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Measurement of the Radiative Transport Properties of Reticulated Alumina Foams

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MEASUREMENT OF THE RADIATIVE TRANSPORT PROPERTIES OF RETICULATED ALUMINA FOAMS

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Washington, D.C., April 4-8, 1993.

ABSTRACT

Reticulated materials can play a key role in numerous solar energy applications including detoxification, power production, and process heat. These open-cell materials allow efficient volumetric absorption of solar energy and free flow of gases. They have large surface areas for supporting catalysts and for convective heating of gases, and they are compatible with high-temperature applications. The radiative transport properties of these materials must be determined before one can calculate how these materials scatter and absorb the sun's energy. However, the complex geometry of these materials is not amenable to direct calculation of their radiative transport properties. This paper presents a method for determining radiative transport properties of reticulated materials. The method has both experimental and analytical components. A polar nephelometer is used to measure the scattering profile of a sample of the reticulated material. The results of a Monte Carlo simulation of the experiment are then combined with the experimental results to give the scatter albedo and extinction coefficient. This paper presents the results of using this method to determine the radiative transport properties of four different porosities (10, 20, 30, 65 pores per inch) of cylindrical reticulated alumina samples ranging in thickness from 0.5 inches to 2.5 inches.

INTRODUCTION

In many applications of concentrated solar energy it is necessary to transfer energy delivered by the concentrator optics to a gas stream. Early concepts for accomplishing this typically involved pumping the gas inside a tube and placing the tube so that the solar flux was absorbed on the outer surface of the tube. Because gases exhibit poor heat transfer characteristics, and because the tube surface area is limited, it became clear that this concept would have little applicability. Recently, a new concept known as volumetric absorption has shown promise. A volumetric absorber is a porous structure placed in the concentrated solar beam. Sunlight can penetrate to the large surface area in the interior of the absorber and gas can easily flow through the absorber. Thus, the absorber provides a large surface area to which both sunlight and gas have access. It is this ability to provide a large heat transfer surface area that distinguishes volumetric absorbers from the early, surface absorption concepts.

Because of these perceived advantages, a number of solar applications of volumetric absorbers have been tested recently. Isshiki (1991) proposed using a volumetric absorber in the heater head of a Stirling engine as a means for simplifying the engine design, for reducing pressure drop in the engine working fluid, and for increasing the thermodynamic efficiency of the engine. Glatzmaier (1990) describes a solar reactor used to destroy hazardous organic vapors in air streams in which a volumetric absorber was used to preheat the air stream before it entered the gas-phase reaction zone. Skocypec and Hogan (1989) describe a receiver in which carbon dioxide reforming of methane was carried out. In this concept, a volumetric absorber was used as a catalyst support and as a means for heating the reactants.

Several materials can be used for volumetric absorbers. Reticulated alumina foams are composed of randomly located spherical voids in an alumina matrix. These were used in the work of Isshiki, Glatzmaier, and Skocypec and Hogan, described above. Alumina honeycomb is another candidate. Wire screen meshes and random wire packing also exhibit the desirable properties described above. The alumina materials are appropriate for very high temperature applications, while the metal wire

applies to somewhat lower temperature applications. In this paper, we focus on the reticulated foams. This is mainly because they have been proven in a number of solar applications, and because they are perhaps the "most random" of the candidate materials and, therefore, present the greatest challenge.

The same properties that make volumetric absorbers attractive for solar applications significantly increase the difficulty of analytically predicting the performance of the absorber. The random nature of the absorbing surfaces cause difficulties in determining such things as the local volumetric absorption of solar energy and the distribution of infrared reradiation throughout the absorber. To reliably predict the performance of a receiver using a volumetric absorber, we need the ability to predict these phenomena.

We are concerned here with determining the radiative transport properties of reticulated foams that are necessary for a complete analytical description of the radiation processes taking place in the absorber. These properties include the extinction coefficient, the single-scatter albedo, and the phase function. Because of the complexity of the reticulated foams, a purely analytical determination of these radiative transport properties is not possible. In the past, inverse analytical methods have been used to solve these types of problems. In an inverse analysis, some means for predicting the radiative transport behavior of the material in question is used in conjunction with experimental data to determine the radiative transport properties. In this paper we describe such an inverse analysis and apply it to the determination of the radiative transport properties of reticulated alumina foams.

BACKGROUND

Dunn (1981) and Subramaniam and Menguc (1991) describe an inverse method using the Monte Carlo method with importance sampling. Their method assumes an isotropic boundary condition and requires as input the optical thickness. The single-scatter albedo, or the spatial distribution of albedo for an inhomogeneous media, can then be determined. The value of importance sampling is that a single Monte Carlo calculation, rather than an iterative procedure between Monte Carlo solutions and the experimental data, suffices.

