Tailoring Artificial Dielectric Materials at Terahertz Frequencies

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Abstract

The Submillimeter Technology Laboratory(STL) at the University of Massachusetts Lowell has developed a methodology of tailoring the complex refractive index for artificial dielectrics at terahertz frequencies. A wide range of precisely controlled optical properties have been achieved for materials such as vinyl acetate, silicone, polyethylene and epoxy resin when combined with powdered loading agents such as boron nitride, silicon, graphite, iron oxide and stainless steel flake. Using this technology, STL has successfully fabricated both narrow-band and wide-band anechoic structures. The method of characterizing materials for the purpose of tailoring their dielectric properties at terahertz frequencies is presented along with several demonstrated applications.

2. Introduction

Because of the increase in the number of optical and quasi-optical measurement systems operating at terahertz frequencies, new materials providing alternative solutions to the design of system components are required. In response to this, STL's recent activities include an aggressive program to evaluate a wide range of materials for terahertz frequency applications[1-3]. Using in-house expertise in the technique of tailoring artificial dielectrics and the polarimetric characterization of materials, a variety of composite structures have been created as far-infrared radiation absorbing material (FIRAM)[4,5].
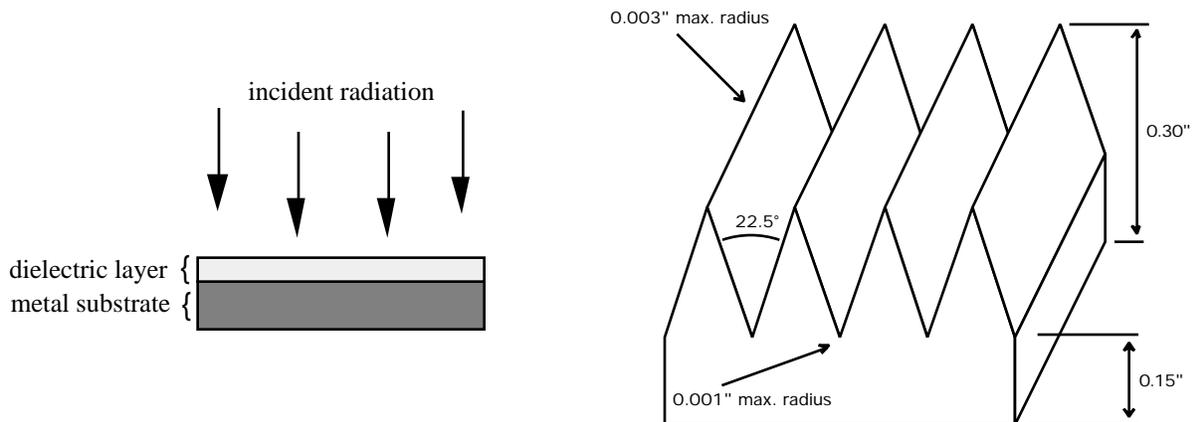

Figure 1.   The FIRAM depicted on the right is a resonant absorption structure which consists of a quarter-wave thick artificial dielectric surface on a metal plate. Shown on the left are design parameters for the wedged-type anechoic implemented by STL[6].

One type of FIRAM, known at microwave frequencies as the Dällenbach layer, consists of a metal substrate coated with an evenly thick homogeneous artificial dielectric material[7]. The second type relies on a wedge-type surface geometry fabricated from a lossy silicone-based material to achieve performance as a submillimeter-wave anechoic. Examples of both FIRAM structures are shown in Figure 1. In each case a –25dB reflectivity reduction was measured at normal incidence, however when appropriately oriented within quasi-optical measurement systems these materials reduce reflections due to unwanted stray radiation by more than –50dB. Considerations such as manufacturablility and cost were addressed in choosing the methodology to implement the anechoic structures.

