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Section A

Sintered Halon as a diffuse reflecting liner for light integration boxes

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Sintered Halon G-80, a polytetrafluoroethylene (PTFE) resin available commercially in powdered form, is a superb diffuse reflector. Sintered Halon has > 96% absolute reflectance at wavelengths between 300 nm and 380 nm, and about 98% absolute reflectance above 380 nm. It is chemically inert, is mechanically strong, does not fluoresce, and the high reflectance is easy to maintain over time. We have used sintered Halon as a lining in light integration boxes of water Cherenkov detectors. Preparation involves packing Halon powder into a mold to a specific density, then heating the material almost to the melting point (sintering). A method of preparing sintered Halon is presented. Physical properties of sintered Halon have been measured and we present those results here as well.

1. Introduction

Motivation for developing a water insoluble material with a high diffuse reflectance in the UV wavelengths arose in the design of the balloon-borne cosmic ray experiment PBAR [1-3] (which measured the flux of antiprotons high in the Earth's atmosphere). The resulting material, sintered Halon, was used subsequently in the SMILI experiment (Superconducting Magnetic Instrument for Light Isotopes) [4].

These experiments called for Cherenkov detectors with a kinetic energy threshold between 400 and 700 MeV per nucleon [MeV/n]. This energy range requires a Cherenkov radiator with an index of refraction between 1.2 and 1.4. The most common substances with indices of refraction in this region are clear liquids. One such liquid, highly purified water, is an ideal Cherenkov radiator with an index of refraction of 1.34 (threshold of 470 MeV/n) at wavelengths around 400 nm. Distilled deionized water is successfully used as a Cherenkov radiator in the IMB detector. The IMB collaboration has found distilled deionized water to have attenuation lengths of more than five meters from 350 nm to 700 nm, with maximal attenuation length of about 40 m at 400 nm [5].

The PBAR and SMILI Cherenkov detectors each required a light integration box to isotropize the Cherenkov light. Isotropization was obtained through the use of a diffuse reflector lining the interior of the box. Photomultiplier tubes (PMT), placed such that they viewed the light integration region, collected the light. Unlike previous Cherenkov detector designs which had the radiator adjacent to an air-filled integration region (e.g. ref. [6]), the integration box was completely filled with the liquid Cherenkov medium to avoid barriers (i.e. potential photon absorbers) between the Cherenkov medium, where the light is produced, and the integration region, where the light is isotropized and collected. Such barriers would otherwise be necessary in the case of liquid Cherenkov mediums in a balloon flight, where agitation of the payload occurs frequently. An early prototype of our detector is described by Bower et al. [7].

Considering that a Cherenkov photon will bounce several times before reaching a PMT, a high value for the reflectance of the lining is essential to the light collection efficiency of the box. The high reflectance is especially important in the UV and visible portion of the spectrum between 300 nm and 500 nm (a requirement imposed first by the fact that, in water, the majority of the Cherenkov radiation occurs in the UV, and second by the quantum efficiency of the photomultiplier tubes viewing the integration region).

Typical highly reflective coatings in the past have been paints based on $BaSO_4$ powder [8]. The reflectance of $BaSO_4$ is 99% at 500 nm, while the reflectance of the paint at this wavelength is 97.3% [9]. (See fig. 1.) Unfortunately, the paint is water soluble, making it unsuitable for our application.

The National Institute for Standards and Technology (NIST) has measured the reflectance of powdered Halon [10]. Halon G-80, a polytetrafluoroethylene (PTFE) resin, is one of many Teflon-like substances [11]. Halon is ideal for use in light integration boxes of

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water Cherenkov detectors since it is chemically inert, does not dissolve in water, does not fluoresce [12], and has a high reflectance in the UV and visible spectrum. Halon is obtained from the manufacturer in powder form, and requires sintering into solid sheets with enough mechanical integrity to withstand mechanical shocks. While it is possible to obtain sheets of compressed and sintered Teflon commercially, these sheets are semitransparent and shiny resulting from a higher presintering packing density. This is manifested in substantially lower reflectance values (80–90%) [13] than that of the "homemade" sintered Halon, rendering the commercial Teflon sheets less desirable for an integration box lining.