Roux and Smith (1981) used a nonlinear least squares convergence technique to minimize the difference between measured and calculated intensity distributions to determine scattering and absorption coefficients. The discrete ordinates method was used to produce the analytical data.

Sacadura, et al., (1986) described an inverse method for the determination of infrared absorption and scattering coefficients and the Henyey-Greenstein asymmetry factor of pure silica fiberglass. They used an optimization method to determine the radiative transport properties from the discrete ordinates method by fitting the experimental directional scattering measurements. Their results demonstrate that detailed directional scattering profiles, as opposed to hemispherically integrated scattering data are required to reliably determine the desired properties.

Skocypec and Hogan (1989) determined the spectral optical thickness and albedo for a reticulated alumina foam doped with rhodium catalyst. They used hemispherical reflectance and transmittance data with the two-flux method to produce estimates of these radiative transport properties.

In the work described here, we have combined the method of Roux and Smith (1981), nonlinear least squares convergence, with the advantages of the Monte Carlo method as described by Subramaniam and Menguc (1991). Our method can be summarized as follows. Assumed values of the radiative transport properties are used in a Monte Carlo calculation of the scattered radiation intensity distribution from a sample of the reticulated foam exposed to a collimated beam of laser light. The root-mean-square difference between this calculated intensity distribution and a measured intensity distribution is determined. The assumed radiative transport properties are adjusted and the Monte Carlo calculation repeated until this root-mean-square difference is minimized.

In this paper we first describe in detail the experimental aspect of the method. Next, we describe the analytical work and how it and the experimental results are combined to determine the desired radiative transport properties. Results are then presented for several samples of reticulated alumina foam. We then draw conclusions and make recommendations for future research.

EXPERIMENTAL METHOD

As discussed above, the inverse method we used requires the measurement of the distribution of scattered radiation from an illuminated sample or "frit." By distribution, we mean that the intensity is measured in the plane of the light source as a function of angle from the incident light beam. To accomplish this, we used a polar nephelometer. A nephelometer is a device used for light scattering measurements that includes a light source, the sample to be tested, and a light detector mounted on a pivoted arm. Mounting the detector on a pivoted arm allows for scattering measurements at different angles.

Figure 1 shows a diagram of the experimental apparatus. We used a collimated argon-ion (488 nm) laser beam as the light source. An integrated beam expander/collimator was located 15 cm from the laser aperture and provided an expanded beam with a diameter of 1.3 cm. Two beam-steering mirrors were used to allow the beam to intersect the center of the sample so that it was normal to the plane of the sample. The incident laser beam was chopped and the scattered signal was detected with a photo-diode detector. The detected signal was analyzed with a lock-in amplifier synchronized to the chopper frequency to eliminate the influence of stray light or emission.

The experimental technique consisted of illuminating a frit with a collimated beam of light and measuring the resultant scattering profile over a 320° arc in the horizontal plane that contained the incident laser beam. The first step was to align the laser and optics so that the laser beam was normal to the plane of the alumina sample. The alumina sample was then mounted in the holder. In order not to contaminate the alumina surface, care was taken to avoid skin contact with the experimental material samples. Alignment of the system was ensured by taking scattering measurements along the entire 320° arc and verifying a symmetric scattering profile. Once the experimental apparatus and the alumina sample were properly aligned, scattering measurements were performed. The photo-diode detector was centered at 0° and when the amplifier fluctuations settled to within 0.5% of the mean signal, the signal was recorded. The detector was then positioned at 10°, first on the positive and then on the negative side, for the next two measurements. This procedure was continued in 10° increments up to 160° so that the scattering results for each sample included 31 scattering measurements; 15 measurements from 10° to 160°, another 15 measurements from -10° to -160°, and one measurement at 0°. No measurements were taken at the 90° locations but tests were performed to ensure that photons were not being scattered from sample's circumferential edge. The last measurement taken was an additional 0° reading to measure how much the amplifier and/or laser drifted between the first and last scattering measurements. We normalized the results from each experimental session by the measurement at 140°. We chose 140° because, at this location, the signal was close to its maximum strength and all equipment on the nephelometer arm was clear of the incoming beam path.