3. Design and Fabrication of the Dällenbach FIRAM

Realization of the Dällenbach layer as a FIRAM starts with development of a particular class of submillimeter wavelength artificial dielectric, generically referred to as metallic paints. The paint, manufactured by Stainless Steel Coatings, Inc. of Littleton, Mass, consists of resins such as vinyl acetate, silicone or polyurethane uniformly loaded with stainless steel flakes. Since the metal flakes are dimensionally small compared to the wavelength, the paint exhibits the optical properties of a homogeneous media.
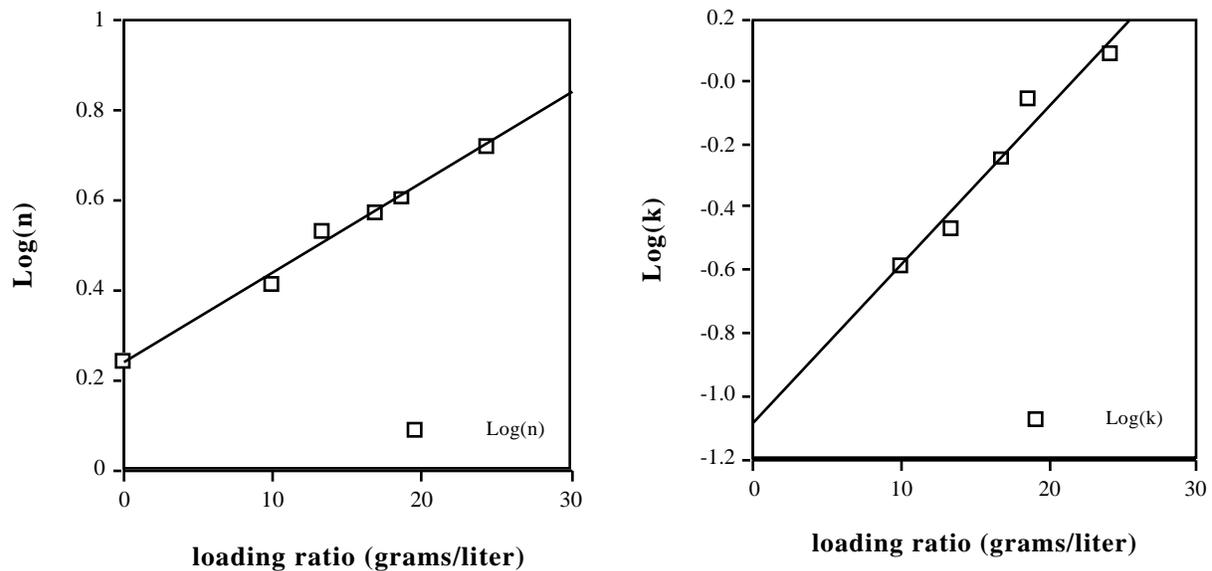

Figure 2. The complex refractive index, n – i k, for the tailored artificial dielectric as a function of the metal flake loading ratio, .

Fourier transform spectroscopy (FTS) transmission and reflection measurements were performed on a series of vinyl acetate based paint samples, each with a different concentration of stainless steel flake. Using a CO_2 optically pumped submillimeter-wave laser operating at 0.584 THz, Brewster's angle was also measured. The complex refractive index, n – i k, of each sample was ascertained by fitting the FTS and laser data to the Fresnel equations. As shown in Figure 2, the material's refractive index as a function of the metal flake loading ratio, , exhibits a power law

relationship. An algorithm was developed to describe the behavior analytically and is expressed as:

$$n \approx 1.73 \times 10^{(0.0201 \, \rho)} \quad \text{and} \quad k \approx 0.082 \times 10^{(0.0504 \, \rho)} \tag{1}$$

for a loading ratio in the regime of $0 \le \rho \le 30$ grams of stainless steel flake per liter of vinyl acetate binder.