Sintered Halon is a sturdy, resilient, and robust diffuse reflector that has the advantage of being manufactured in sheets that can be attached with common fasteners. These sheets make excellent diffuse reflectors whether in a liquid or in an air filled integration box. It is possible to bend thin sheets of sintered Halon to conform to curved surfaces. In contrast, the mechanical integrity of $BaSO_4$ paint is poor compared with that of sintered Halon. The $BaSO_4$ paint tends to chip and flake when subjected to moderate shocks. Due to the extremely low viscosity of this paint, it is difficult to apply the paint to curved surfaces. When soiled, the $BaSO_4$ paint cannot be refurbished, and thus must be replaced. Sintered Halon can be washed or lightly sanded to freshen the surface if reflectance is degraded due to dirt or grease deposits. Sintered Halon is denser ($\rho \approx 1.4$ g/cm^3) than $BaSO_4$ paint, and to insure structural integrity in large sheets (> 600 cm^2), approximately 6 mm sheet thickness is required. The Halon sheets may be cut to fit the integration region and can be held in place with a series of nylon screws. We have recycled sintered Halon from old detectors and put it in new detectors, merely cutting the new desired shapes from the old pieces and lightly sanding the old pieces to regain reflectivity.

Sintered Halon has been used in other applications, including gray-scale standards [14] and laser cavity linings [15]. A paint based on PTFE has been manufactured [16]. While the paint is washable and a thinner coating is required to form the reflective surface, the reflectance is degraded due to the alcohol binder used in the paint to 95% in the visible and about 90% in the important UV region, much lower than the sintered Halon (fig. 1).

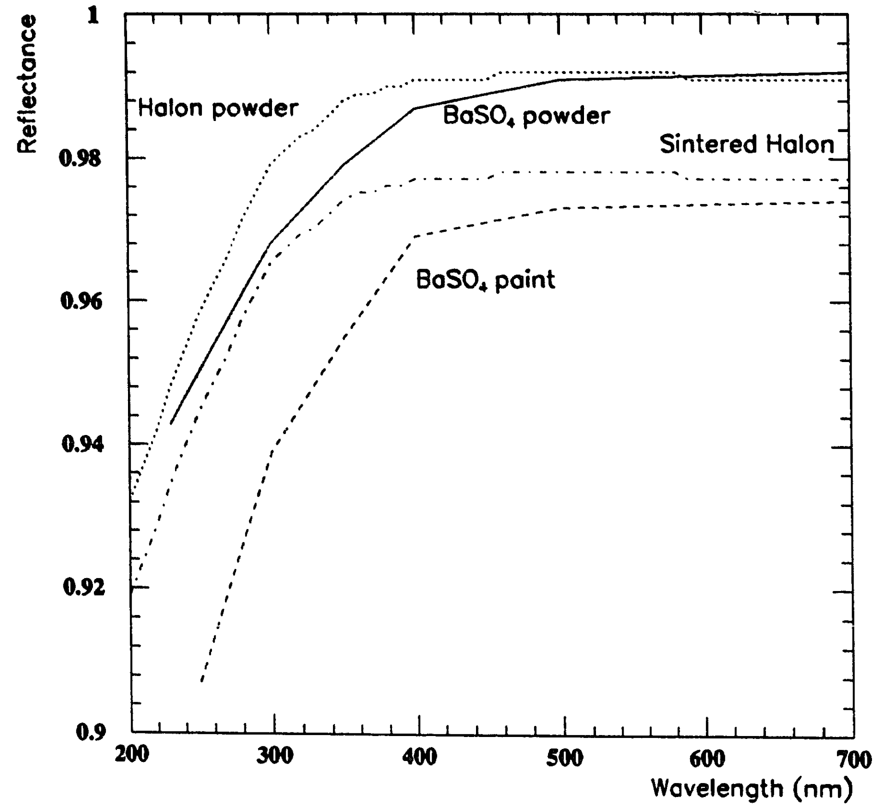

Fig. 1. Shown is the diffuse reflectance of $BaSO_4$ powder (solid line) [8], $BaSO_4$ paint (long dash) [9], compressed Halon powder (dots) [10], and sintered Halon (dot-dash) in the relevant wavelength range 200–700 nm. The reflectance of the sintered Halon is taken to be 98.6% that of the Halon powder (see discussion in text). Note that Halon powder is the best overall reflector up to 600 nm, where $BaSO_4$ powder becomes the better reflector, and that sintered Halon is a better reflector than $BaSO_4$ paint.