The alumina samples tested ranged in thickness from 1.27 cm to 6.35 cm. The samples were all disk shaped and had a diameter of 8.89 cm. We performed calculations that indicated that this diameter was sufficiently large to simulate an infinite slab. The alumina foam is typically characterized by the number of pores per linear inch and samples with 10, 20, 30 and 65 pores per inch were tested. Pore size was not uniform for any pore-per-inch classification but the average pore size decreased as the number of pores per inch increased. Table 1 summarizes the sample characteristics.

Scanning electron microscope photographs of a 65-pore-per-inch (ppi) alumina frit in Figure 2 reveal the structural complications of the reticulated alumina. It can be seen that the material has two distinct length scales, both of which conceivably contribute to the light-scattering properties of the foam. There are small (on the order of 1μm), somewhat

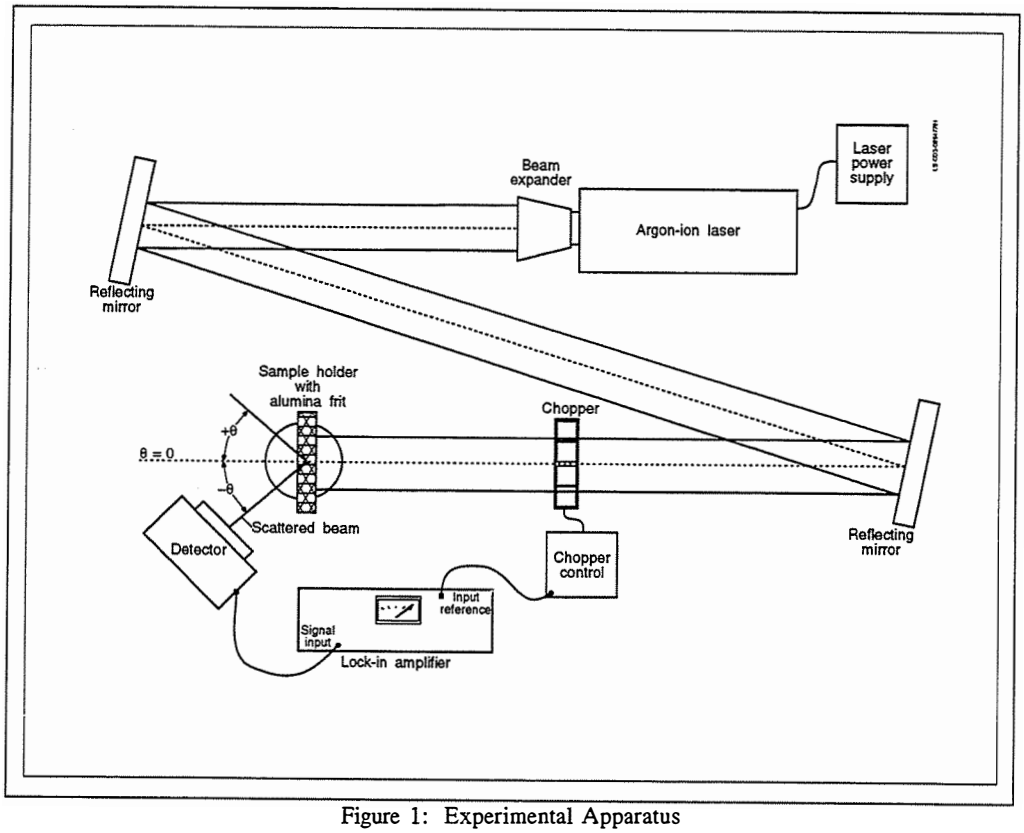

Figure 1: Experimental Apparatus

Table 1: Sample Characteristics

Pores per Inch	Thicknesses of Alumina Samples*				
	1.27 cm	2.54 cm	3.81 cm	5.08 cm	6.35 cm
10 ppi				⊗	
20 ppi		⊗	⊗		⊗
30 ppi		⊗			
65 ppi	⊗				

* All samples have a diameter of 8.89 cm.

spherical alumina particles, Figure 2a, and there are larger (on the order of 10-100 μm) cavities, or pores, bound by diffusely reflecting surfaces composed of the alumina particles, Figure 2b.

ANALYTICAL APPROACH

For modeling purposes the reticulated alumina was treated as a homogeneous, participating media that can scatter and absorb radiation. Emission was not considered because the alumina samples stayed close to room temperature during the experiment.

As mentioned above, we used the Monte Carlo method to model radiative transport in this study. The Monte Carlo method is a statistically based modeling method that simulates the lifetime of a bundle of photons as it progresses through the participating media. The technique is applied to simulate enough photon-bundle lifetimes to determine an average representation of the radiative transport characteristics of the medium. In this study the Monte Carlo method was used to simulate the scattering experiments described in the previous section.