This technique of characterizing and tailoring the dielectric constant of metal loaded paints at terahertz frequencies had been previously demonstrated at STL[8]. When tied to theoretical modeling of the paint's reflectivity as a function of thickness, dielectric constant and frequency using the Fresnel equations, the dielectric properties of metallic paints are ideal for providing an anechoic layer on metal surfaces. As shown in Figure 1, these paints provide the vehicle for which phase and amplitude matching of the incident electric field can occur. The paint's complex refractive index model, defined by equations 1, allows calculation of the FIRAM's optical behavior using the Fresnel equations[9]. With a reflectivity of ≈ 1 for aluminum[10], the resonant structure's theoretical reflectivity was approximated by:

$$R \approx \left| \frac{r + e^{-2i\delta}}{1 + r \, e^{-2i\delta}} \right|^2 \tag{2}$$

$$\text{where} \qquad r = \frac{N - 1}{N + 1} \qquad \text{and} \qquad \delta = (2 \pi N t) / \lambda$$

are the paint's front surface reflectivity and phase thickness, respectively.

Upon evaluating equation 2 one concludes that a dielectric layer with phase thickness equal to a quarter wavelength should cause destructive interference at the material's front surface. Furthermore, if the amplitude of the electric field reflected from the paint's front surface equals that of the back surface which suffers absorption, all of the incident electric field is reflected back into the material and complete resonant absorption, i.e. the FIRAM, is established.

Using the theory described, calculations were performed to predict fabrication parameters for a range of FIRAM samples using the paint's experimentally determined dielectric properties. The far-infrared reflectivity of these samples were measured over a frequency regime of 300 GHz to 3 THz using FTS reflectivity measurements. Due to uncertainties in the refractive index model (Equation 1), the predicted fabrication parameters were only adequate for evaluating the FIRAM's general reflectivity behavior. Therefore several samples of metallic paint were prepared and tested with loading ratios ranging from 15 to 25 grams of stainless steel flake per liter of vinyl acetate binder. Also each paint sample was applied to several aluminum substrates in thicknesses which varied from 0.0005" to 0.005".

The frequency at which the reflectivity null occurred for each sample was identified and the FTS measurement results were compared to theoretical predictions. Careful analysis of the structure's multiple reflection theory at the frequency of interest (0.584 THz), allowed modification of the paint's refractive index model to achieve tighter characterization tolerances. Utilizing the modified model, the artificial dielectric layer was specifically matched in metal loading and thickness to create a Dällenbach layer which produced an RCS reduction of −27dB at

0.584 THz. See Figure 3

    Reflectivity data on additional samples, shown in Figure 4, indicate the flexibility of fabricating FIRAMs for use at other frequencies. With a reflectivity null occurring at 0.584 THz for a 2.5 mil thick layer as compared to the one well above 3 THz for a 0.4 mil thick layer, one determines that the loading ratio of an artificial dielectric material could be designed and the Dällenbach FIRAM optimized at any terahertz frequency.
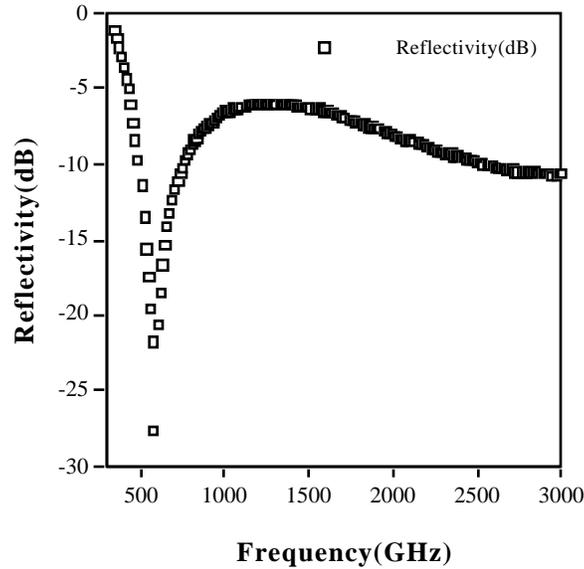

