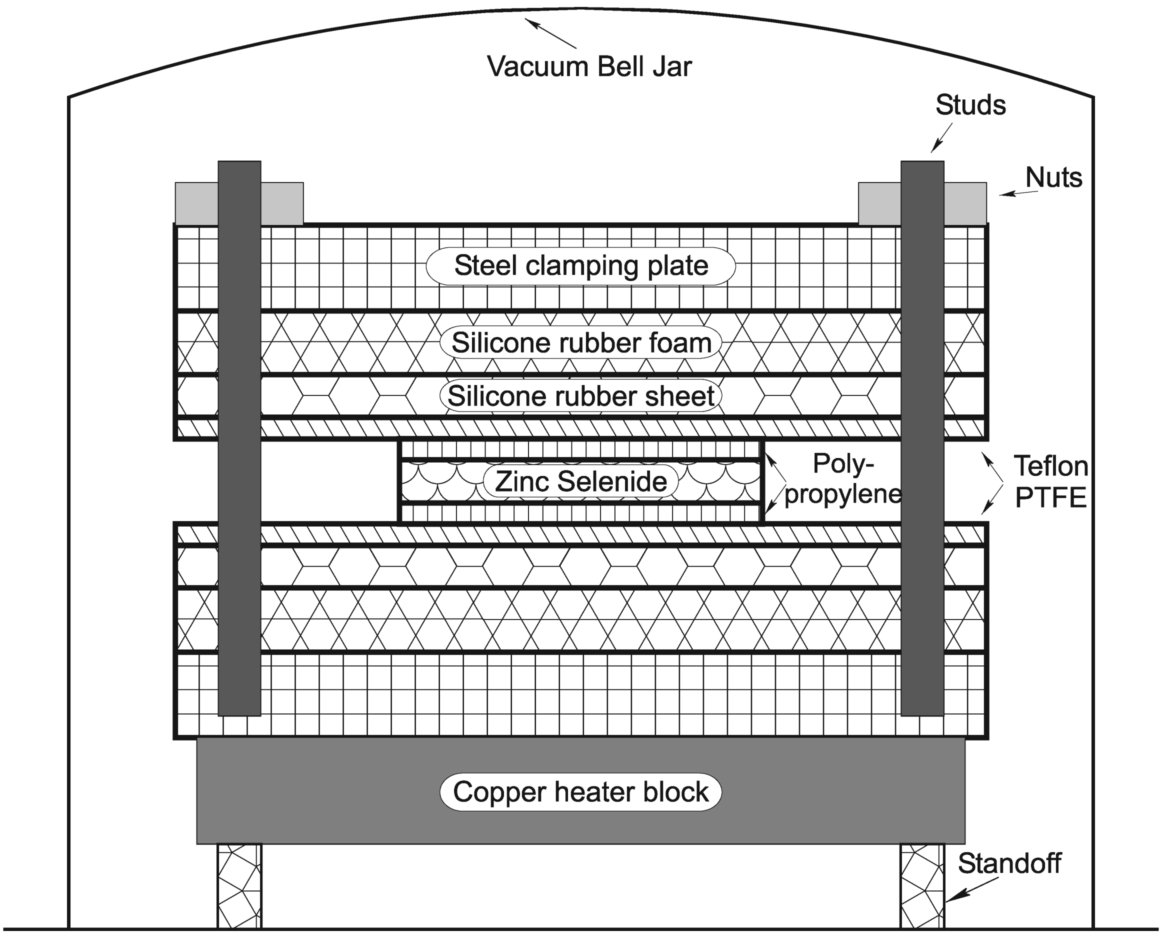

Fig. 1. Schematic of the vacuum oven press assembly used to apply the antireflection PP coating to the ZnSe window.

vacuum of $< 0.1\,\text{Pa}$ has been achieved, the copper block is heated to 190 °C. At this setting the measured temperature at the ZnSe window, determined by a colocated thermocouple, is ~150 °C. The vacuum oven is operated overnight and then allowed to fully cool to ambient temperature before readmitting the atmosphere. The sandwich can then be disassembled yielding the PP antireflection coated ZnSe window. In practice six ZnSe windows with a of diameter 12.7 mm can be coated on both sides in a given run with the present configuration.

4. Application for an Infrared Radiometer

We have developed a $20\,\mu\text{m}$ infrared radiometer for millimeter astronomy (IRMA) for measuring atmospheric water vapor column abundance above high altitude telescope sites [4]. The instrument uses a mercury–cadmium–telluride detector cooled to ~70 K with a Stirling cycle cooler, which requires that the detector be housed in a small vacuum vessel. Since the vacuum vessel must be permanently sealed and maintain a pressure $\leq 0.02\,\text{Pa}$, with a hold time of years, an extremely low leak rate is required to accommodate the relatively small getter capacity. Moreover, since the radiometer will encounter a range of environments (in terms of temperature, pressure, and humidity) from the controlled conditions in the laboratory to the hostile conditions at remote mountain sites, the window must be leak proof, nonhygroscopic, and robust to changing environmental conditions. ZnSe met all these requirements and was selected as the window material.