The advantages of the Monte Carlo method are its mathematical simplicity and its applicability to complex geometries and properties. In addition, it is the best method for problems involving collimated light sources, Bohn and Mehos (1989). Because it is a statistically based technique, simplifying assumptions are not required as is the case with methods that directly solve the Radiative Transport Equation.

There are drawbacks to the Monte Carlo method. As a statistically based method its accuracy is a function of the number of photon bundles tracked - decreasing the variance of a Monte Carlo solution requires one

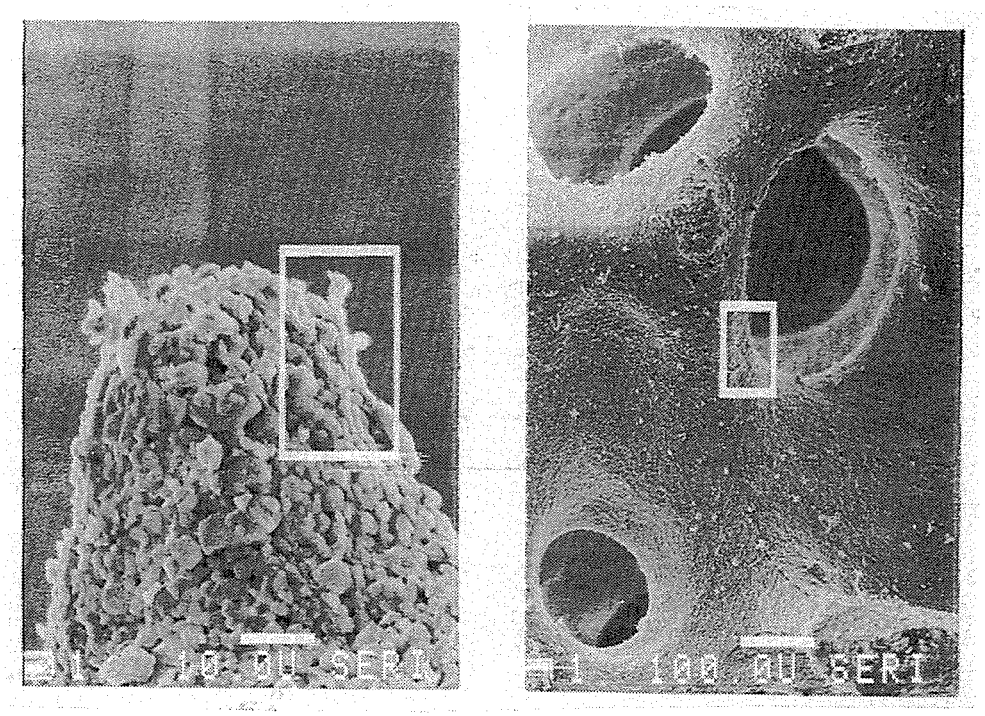

Figure 2: SEM Photographs of 65 pore-per-inch Alumina Frit. Figure 2a: Alumina particles, 1 μm; Figure 2b: Pores bound by diffusely reflecting surfaces, 10-100 μm.

to track more bundles. It is, therefore, desirable to track as many bundles as possible when performing a Monte Carlo simulation. If there are computer-time constraints, it may be necessary to accept a larger uncertainty in the results. Also, the method can become computer intensive when simulating the radiative transport behavior of a material with a very high scattering albedo or a large optical thickness because these conditions create longer bundle lifetimes. On the other hand, with the advent of very powerful personal computers, computer time is no longer cost prohibitive.

The Monte Carlo simulation used for this study requires as input values of radiative transport properties for the material sample being modeled, i.e. the phase function, scatter albedo, and extinction coefficient, as well as the number of photon bundles to be tracked and the sample thickness and diameter. The code output is the number of photon bundles leaving the sample as a function of angle θ (see Figure 1). The photon bundle distribution is then normalized by the value at 140° and converted to a flux for direct comparison with the experimental data.

Because the procedure for determining the correct property values to use as code input is iterative, it is important to have good initial approximations to these values. As discussed previously, the interior surfaces of the alumina foam consist of randomly oriented spherical voids. The surface of these spherical voids are diffuse reflectors due to the random orientation of the alumina particles that make up the surface. This suggests that a photon bundle scattering from a surface in the interior of the foam has an equal probability of being scattered in any direction. Therefore, we used an isotropic phase function in our calculations.