Figure 3.   The reflectivity as a function of frequency for the vinyl acetate specifically matched in metal loading and thickness to provide an RCS reduction of –27dB at 0.584 THz.
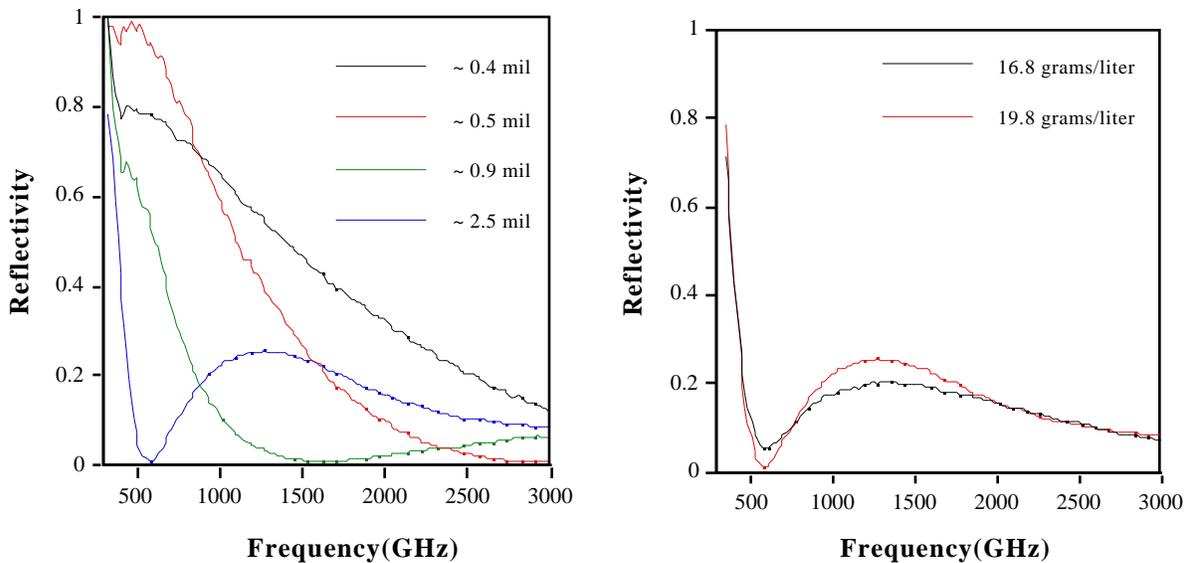

Figure 4.   With a loading ratio of   = 19.8 grams/liter, the reflectivity of four Dällenbach layers of thickness varying from 0.4 mils to 2.5 mils is shown on the left. Optimization of the material's loading ratio is depicted on the right for two 2.5 mil thick structures.

Application of the metal loaded vinyl acetate as a Dällenbach was performed using a hand-held spray gun. While this method was suitable for prototype studies, the thickness tolerance and uniformity over large-area samples could not be achieved reliably. Techniques currently under consideration to achieve production thin-film materials are: robotic spraying, extrusion blow molding, compression molding, casting and spin coating. For this purpose, research on alternative materials such as low density polyethylene (LDPE) and epoxy resins is being conducted.

Performing FTS measurements on a series of both epoxy resin based samples and LDPE based samples, each with a different concentration of stainless steel flake, produced behavior similar to the properties described for metal loaded vinyl acetate. However, increased flexibility in tailoring the refractive index was achieved for these materials by introducing powdered silicon (a low loss, high n material) as a second loading agent. By adjusting the powdered silicon and stainless steel flake concentration levels, the real and imaginary component of a material's complex refractive index can be controlled independently. As shown in Figure 5, STL has already fabricated Dällenbach layers using the spin coating technology. Since large area, uniformly thick dielectric layers are achievable by this method, efforts to exploit this technology for applications such as AR coatings and optical band-pass filters are under consideration.
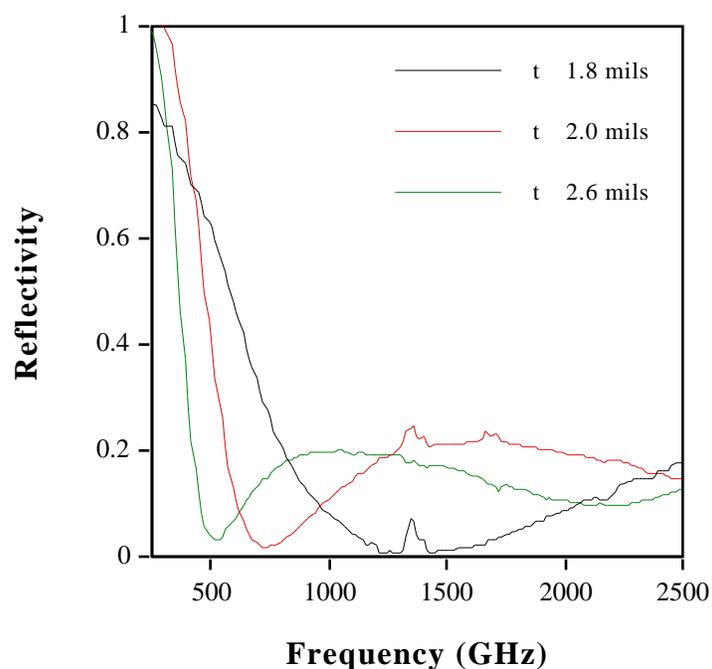