2. Recipe for preparation of Halon sheets

Published reports exist on the reflectance properties of pressed Halon [10,12], but there are few references on sintered Halon manufacturing [3,14], and they are rather Spartan in detail. Therefore a detailed recipe is presented in this article. This procedure is an expansion of a formula developed at NIST [17]. The recipe is simple, and leaves little to acquired skill. The Halon powder is prepared, then packed into a mold in order to obtain the desired density and shape. The packed molds are stacked inside a kiln, and a heating recipe stringently followed. After cooling, the Halon sheets are removed and checked for structural integrity. The molds may then be reused.

2.1. Necessary equipment

Equipment necessary to produce simple flat sintered Halon sheets ready for use in a light integration box includes a standard household blender to break down lumps in the powder, glass and aluminum sheets for molds, a kiln capable of achieving and maintaining with precision the temperatures required during sintering (365 ± 10 °C), and thermometers for monitoring the temperature. Cloth-backed aluminum oxide sandpaper with grits between 80 and 150 is suggested.

2.2. Preparation of Halon powder

The quantity of Halon used to fill the mold is determined by the amount required to pack the powder in the mold to a density of 1 g/cm^3. This density has been determined to provide optimal reflectance for the powder [12]. Before filling the mold, the Halon powder is premeasured and run through a few quick cycles in a household blender to remove any lumps present when it is obtained from the supplier. As the blender must remain clean during use, it is best to dedicate the blender to the job of breaking up the Halon in order to avoid contaminants.

Halon particle size averages between 20 and 25 μm [18]. While it is possible to obtain PTFE resins with larger particle sizes, research has found that Halon grain dimensions on the order of 50 μm and less provide better reflectance properties [15].

2.3. Molds

The molds consist of a central form of 0.635 cm (0.25 in.) aluminum plate with the desired shape cut out of the center, glass plates placed above and below the Halon powder, and two aluminum plates to lend support to the configuration during any transport and when stacked in the kiln (see fig. 2). Allow for an additional 20% extra in each length of the central shape-determining form to account for shrinkage of the Halon during sintering and any edge deformities which may arise. Pre-sintered Halon thickness values above 0.635 cm are more sensitive to the sintering process and may result in burnt edges and unsintered centers, much like improperly baked bread. Also, thicker sheets require longer cool-down periods to avoid curling the sheets. We are unfamiliar with the characteristics of thinner sintered sheets.

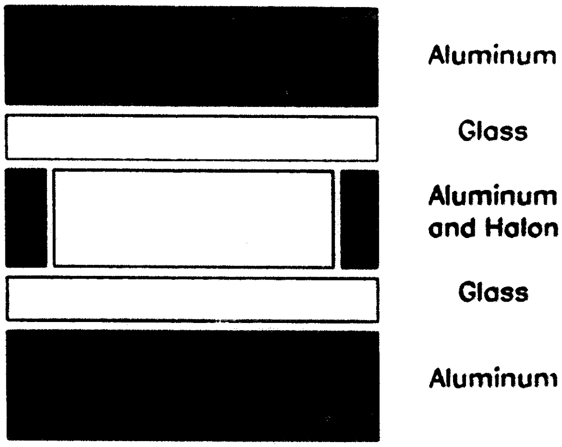

Fig. 2. Arrangement of kiln mold for producing sintered Halon. Layers of sheets of 0.635 cm (0.25 in.) aluminum plate and 0.3175 cm (0.125 in.) window glass plate sandwich a 0.635 cm thick region of Halon powder packed to a density of 1 g/cm^3. The 0.635 cm thick central aluminum form is removed before the sintering process is begun. It should be replaced with some ceramic spacers of the same thickness to retain the pressed Halon powder's shape.

