Two-layer anti-reflection coating with mullite and polyimide foam for large-diameter cryogenic infrared filters

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We have developed a novel two-layer anti-reflection (AR) coating method for large-diameter infrared (IR) filters made of alumina, for the use at cryogenic temperatures in millimeter wave measurements. Thermally-sprayed mullite and polyimide foam (Skybond Foam) are used as the AR material. An advantage of the Skybond Foam is that the index of refraction is chosen between 1.1 and 1.7 by changing the filling factor. Combination with mullite is suitable for wide-band millimeter wave measurements with sufficient IR cutoff capability. We present the material properties, fabrication of a large-diameter IR filter made of alumina with this AR coating method, and characterizations at cryogenic temperatures. This technology can be applied to a low-temperature receiver system with a large-diameter focal plane for next-generation cosmic microwave background (CMB) polarization measurements, such as POLARBEAR-2 (PB-2).

Keywords: AR coating, IR filter, Cosmic Microwave Background, Millimeter wave, Gravitational Waves, POLARBEAR-2

I. INTRODUCTION

As astronomical and cosmological observations always demand higher sensitivities, importance of large focal planes with detectors at cryogenic temperatures ever increases. The observation of the cosmic microwave background (CMB) polarization, which is one of the best probes for studying the early universe, is an outstanding example where such cryogenic large detector arrays are needed. Recently, a few CMB polarization experiments have reported the detection of the faint sub-degree-scale B-mode signal, the odd-parity mode of the CMB polarization pattern. The use of a large array with an order of 10,000 polarization detectors, such as the focal plane of POLARBEAR-2 (PB-2), SPT-3G, and BICEP-3, is essential for characterizing the B-mode power spectrum.

The need of a large focal plane leads to a challenge in the thermal design, as large thermal load is expected from a large optical window. In order to keep the detector sensitivity high enough, efficient infra-red (IR) filters should be considered. One promising solution to this problem is to introduce an alumina plate as an IR filter with high thermal conductivity. The alumina filter absorbs the incident IR emission efficiently. The absorbed power is conducted from the central region of the filter to the edge, which is thermally connected to the cryogenic stage. The thermal conductance of alumina in the temperature range between 50 and 100 K is very high; three orders of magnitude as large as those of conventional plastic filters, such as PTFE, nylon, and black polyethylene. The temperature gradient and optical loading of the alumina plate are thus very small even with a large diameter, which is ideal as the IR filter material.

A remaining technological challenge on the use of alumina IR filter is to establish AR coatings for a large frequency coverage. Alumina is known to be highly reflective in the millimeter wavelength. We thus need an AR coating on each surface of the alumina disc. AR coating on such a large surface is easily peeled off owing to the thermal shock at cold temperatures. The aforementioned next-generation CMB projects demand multi-layer AR coatings on large IR filters with a typical diameter of 500 mm. In the previous studies, the POLARBEAR group attempted to develop a two-layer AR coating method with epoxy on an alumina surface, with a diameter of 50 mm. However, the epoxy and alumina cracked during thermal cycling. To avoid the cracking, stress-relief grooves were adopted to reduce the stress between alumina and the epoxy layer. This technology...
was also used in the BICEP-3\textsuperscript{12} group, which succeeded in making a single-layer AR coating with epoxy for a diameter of about 500 mm. However, this method is expensive. Also, it is not easy to maintain the thickness uniformity of the AR layers on the thin alumina plate. Alternatively, the ACTpol\textsuperscript{13} group succeeded in making two- and three-layer AR coatings on silicon lenses, whose diameters were approximately 300 mm. They tried and succeeded using the technology of subwavelength grating (SWG). It is theoretically possible to apply this technology to an alumina surface; however, it is difficult because the dicing blade is subject to wear so that groove pitch and depth are changed. It is also difficult to make a large ingot of silicon. Machining the material is also very expensive. Yet another method studied for the SPIDER project is a polyimide sheet as an AR coating on the sapphire with a diameter of about 250 mm\textsuperscript{19}. The SPIDER method is very easy to perform and less expensive than the method with epoxy or SWG.

Finally, the first application of thermal sprayed mullite as AR coating was made on a 50 mm diameter alumina disc\textsuperscript{21}. It is also possible to tune the dielectric constant of a thermal sprayed layer by spraying microspheres and alumina powder with different ratios. A three-layer thermal sprayed broadband AR coating that achieves over a 100 % fractional bandwidth has been demonstrated on a 50 mm diameter alumina disc\textsuperscript{22}.