Figure 5. With an 8% loading ratio for stainless steel loaded epoxy resin, the reflectivity of three Dällenbach layers of thicknesses varying from 1.8 mils to 2.6 mils is shown.*

*The reflectivity peak observed at 1.4 THz is an artifact of the FTS measurement system.

4. Design and Manufacture of the Silicone-based Anechoic Structure

Silicone-based anechoics using wedge-type surface geometries have also been designed by STL researchers for use at terahertz frequencies. These materials provide more than –60dB of reduction in reflectivity for large-scale quasi-optical measurement systems. The geometry of the grooved surface shown in Figure 1 is manufactured in 2´ x 2´ sheets to precise tolerances through a pressure injection molding process.

As demonstrated by Janz and co-workers[11] in the millimeter wavelength regime, wedge and pyramidal–structured surface geometries improve a material's absorption properties by increasing the number of surfaces incident radiation must encounter before backscattering to the receiver occurs. Measurements performed by these and other researchers[12] have shown that the reduction in reflectivity achieved may be expressed by:

$$R_s \approx R_f^{\left(180°/ \theta_g\right)} \tag{3}$$

where R_f is the material's front surface reflectivity and θ_g the structure's groove angle. Since this type of FIRAM is generally fabricated from homogeneous lossy dielectric materials which exhibit front surface reflectivities (R_f) of less than 10%, anechoic structures can be designed to provide more than –80dB of reflectivity reduction for a groove angle of 22.5° .

Using a silicone elastomer and electrically insulating siliceous filler, Prewer and Milner of Thorn EMI Technology Inc. fabricated the first samples of terahertz frequency anechoic in the form of pyramidal surface structured tiles[13]. Measurements performed during July of 1989 at STL further documented their success when specular and diffuse reflectivity levels of better than –40dB were observed at 0.584 THz for almost all incident directions. However wedge and pyramidal structured anechoic materials suitable for large-scale use at terahertz frequencies were not commercially available at that time.

Therefore STL researchers initiated a project to design and fabricate silicone-based wedge-structure anechoic material for use with its submillimeter-wave measurement systems. A method of estimating refractive indices was used to characterize a variety of materials in search for a lossy dielectric exhibiting low front surface reflectivity. Using cost-efficient molding techniques, prospective dielectric materials such as the widely available plastics and elastomers promised to provide good anechoic properties if constructed with modified surface geometries.

Chosen as most suitable due to its mechanical and optical properties was iron-oxide-loaded silicone. The front surface reflectivity for a flat 0.25" thick sample of the material was measured at 1.56 THz and found to be $R_f \approx 5.57\%$. The transmissivity was also measured and found to be T $\approx$ 1.8% for a t = 1.9mm thick sample of the same material. The Fresnel equations for the multiple reflection theory transmissivity:

$$T = \left| \frac{\left(1 - r_f^2\right) e^{-2\pi Nt/\lambda}}{1 - r_f^2 e^{-4\pi Nt/\lambda}} \right|^2 \tag{4}$$

and front surface reflectivity:

$$R_f = |r_f|^2 \qquad \text{where } r_f = (N-1)/(N+1)$$

were used to estimate a complex refractive index of N   1.62 – 0.03 i, for the iron oxide loaded silicone. With more than a –100dB reflectivity reduction expected from a structure such as the design depicted in Figure 1, STL created a 3.5" x 24" prototype aluminum mold structure for conceptual evaluation. A reduction in reflectivity of better than –80dB was realized for all incident angles except normal incidence and those angles satisfying the grating equation.   At those orientations values of better than –40dB were observed.