The mold is packed by first placing the aluminum form on one of the glass sheets, then filling it uniformly with the prepared powder. It is important to use a flat, clean tool of large area to compress and pack the powder into the form. After packing, the central aluminum form is removed. If left in contact with the Halon during the sintering process, the aluminum will contaminate the Halon, lowering the final reflectivity. The other glass sheet is then placed on top of the packed powder. It is best to provide spacers (e.g. ceramic tiles of the correct thickness) around the Halon shape to prevent deformation caused by unequal pressures due to stacking or transport. The top and bottom aluminum support plates are put in place adjacent to each glass plate, forming a Halon "sandwich". This sandwich is kiln-ready, and can be stacked with others to make use of all possible space in the kiln. Spacers between molds to allow heat circulation are recommended.

2.4. Sintering process

It is of vital importance to find a suitable oven/kiln for the sintering process. We have had success using ceramics kilns. The desired sintering temperature is 365 ° C, far below the usual temperatures used in firing pottery clay. It is important to control the temperature throughout the Halon within ± 10^3C during the sintering process. The Halon temperature should never exceed 425 ° C, since at that temperature chemical decomposition occurs and harmful products are given off [19]. One type of kiln which exhibits good temperature control and the ability to run at low temperatures uses electric coils to produce the heat. The coils are arranged around the kiln interior, and if the Halon is brought up to temperature slowly, uniform heating is maintained.

The Halon baking recipe to produce approximately 0.5×0.5 m^2 sintered Halon pieces is as follows. Bring the Halon up to 365 ° C slowly to allow for even heating. We recommend 4–6 hours for this process. Maintain the temperature for one hour at 365 ± 10 ° C. At the end of the heating process, bring the Halon down to room temperature slowly. Too quick a cooling rate results in "cupping" (i.e. non-planar sheets). We allowed 8–15 hours for the kiln to reach room temperature.

Constant monitoring of the temperature is required. Several locations in and around the Halon should be monitored simultaneously. We used three temperature probes during the sintering. One probe was positioned to monitor kiln air temperature. Two others were placed such that they were in contact with the Halon during sintering. We placed the probes in Halon near the bottom and top of the stack of molds in order to monitor location-dependent temperature variations. If the temperature is not uniform, heating can occur too quickly for thermal equilibrium to be established.

The quality of the resulting Halon pieces may vary. Signs of improper sintering are crumbly, soft pieces, indicating a temperature that was too low, or pieces with brown edges, indicating uneven heating or temperatures that were too high. Early attempts may show splits in the Halon sheets caused during shrinking if the Halon sticks to the glass. After the first or second time the glass in the molds is used, enough residual Halon powder will remain on the glass to form a slippery coating, allowing later pieces to shrink without cracking. It is important to keep the glass and Halon powder free of dust and dirt during the process, since these contaminants will be baked into the finished piece.

2.5. Machining sintered Halon

The sintered Halon slabs are firm but soft enough to cut easily with a razor blade. If holes are to be made, extremely slow speed drilling is recommended to prevent tearing the material. Never use contaminating lubricants such as machine oil during machining. Distilled water is a sufficient lubricant, if one is required. When the Halon sheets are ready to be attached in the detector, they should be sanded to roughen the surface of the Halon to remove any specular component present after the sintering process. This also increases the reflectance. A clean belt sander with aluminum oxide cloth belts of grits from 80 to 150 is well suited for this process. The belt sander can also be used with the rougher grit sandpaper to adjust the thickness of the Halon. Sanding by hand can be used for detailed shaping of the sintered Halon. Lightly hand-sanding the Halon restores the diffuse reflecting properties and noticeably improves reflectance if performance degrades due to contaminant buildup.