The initial estimates of the scatter albedo values for the alumina were in the 0.95 to 1.0 range. This range was based on work by Makino and Kunitomo (1984) which showed that absorption of visible light in alumina crystals is three to four orders of magnitude lower than scattering. Thus, 0.95 was viewed as a lower limit of the albedo value for the reticulated alumina.

Initial values of the extinction coefficients were derived from a simplified geometric model that models the alumina as a network of packed spherical voids in an alumina structure. The geometric model uses the frit's manufacturer specifications (Hi-Tech Ceramics, 1991) for the alumina void fraction and average pore diameter to calculate the average diameter of the spherical voids. This void diameter was assumed to scale proportionately to the average path length of a given photon bundle. (The results of this study showed this proportionality factor to be 0.5.) The path length was then inverted to give the extinction coefficient. The values calculated with the geometric model are in Table 2.

Table 2: Geometrically Predicted Extinction Coefficients for the 10, 20, 30, 65 ppi Alumina

Pore Size ppi	Extinction Coef. m^{-1}
10	698
20	1101
30	1396
65	3023

Although the reticulated alumina has a definite surface-and-pore structure, it was modeled as a homogeneous material. This assumption is reasonable as long as the frit is large enough so that the typical photon path crosses many voids, i.e., travels many path lengths.

Each time a simulation was done with the Monte Carlo code, the normalized output was compared with the normalized experimental results. This comparison was quantified by determining the root-mean-

square difference between the forward-scattered portion of the two distributions. Once the Monte Carlo scattering profile that provided the best agreement with the experimental was found, i.e, the minimum difference, its radiative transport properties were assigned to that alumina sample.

In order for the method to be considered valid the scatter albedo and extinction coefficient must be independent of sample thickness, i.e. $\omega \neq \omega(L)$ and $\beta \neq \beta(L)$. This is because the extinction coefficient and scatter albedo are material properties, which should not be functions of sample geometry. Therefore, scattering experiments and subsequent analysis were performed on three thicknesses of the 20 pore-per-inch samples. These runs demonstrated that the properties were independent of sample geometry. The results also determined the values of the albedo for reticulated alumina and the extinction coefficient for the 20 ppi samples. In addition, we used these results to give the constant of proportionality in the geometric model for path length. Scattering experiments were then performed on three additional porosities to determine the dependence of the extinction coefficient on porosity.

UNCERTAINTY

The following were identified as potential sources of bias error in the experimental data: detector linearity, beam and/or sample misalignment, and amplifier and/or laser drift. Angular adjustment of the nephelometer arm and equipment fluctuations were identified as potential sources of precision error in the experimental data. We quantified the error associated with each potential source and then calculated the effect those errors would have on the difference values (see Table 3) that we used as the basis for property value selection. The resulting uncertainty in the root-mean-square differences in Table 3 are +0.0128 and -0.00975.

RESULTS AND DISCUSSION

As mentioned in the previous section, we performed scattering measurements on three different thicknesses (2.54 cm, 3.81 cm, and 6.35 cm) of the 20 ppi samples. If one appropriate extinction/albedo combination could be found for all three sample thicknesses, the predicted extinction coefficient and scatter albedo could be considered independent of sample geometry. For each 20 ppi sample thickness, the Monte Carlo code was used to test the appropriateness of a wide range of extinction coefficients, from β=400 m^{-1} to β=1600 m^{-1}, within the albedo range 0.95 to 1.0. The root-mean-square difference between the experimental and analytical forward-scattering results (δ_{EA}) was calculated for each extinction-coefficient/albedo combination tested on the 20 ppi samples. Table 3 contains the sums of the normalized δ_{EA} values for the 2.54 cm, 3.81 cm, and 6.35 cm, 20 ppi samples for the fifteen different extinction/albedo combinations.

The extinction-coefficient range and albedo combination that provided an overall minimum difference, δ_{EA}, between the Monte Carlo and experimental scattering profiles for the three thicknesses tested was β=1100 m^{-1} to 1200 m^{-1} and ω=0.999, respectively. Ranges of extinction coefficient results are given because the Monte Carlo calculations were only performed for extinction coefficients at intervals of 100 m^{-1}. Without a finer resolution of extinction coefficient calculations, it is not possible to say exactly where the best agreement between analytical and experimental lies. Given the calculated experimental uncertainties and the uncertainties that would be inherent in any modeling applications of the reticulated alumina, it is doubtful that a finer resolution of extinction coefficient would provide more useful information.

The agreement between the Monte Carlo calculation for β=1100 m^{-1} and ω=0.999 for all three sample thickness is shown in Figures 3 through 5. The Monte Carlo results exhibit a certain amount of random variation due to the statistical nature of the Monte Carlo method. This variation decreases as larger numbers of photon bundles are run.