Therefore a contract was established to manufacture 24" x 24" iron-oxide-loaded solid silicone sheets with the wedge-type surface geometry. The final anechoic structure was evaluated at 0.584 THz using a CO_2 optically pumped submillimeter laser. The reflectivity was measured by illuminating 6" x 24" samples, mounted on a rotary stage, with a 2" FWHM diameter beam. Measured values were ratioed to the measured return for a flat front surface mirror at normal incidence. Figure 6 depicts the measurement configurations used for evaluating a sample's reflectivity.
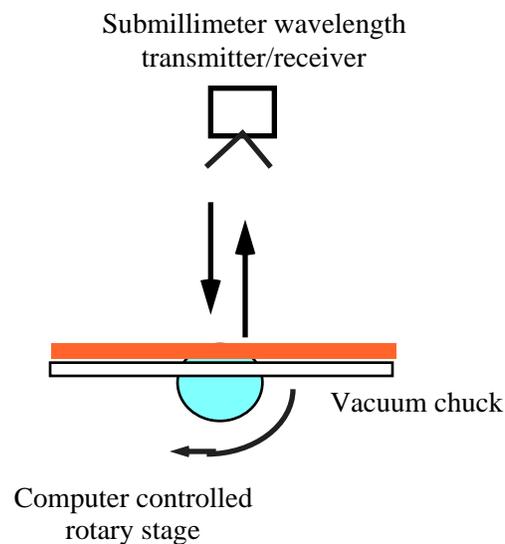

Submillimeter wavelength
transmitter/receiver

Vacuum chuck

Computer controlled
rotary stage

Figure 6.   Shown is the configuration of the submillimeter-wave measurement system used to evaluate a material's reflectivity monostatically.

As observed in Figure 7, results for STL's silicone-based FIRAM indicate that performance is angular dependent. While the reflectivity peaks at normal incidence and those angles satisfying the grating equation have a higher value than was observed for the prototype structure, an overall performance of better than –60 dB for production grade material was considered a major success. Differences between the prototype and production grade FIRAM are strictly related to the machininglimitations for each mold structure. While a peak radius of better than 0.003" was achieved during the prototype study for the milled aluminum mold, only a radius of   0.006" could be achieved for the steel precision ground production mold.