2.6. Measured reflectance values

We have measured the absolute diffuse reflectance of several sintered Halon samples. A Cary 14 spectrophotometer with an integrating sphere attachment was used to make the measurement. Both packed $BaSO_4$ powder and Halon powder were used as the reference reflector. The reflectance properties of pressed Halon powder are well documented [10,12]. We obtained results indicating that there is no difference between the reflectance of sintered Halon and powdered Halon to within the accuracy of the spec-

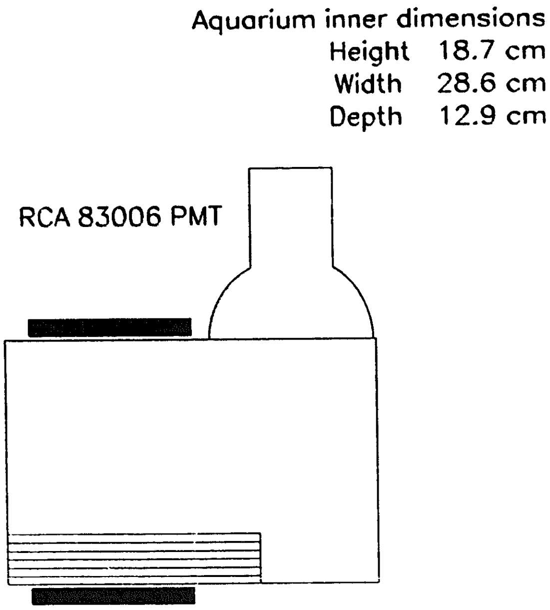

Fig. 3. Experimental setup to find Halon component of CK radiation. An aquarium of the dimensions shown was lined with the highly reflective sintered Halon, except for a hole for the PMT. Signals were recorded with and without six 0.635 cm ($\frac{1}{4}$ in.) pieces of Halon resting on the aquarium floor (shown as cross hatched boxes), using atmospheric muons to produce the Cherenkov light. Trigger paddles (solid boxes) above and below the aquarium provided the muon trigger. Details are presented in the text.

trophotometer (±1%). Detailed predictions of the Cherenkov photoelectron yields for both the PBAR [13] and SMILI [20] detectors, as well as a prototype Cherenkov counter [7], indicate that a sintered Halon reflectance of 98.6% that of powdered Halon best fits the measured photoelectron yield. Tests with the Cary 14 to determine the dependence of reflectance on Halon sheet thickness indicate no measurable dependence down to a 2.5 mm thickness. These results are in agreement with earlier measurements [12], which note no change in reflectivity of compressed Halon powder (ρ = 1.0 g/cm^3) down to a thickness of 4 mm.

The reflectances of Halon powder, BaSO$_4$ powder, sintered Halon, and BaSO$_4$ paint are compared in fig. 1. Note that both the Halon powder and sintered Halon are better diffuse reflectors in the UV region of the spectrum than the BaSO$_4$ powder and paint respectively, and are only outperformed by their BaSO$_4$ counterparts in the relatively unimportant wavelength range (for this detector) above 600 nm.

3. Sintered Halon as a Cherenkov radiator

In our detectors, the high energy charged particles of interest passed through both the Cherenkov medium and the sintered Halon lining. It is known that Teflon is an excellent Cherenkov radiator, with an index of refraction of about 1.33 (e.g. ref. [21]). However, the Teflon used in such detectors is much more densely packed than the Halon lining used here, and is partially translucent to the eye. We therefore proceeded to perform tests using atmospheric muons to determine first if Cherenkov radiation was emitted by the Halon lining, and second to quantify its contribution to the total signal.

The experimental setup consisted of a 9.5 l (2.5 gal) aquarium lined with a 0.635 cm layer of Halon (fig. 3). Air filled the integration region. The signal from a single RCA 83006 PMT was recorded by an Ortec 7100 multichannel analyzer (MCA) after the raw PMT signal was amplified in a charge-sensing preamplifier and shaped by an Ortec 575 pulse-shaping amplifier. To ensure that only signals from vertical muons traversing the whole of the aquarium were recorded by the MCA, trigger paddles (each consisting of a PMT and thin scintillator) placed above and below the aquarium provided a gate upon coincidence.