In this study, we newly employ the polyimide foam coating on the mullite coating described above to establish a two-layer AR coating method. We apply this technique to a thin and large alumina disc with a 460 mm diameter as an IR filter, and demonstrate the performance of the AR coating.

We fabricated a 2 mm thick and 460 mm diameter alumina filter, and demonstrated the performance of the new AR coating. Our development focuses on the use in future CMB measurements but the method developed can also be used for other applications. In Section || we describe the design of a large-diameter alumina IR filter with our new AR coating method. Section || explains our measurements of material properties. The fabrication process is detailed in Section ||. Section || shows our characterization of the AR coatings on a large-diameter alumina IR filter. We discuss our results in Section || and give conclusions in Section \textsuperscript{|||}.

II. DESIGN

To be concrete, our AR-coating development targets next-generation CMB experiments, where typical requirements on the IR filter and the AR coating are as follows:\textsuperscript{23}:

- The filter diameter must be extendable to 450 mm or larger;
- The AR coating is robust against thermal shock, so that it is usable in cryogenic temperatures;
- The uniformity of the additional optical path length due to the AR coating should be less than $\lambda/50$, where $\lambda$ is the wavelength of the incoming electromagnetic wave;
- Transmittance should be above 95 % at the detection bands, which we assume 95 GHz and 150 GHz with a 30 % fractional bandwidth for each;
- The 3 dB cutoff frequency should be below 1 THz.

The filter diameter requirement satisfies the requirement for a large focal plane with $O(10000)$ superconducting millimeter-wave sensors. The requirement on robustness against thermal shock is important for use at low temperatures. The thickness of the AR coating is designed to be $\lambda/4$ so that the reflected beam is canceled. The uniformity must be better than $\lambda/50$, which corresponds to a transmittance uncertainty of 1 %. The requirement on transmittance in detection bands comes from the design sensitivity of experiment. The requirement on the cutoff frequency is derived to reduce the IR emission whose main frequency range is 10-100 THz.

Figure 1 shows a schematic view of two-layer AR coatings on a large-diameter alumina disc. The design satisfies the typical requirements mentioned above. The AR coatings consist of the thermally-sprayed mullite, which has the index of refraction (IOR) of 2.5 as the first layer and polyimide foam (called Skybond Foam\textsuperscript{15} hereafter) as the second layer. A thin layer of low-density polyethylene (LDPE) is placed in between two AR layers as a glue layer. We employ the mullite as the first layer because the coefficient of thermal expansion (CTE) is close to that of alumina. The idea of using the Skybond Foam for the second layer is a natural extension of the polyimide sheet adopted by SPIDER, which was explained in the previous section. An important advantage of the polyimide foam is that the IOR can be controlled by changing the filling factor of the polyimide, which gives us an additional tuning knob to optimize the AR coating properties for given observation frequencies. Our example design for this study is optimized to achieve the highest integrated efficiency for simultaneous measurements centered at 95 GHz and 150 GHz with a 30 % fractional bandwidth for each. Here we fix the thickness of the alumina and the IOR of mullite, while the IOR of the Skybond Foam and the thicknesses of mullite and the Skybond Foam layers are free parameters. For the design in Fig. 1 the optimized IOR for the Skybond Foam is 1.43. Measurements of the properties of each AR layer as well as those of alumina we have used in this study are described in the next section.

III. MATERIAL PROPERTIES

We have measured basic properties of alumina, mullite and the Skybond Foam. The measurements include thicknesses, IORs, and the loss tangent values. Table \textsuperscript{||} summarizes our results.
The sintered polycrystalline alumina was manufactured by Nihon Ceratech corporation with a purity of 99.5 % (labeled as 99.5LD)\textsuperscript{18}. The loss tangent of the 99.5 % purity of the alumina is low in the detection band and high in the sub-millimeter wavelength. The large gradient of the loss tangent between the detection band and the sub-millimeter region results in a sharp IR cutoff. We have also tested alumina with purities of 99.9 % and 99.99 % from the same company and have concluded that 99.5LD is the best material. More details are described elsewhere\textsuperscript{9}.