As a comparison to the evaluation of STL's FIRAM, reflectivity measurements were performed on other anechoic materials often used at terahertz frequencies. Figure 8 represents the reflectivity measurements for Thorn EMI's silicone-based RAM 100 and RAM 403. Shown in Figure 9 is reflectivity for Masland Étoile pure wool pile carpet and Emerson & Cuming's Eccosorb™ AN-73.
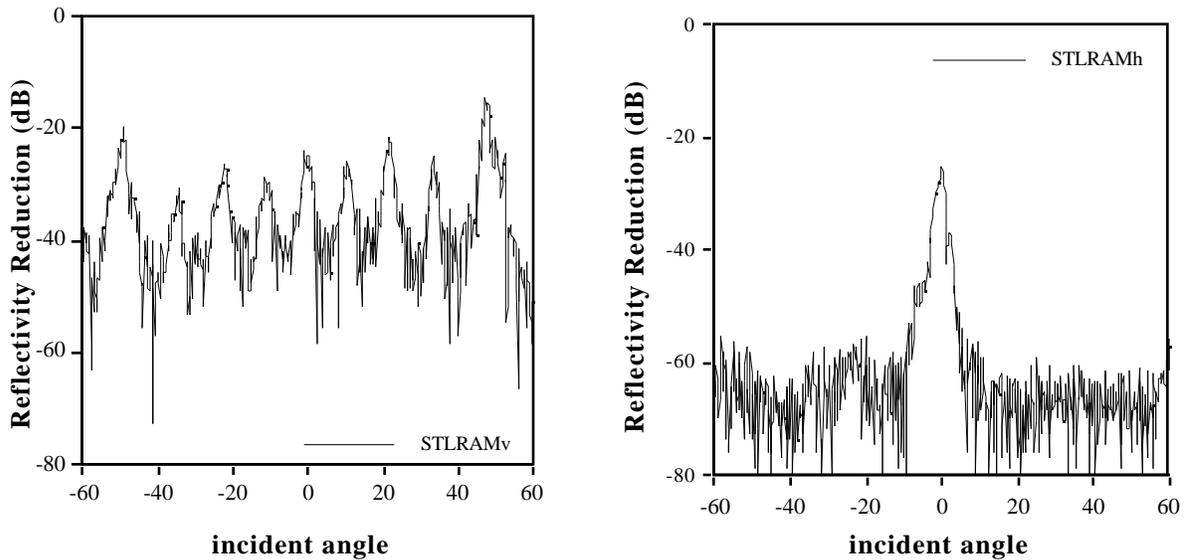

Figure 7   The reflectivity reduction for STL's FIRAM. Shown on the left, and right, are measurements performed with the structure's grooves perpendicular, and parallel, to the plane of incidence.
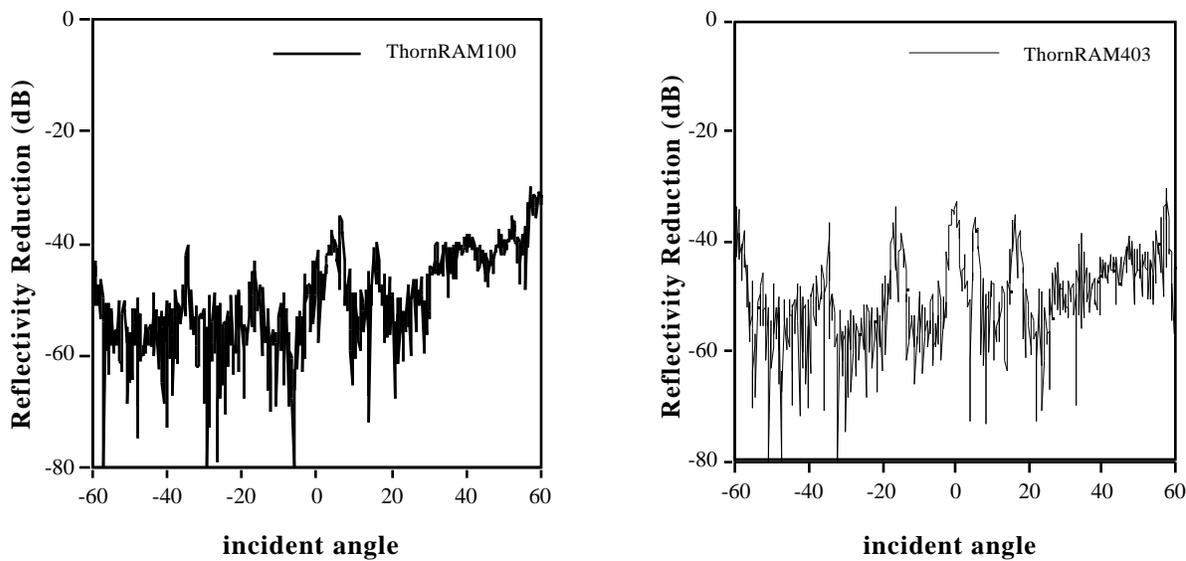