For the 20 ppi samples, the plots show that the scattering profile associated with the β=1100 m^{-1} and ω=0.999 extinction and albedo combination provides a very good fit for the 3.81 cm sample (Figure 4) and fairly good fits for the 2.54 cm and 6.35 cm samples (Figures 3 and 5, respectively). Two property combination provided slightly better predictions than β=1100 m^{-1}, ω=0.999 for the 2.54 cm sample, β=600 $^{-1}$, ω=0.99 and β=1100 m^{-1}, ω=1.0. This is perhaps an indication that the homogeneous modeling assumptions break down for such a low porosity and thickness combination, i.e. the 2.54 cm sample has too few pores. Also, an extinction coefficient of β=1200 m^{-1}, which is still within the designated extinction coefficient range, provided the best prediction for the 6.35 cm sample.

Different sample porosities were then tested to examine the effect of sample porosity on the extinction coefficient. Additional scattering measurements were performed on 5.08 cm/10 ppi, 2.54 cm/30 ppi, and 2.54 m/65 ppi alumina samples. Because the results of the 20 ppi samples lead to the conclusion that the albedo for the reticulated alumina is 0.999, this was the only scatter albedo considered for these additional samples. The results are β=900 m^{-1} to 1100 m^{-1} for the 10 ppi sample, β=1100 m^{-1} to 1300 m^{-1} for the 30 ppi sample, and β=2400 m^{-1} to 2600 m^{-1} for the 65 ppi sample.

Table 3: Root-Mean-Square Difference (δ_{EA}) between Monte Carlo Predictions and Experimental Scattering Results for the 2.54 cm, 3.81 cm and 6.35 cm 20 ppi Frits

Albedo	Extinction Coefficients					
	400 m^{1}	600 m^{1}	1100 m^{1}	1200 m^{1}	1500 m^{-1}	1600 m^{1}
0.95	0.392					
0.99	0.963	0.204				
0.995	1.65	0.286				
0.999	2.28	1.19	0.176	0.220	0.429	
1.0	3.0	1.67	0.571		0.371	0.348