### B. Mullite

Mullite is a high-performance ceramic stable at atmospheric pressure. We place the mullite on both alumina surfaces using the thermal-spraying method developed by Tocalo corporation\textsuperscript{12,13}. We measure the transmittance of the mullite at 298 K and 81 K. The measured frequencies are between 72 and 148 GHz. This measurement yields fringe patterns from the interferogram of the alumina and mullite layers. Mullite has asperity structure, with a surface roughness $R_s$ and a ten-point average roughness $R_z$ of 6 µm (6 µm) and 36 µm (34 µm) at one (the other) side, respectively. We apply the effective medium theory (EMT)\textsuperscript{20} to the surface of mullite to obtain the effective IORs as follows:

$$n_{\text{eff}} = n_m \sqrt{\frac{2(1-f)(n_0^2 - n_m^2) + n_0^2 + 2n_m^2}{2n_m^2 + n_0^2 + (1-f)(n_m^2 - n_0^2)}},$$

where $n_{\text{eff}}$, $n_m$, and $n_0$ are the effective IOR, IOR of mullite and vacuum, respectively, and $(1-f)$ is the filling factor, defined as $1 - R_s/R_z$. We can approximate the asperity surface as the virtual layer from the EMT as illustrated in Fig. 2. The estimated IORs and loss tangents in Table I are obtained using the EMT.

### C. Skybond Foam

The Skybond Foam is an polyimide foam, which is manufactured by the IST corporation\textsuperscript{15}. Figure 3 shows photomicrographs of the Skybond Foam with a filling factor of 10 % and 60 %. The filling factor is defined as the volume-filling fraction of the polyimide in the mixture, which can be controlled from 10 % to 100 %. Each cell of the Skybond Foam has a size of $\sim 10 \text{ } \mu\text{m}$. In general, the IORs and loss of the material depend on the filling factor\textsuperscript{21}. We measure the IOR and loss tangent of Skybond Foam with different filling factors between 10 and 100 % (10, 20, 30, 50, 60, and 100 %). The size of each sample is 100 mm $\times$ 100 mm with a thickness of 10 mm. The thick Skybond Foam was chosen to perform measurements with high signal-to-noise ratios.

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**TABLE I. Basic properties of alumina and AR materials.**

The thickness $d$, the IOR $n$, and the loss tangent $\tan \delta$ are listed. Our measurements for $n$ and $\tan \delta$ are carried out both at room temperature and at the temperature of liquid nitrogen, whereas the thickness values are only measured at room temperature. The errors include both statistical and systematic uncertainties. The systematic errors mainly arise from the uncertainty of the AR thicknesses and temperature variations during the measurements.

| Material       | $T$ [K] | $d$ [mm] | $n$ | $\tan \delta$ [x$10^{-4}$] |
|----------------|---------|----------|-----|----------------------------|
| Alumina        | 298     | 1.98 ± 0.01 | 3.144 ± 0.005 | 3.7 ± 1.7 |
| Mullite        | 298     | 0.24 ± 0.02 | 2.52 ± 0.02 | 121 ± 16 |
| Skybond Foam   | 298     | 0.39 ± 0.02 | 1.436 ± 0.025 | 120 ± 2 |
| Alumina        | 81      | –         | 3.117 ± 0.005 | 3.0 ± 1.1 |
| Mullite        | 81      | –         | 2.46 ± 0.03 | 53 ± 10 |
| Skybond Foam   | 81      | –         | 1.425 ± 0.025 | 25 ± 1 |

The IOR and the loss tangent are obtained from transmission measurements. A detailed description of the measurement system is available elsewhere\textsuperscript{2}. The beam at millimeter wavelength (72-108 GHz and 108-148 GHz) is produced by a signal generator with a sixfold and eightfold frequency multiplier\textsuperscript{16}. The frequency range of the signal generator is between 12 and 18 GHz. The beam is collimated by an ultra-high-molecular-weight polyethylene lens and the measured sample is placed immediately after the aperture. The transmitted radiation is detected by a diode detector. The signal is chopped at 13 Hz for modulation with a lock-in amplifier, and the modulated signal is obtained using a DAQ. The detector is moved along the optical axis for more than a half wavelength to subtract the effect of a standing wave in the measurement setup. We measure the power with and without the sample and take the ratio between them to obtain the transmittance. The measured transmittance is fitted with the Fresnel formulas\textsuperscript{17}. In the following sections, we describe more details on each material.
FIG. 2. The schematic view of the mullite surface and the model with the effective medium theory (EMT). The mullite has an asperity surface, which we can approximate as the virtual layer based on the EMT. The effective IOR of the virtual layer is described by Equation (1). This equation depends on the filling factor which is given by the ratio between $R_a$ and $R_z$.