An identical signal was obtained from the Halon lining when the box was empty (except for the lining) and when the aquarium contained six 0.635 cm (0.25 in.) pieces of Halon stacked in the region of the box

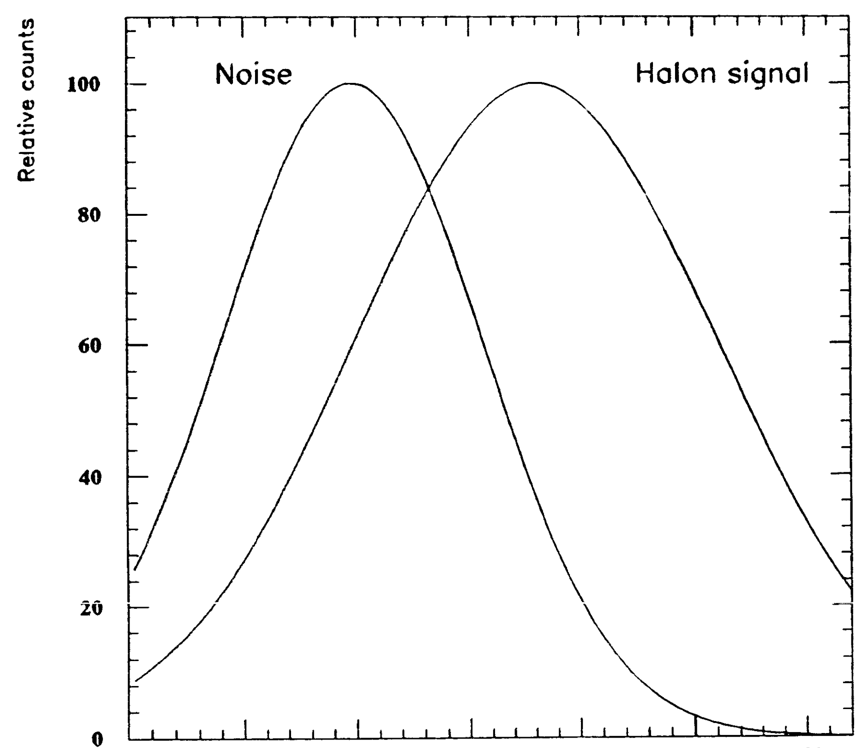

Fig. 4. Shown are Gaussian fits (counts per channel normalized for ease of presentation) to multichannel analyzer pulse height distributions from tests to quantify the amount of light emitted when a relativistic particle passes through sintered Halon. The test setup is described in the text.

sensitive to the trigger coincidence. The same signal was also obtained when a single extra slab of sintered Halon was placed at the top of the inside of the aquarium. (This was done to allow a chance for the downward-going Cherenkov light cone from the downward-going muons to enter the integration region.) A noise distribution was obtained by covering the face of the aquarium PMT and recording signals when a coincidence occurred. The spectrum obtained in this fashion matched one obtained during free-running of the PMT (no coincidence to reject noise or off-axis particles), and was different than the signal obtained from the lining. Also, covering the part of the aquarium walls sensitive to the coincidence trigger (i.e. the inner top and bottom of the aquarium in the path of atmospheric muons) with cellulose nitrate paper (e.g. Millipore paper) yielded the same noise distribution. The noise and signal pulse height distributions are shown in fig. 4.

The signals obtained with and without the extra Halon pieces present were the same, indicating that only a depth of 0.635 cm or less of Halon contributes to the signal. The ratio of the number of photoelectrons detected from Halon only to the number obtained with water present (after scaling by the depth of the water in the aquarium and assuming the Halon signal to be constant regardless of the water depth in the detector) was 9.3% cm. Application to the SMILI detector by scaling upwards to the 5 cm thickness of the water radiator indicates the approximate ratio of Halon signal to water signal to be 1.9%. The additional signal had no effect on data analysis.

4. Conclusion

The high value of absolute reflectance and nearly perfect diffusive properties of Halon make it an ideal candidate for light integration boxes. We have found that sintering Halon is an excellent way to create a rugged material for light integration box linings without degrading the reflectance properties substantially. Sintered Halon is easy to manufacture, easy to machine, chemically inert, mechanically strong, and reusable.

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