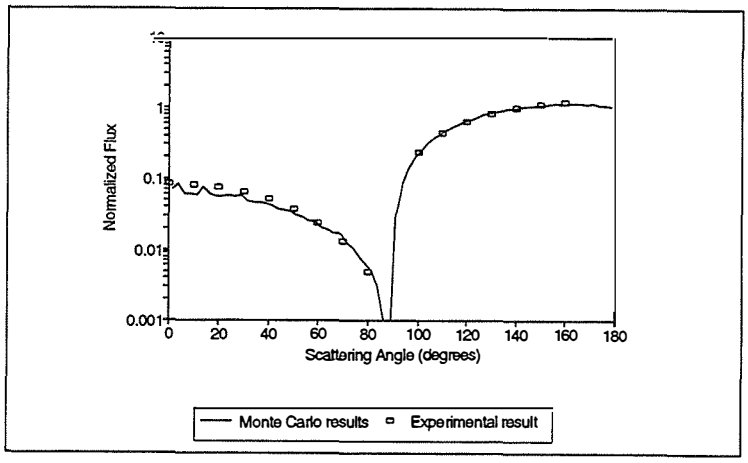

Figure 3: Scattering Results of 2.54 cm, 20 ppi Frit;
β=1100 m^{-1},ω=0.999

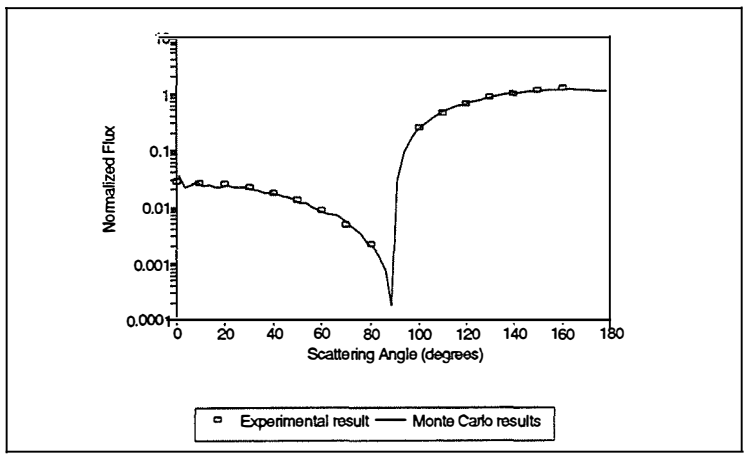

Figure 4: Scattering Results of 3.81 cm, 20 ppi Frit;
β=1100 m^{-1},ω=0.999

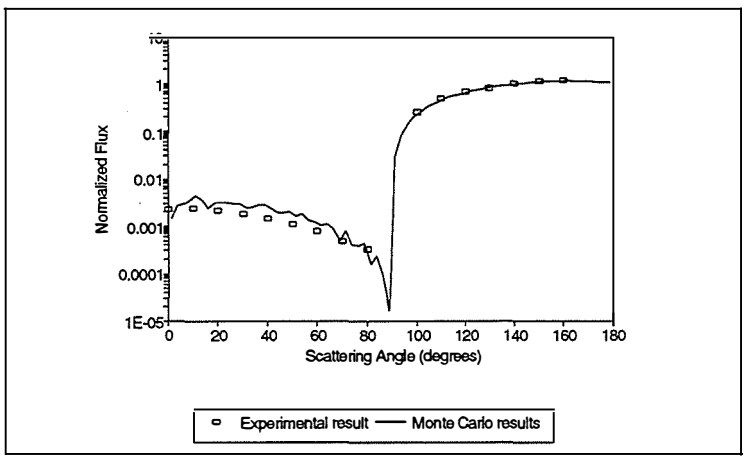

Figure 5: Scattering Results of 6.35 cm, 20 ppi Frit;
β=1100 m^{-1},ω=0.999

Plots of the Monte Carlo predictions and experimental scattering results for the 10, 30, and 65 pore-per-inch samples are shown in Figures 6 through 8. Table 4 provides a summary of the extinction coefficients, as determined by the experimental and Monte Carlo results, for the different alumina porosities.

Figure 9 provides a comparison of the determined extinction coefficients (Table 4) and the geometric extinction-coefficient predictions (Table 2). Figure 9 shows fairly good agreement between the extinction coefficients that were determined with the geometric model and those that were determined by the inverse method. Recall that results of the inverse method at 20 ppi were used to "calibrate" the geometric model. However, the 30 and 65 ppi predictions are close to their determined ranges; they have 7% and 17% differences, respectively. A 22% difference exists between the two 10 ppi extinction values; it is possible that a homogeneous model is not appropriate for a sample only 20 pores thick.

CONCLUSIONS AND RECOMMENDATIONS

Based on the results in the previous section, the following conclusions can be drawn:

- One extinction-coefficient/scatter-albedo combination was found that provided an overall minimum normalized δ_{EA} for the three different thicknesses of 20 ppi samples. Results from the 2.54 cm/20 ppi sample indicated that the homogeneous assumption may not be justified for samples with a thickness of 20 pores or less.

- The scatter albedo for the reticulated alumina at 488 nm was determined to be 0.999. This is consistent with reports showing that alumina has a very low absorption coefficient in the visible wavelengths.