FIG. 3. Left: Photomicrograph of Skybond Foam with a 10% filling factor. The bubbles are pressed with a 10 ton weight. The average diameter of the unit cells is 10 µm. Right: Measured sample of Skybond Foam with a 60% filling factor. The measured IOR and loss tangent at 298K and 81 K are shown in Fig. 4. The error bars in the y axis correspond to systematic error of millimeter source fluctuation. The size of the measured samples are 100 mm × 100 mm × 10 mm.

FIG. 4. The IORs (top) and loss tangents (bottom) as a function of filling factor. The sample is placed at 298 K or 81 K. We decide to use a filling factor of 60% for the AR material because it gives the IOR closest to the target value of 1.43.

FIG. 5. An alumina filter with a two-layer AR coating on both surfaces. The diameter of the alumina disc is 460 mm. The diameter of the AR layers is 440 mm. The 10 mm edge part of the alumina discs is without the AR coatings and is used to have a direct thermal contact.

The measured IOR and loss tangent at 298K and 81 K are shown in Fig. 4. The black curve is the best fit for the 81 K measurement, where the EMT with Eq. (1) is used. The estimated IORs are $n_0 = 1.049 \pm 0.005$ and $n_m = 1.748 \pm 0.006$. We decide to use a filling factor of 60% for the AR material because it gives the IOR closest to the target value of 1.43.

IV. FABRICATION

Figure 5 shows a photo of the fabricated alumina IR filter with the AR coating with mullite and Skybond Foam. We use an alumina disc with a diameter of 460 mm and a thickness of 2 mm. We place the mullite layer with thermal spraying on both sides of the alumina. The thickness of the mullite is (0.24 ± 0.02) mm as already listed in Table I. We place a sheet of low-density polyethylene (LDPE) with a thickness of 30 µm as the bond layer. This layer is sandwiched by the Skybond Foam and mullite surface. The thickness of the Skybond Foam is (0.39 ± 0.02) mm as already listed in Table I. The surface of Skybond Foam is then shaped with a milling
TABLE II. Measured uniformities of the Skybond Foam and mullite. The mean and rms of the thickness, $d$, and IOR, $n$, are calculated from the measurements shown in Figs. 7 and 8.

|            | $d$ [mm] mean | $d$ [mm] rms | $n$ mean | $n$ rms | Optical path length [mm] |
|------------|---------------|--------------|----------|---------|-------------------------|
| Mullite    | 0.250         | 0.010        | 2.46     | 0.063   | 0.016                   |
| Skybond Foam | 0.391         | 0.008        | 1.43     | 0.020   | 0.009                   |

machine, whose diameter and rotating speed of the blade are 4 mm and 6000 rpm, respectively. The uniformity of the thickness is improved by a factor of two through the machining process. The stack of Skybond Foam, mullite and alumina layers are then baked in an oven. We sandwich the stacked layer with 10 mm thick aluminum boards and press them using lead blocks with a pressure of 3,000 N/m². We adopt the baking recipe established by the SPIDER group. We start the baking cycle by slowly ramping up the temperature from room temperature to the baking temperature of 160 °C over 6 hours to avoid thermal shock. After keeping the baking temperature for 6 hours, we spend another 6 hours to ramp down to room temperature.

V. CHARACTERIZATION

We perform a series of measurements to verify that the fabricated large-diameter sample satisfies each requirement listed in Section II. First of all, we run a thermal cycle test from 300 K to 77 K with conductive cooling. The AR coating passes the thermal cycle test over ten times. No damage such as cracks due to thermal contraction is found.

To check the uniformity, we select 9 positions shown in Fig. 6 and measure the IOR and loss tangent at room temperature for a frequency range between 72 and 108 GHz. The instrument described in Section III is used for the measurements. The results are shown in Fig. 7.

We then measure the thickness uniformity of the Skybond Foam and mullite using a coordinate measuring machine (CMM). The results are shown in Fig. 8. We calculate the optical path length in both Skybond Foam and mullite with the measured IOR and thickness. These fluctuations are less than $\lambda/50$ as shown in Table II.