Figure 2 shows the measured transmission as a function of wavelength for an uncoated ZnSe window of thickness 1 mm (solid curve), and one that has received a $3.3\,\mu\text{m}$ PP antireflection coating on both surfaces (dashed curve). These measurements were obtained with an ABB Bomem MB 102E Fourier transform spectrometer with the window placed in the $f/4.5$ sample chamber beam. The dotted curve shows the theoretical prediction for these conditions. The improved transmission of the infrared radiometer of a factor of ~35% over the range of interest (indicated by the vertical curves) agrees well with theory. The absorption features at shorter wavelengths are due to the PP itself and will have to be considered in antireflection coatings designed for other regions.

This simple yet effective method for antireflective coating ZnSe results in a significant increase in transmission at a wavelength of $20\,\mu\text{m}$. IRMA units employing these antireflection coated windows have been in operation for several years at the Gemini South telescope in Chile as well as shorter periods with several other telescope organizations [e.g., Las Campanas, European Organization for Astronomical Research (ESO), Thirty Meter Telescope (TMT), SMA, James Clerk Maxwell Telescope (JCMT)]. Throughout all of these deployments we have never experienced any problems with the PP coating, nor have we observed any signs of delamination despite the daily thermal cycling experienced at the high altitude sites. The PP coatings are nonhygroscopic and have proven to be robust in the IRMA

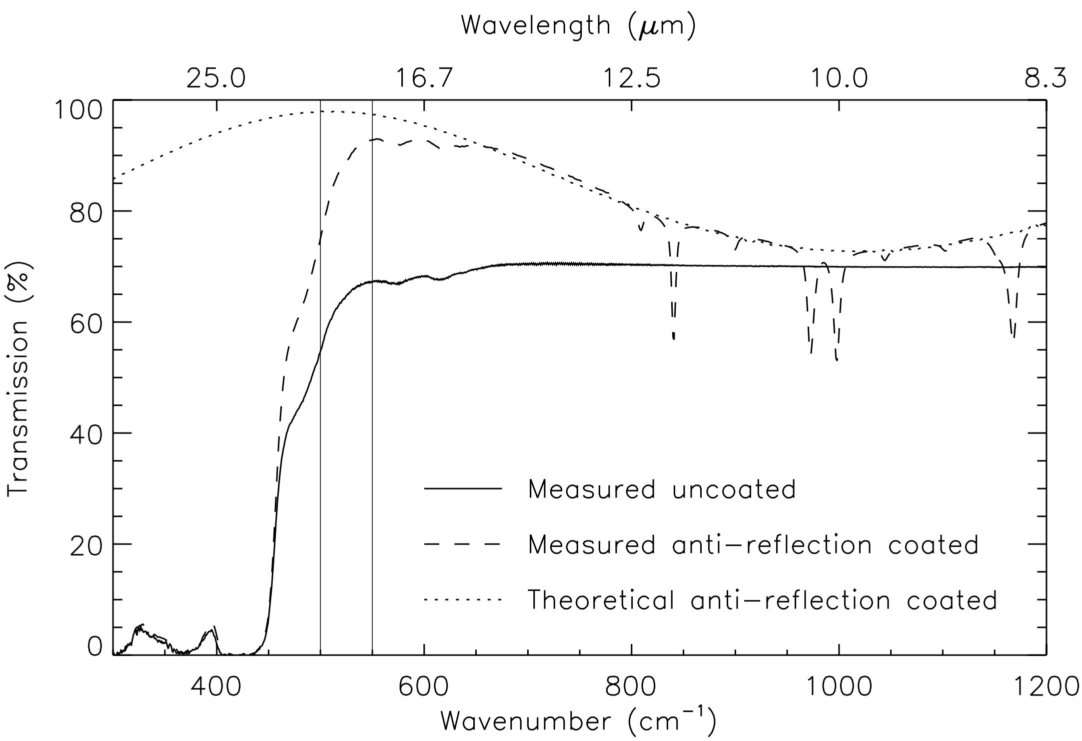

Fig. 2. Measured transmission of a 1 mm thick piece of ZnSe with (dashed curve) and without (solid curve) a $3.3\,\mu\text{m}$ coating of PP on both surfaces. The dotted curve shows the expected improvement predicted by theory. The bandpass of the radiometer is indicated by the vertical lines.

units where they view the atmosphere via a 90° off-axis parabolic mirror. In 2008 an IRMA unit will be deployed in Antarctica. In rigorous cold testing and thermal cycling down to −80 °C in a laboratory freezer the coating remains intact with no signs of delamination.

5. Conclusion

A simple, inexpensive, and effective method of applying an antireflection polypropylene coating to ZnSe windows has been presented. The measured increase in transmission at 20 μm agrees well with theoretical predictions. The coating has proven to be robust over a wide range of environments and has never been seen to delaminate. The method of application renders the coating useful in applications employing closed-cycle coolers where vacuum integrity and extremely low outgassing are required.

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