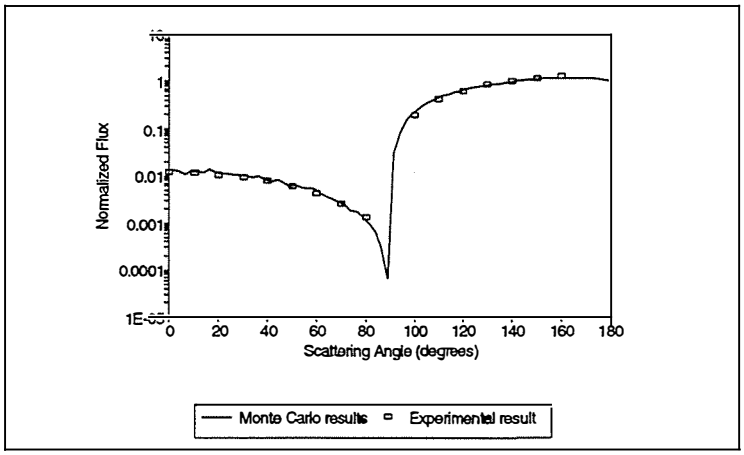

Figure 6: Scattering Results of 5.08 cm, 10 ppi Frit; β=1000 m^{-1},ω=0.999

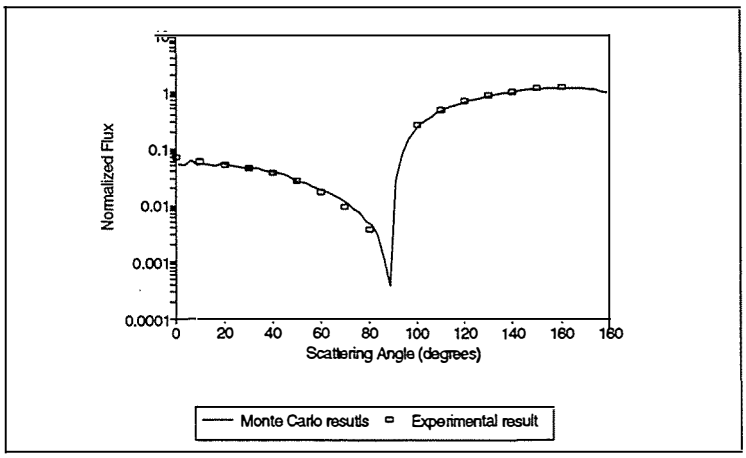

Figure 7: Scattering Results of 2.54 cm, 30 ppi Frit; β=1200 m^{-1},ω=0.999

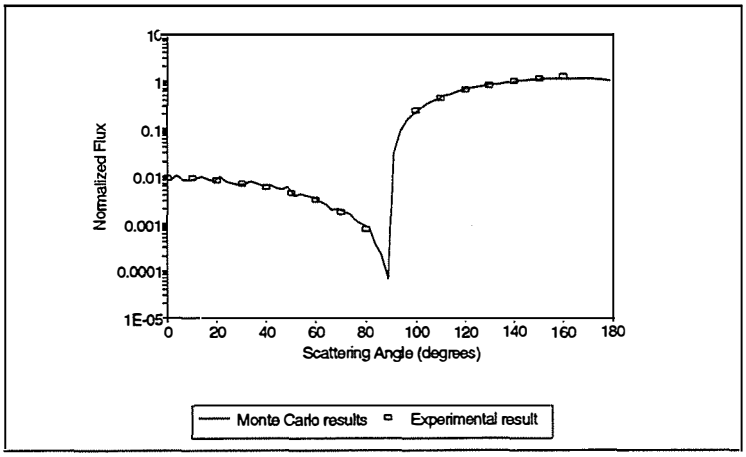

Figure 8: Scattering Results of 1.27 cm, 65 ppi Frit; β=2500 m^{-1},ω=0.999

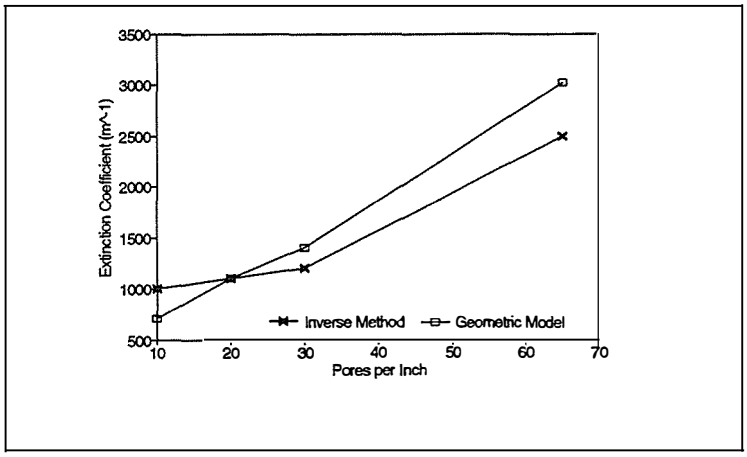

Figure 9: Extinction Coefficient as Function of Alumina Porosity

Table 4: Summary of Extinction Coefficients for Four Different Alumina Foam Porosities

Pores per Inch	10	20	30	65
Extinction, m^{-1}	900-1100	1100-1200	1100-1300	2400-2600

- The extinction coefficient increases with the number of pores per linear inch. The increasing trend is consistent with the physical interpretation of the problem that was modeled with the Monte Carlo code. The extinction-coefficient values determined with the experimental and analytical results are summarized in Table 4.

- The agreement between the experimental scattering profiles indicates that the isotropic phase function assumption was valid.

- The geometric method appears to be a simple and reasonably accurate means for estimating the extinction coefficient for reticulated foams.

The following recommendations are made:

- The effect of wavelength on the radiative characteristics of the reticulated alumina should be investigated. Because of the potential solar applications of this material, characterizing its radiative properties in the ultraviolet and infrared wavelengths is of particular interest.

- More data should be gathered to determine the minimum thickness of a sample, in number of pores, for which the homogeneous model is applicable.

- The possibility of modeling the reticulated alumina as a inhomogeneous material should be investigated.

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