We measure the transmittance of the alumina filter with the same setup described in Section III at room temperature and at 81 K cooled with liquid nitrogen. Since the uniformity measurements at room temperature guarantee that the fabrication process is independent of the size of the filter, to perform measurements with liquid nitrogen, we fabricated a sample with 50 mm in diameter to avoid mechanical conflict with the measurement system. Figure 9 shows the result. The black lines show fit results with the EMT. The estimated average transmittances are 95.9 % and 95.8 % in the 95 GHz and 150 GHz

FIG. 6. Definition of the points of uniformity measurements. The point number 5 is at the center of the sample. We measure transmittances at the 9 points for estimation of the IOR. The measured IOR’s are shown in Fig. 7.

FIG. 7. Uniformity of IORs (top) and loss tangents (bottom) for the 9 measurement points. The Skybond Foam and mullite are measured with the millimeter wave source with a frequency range from 72 to 108 GHz. The sample is placed at 298 K. The error bars correspond to statistical uncertainties.
FIG. 8. Thicknesses of the Skybond Foam and mullite layers which are characterized by CMM to a precision of 2 µm. The red and blue points are the characteristic directions of the X and Y-axes, respectively. The each position corresponds to the distance from the center of the sample. The acceptable thickness uncertainties of Skybond Foam and mullite are 0.037 mm and 0.022 mm, respectively. The error bars correspond to systematic errors of the thickness rms of alumina. These results meet our requirements.

FIG. 9. The transmittances of the IR filter the function of the frequency at 298 K (top) and 81 K (bottom). Two frequency multipliers (sixfold and eightfold) are used to cover the entire frequency range.

FIG. 10. The transmittance of the IR filter at the THz band. The red crosses represent measurements at 298 K and the blue open circles are for 19 K.

The transmittance of the alumina filter at sub-millimeter wavelengths is measured using a Fourier transform spectrometer (FTS) with an InSb bolometer. A mercury lamp is used as a broadband thermal source. The detailed description of the FTS is found elsewhere. The transmittance is measured from 200 GHz to 1,500 GHz at two different temperatures of 19 K and 298 K. We prepare the alumina filters with a diameter of 20 mm for the FTS measurements.

The measured spectrum at 19 K has a higher transmittance than the spectrum at 298 K as shown in Fig. 10. The 3 dB cutoff frequencies of the filter are estimated as 400 and 650 GHz at 295 and 19 K, respectively. These 3 dB cut off frequencies are sufficiently lower than our design goal.

VI. DISCUSSION

We have demonstrated that the AR coating method with mullite and Skybond Foam satisfies all the requirements listed in Section II, which are typical in next-generation CMB experiments. Although we have focused on the measurements at 95 GHz and 150 GHz, a future CMB satellite mission LiteBIRD for example requires wider frequency ranges from 40 GHz to 400 GHz using two telescopes. Skybond Foam is tunable in IOR and can thus be considered as a candidate technology for such a space mission. We have newly applied the EMT in millimeter wave measurements to have the better surface model, which were not adopted in previous works. The fit results are in better agreement with data. The performances of the new AR coating method described in this study are as good as those with epoxy with grooves. The fabrication time of the method with mullite and Skybond Foam is much shorter than the epoxy coating, which is a clear advantage. Finally, this method can in principle be applied to curved surfaces, so that applications for lenses

bands, respectively. We estimate the emissivity, $\epsilon$, from the following equation:

$$\epsilon = A = 1 - R(d, n, \tan \delta, \nu) - T(d, n, \tan \delta, \nu),$$

where $A$, $R$ and $T$ are the absorption, the power reflectance, and the transmittance of the filter, respectively. The reflectances calculated from the parameters given in Table I are 2.1 % and 1.1 % at 95 GHz and 150 GHz bands. The estimated emissivities are 2.7 % and 4.2 % in the 95 GHz and 150 GHz bands, respectively.
should also be considered.

VII. CONCLUSION

We have newly introduced polyimide foam (Skybond Foam) as the AR coating material. We show that the IOR of Skybond Foam is controlled between 1.1 and 1.7 by changing the filling factor. The tunable IOR allows us to design a multi-layer AR coating much easier. We have made a two-layer AR coating with thermally-sprayed mullite as the first layer and Skybond Foam as the second layer on a 2 mm-thick alumina plate with 460 mm in diameter. We have confirmed that sample uniformity in the IOR and thickness is satisfactory for 150 GHz measurements. We have also confirmed that the AR coating is robust against thermal cycles. The measured transmittances of this filter at 81 K are 95.8 % for 95 GHz and 95.9 % for 150 GHz. The measured 3 dB cut-off frequency is 650 GHz at 19 K. The results satisfy requirements on IR filters for next-generation CMB polarization measurements where a large focal-plane and a large window are needed.

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