Figure 8   The diffuse reflectivity relative to a flat plate at normal incidence for Thorn EMI's RAM 100 and RAM 403.
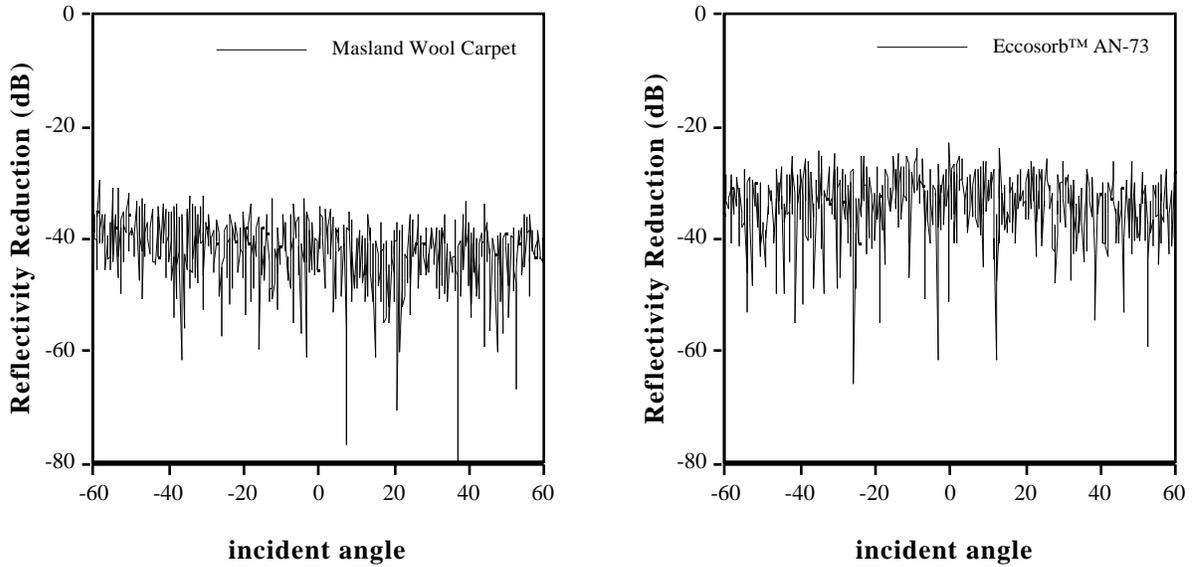
Figure 9. The diffuse reflectivity relative to a flat plate at normal incidence for Masland Étoile pure wool pile carpet and Eccosorb™ AN-73.

5. CONCLUSIONS

We have demonstrated that artificial dielectric layers can be optimized to operate as resonant absorbers for any frequency over the regime of 0.3 THz to 3 THz. We also have determined that these anechoic coatings can be designed to provide more than –25dB of reduction in reflectivity. Since the materials are relatively inexpensive and suited to commercial manufacturing methods, the technology is practical for a variety of applications at terahertz radar frequencies.

Also critical to implementation of terahertz radar systems are low cost, large area FIRAM which may be used to suppress unwanted stray radiation. Measurements performed on the silicone-based FIRAMs demonstrated that, if these materials are properly oriented, a reduction in reflectivity of better than –60dB is feasible even for large-scale operations.

6. REFERENCES

1. R.H. Giles, A.J. Gatesman, and J. Waldman, "A Study of the Far-Infrared Optical Properties of Rexolite™", International Journal of Infrared and Millimeter Waves, <u>11</u>, 1299 (1990).

2. A.J. Gatesman, R.H. Giles, and J. Waldman: "Submillimeter Optical Properties of Hexagonal Boron Nitride", <u>Proc. of MRS Society</u>, <u>242</u>, 623 (1992).

3. A.J. Gatesman, R.H. Giles, and J. Waldman, "A High Precision Reflectometer for the Study of Optical Properties of Materials in the Submillimeter", <u>Proc. of the 17th Int. Conf. on Infrared and Millimeter Waves</u>, Los Angeles, CA, Dec. 1992.

4. R.H. Giles, A.J. Gatesman, A.P. Ferdinand, and J. Waldman, University of Lowell: "Design and Fabrication of Narrow Band Radar Absorbing Materials at Terahertz Frequencies", IEEE Proceedings from the 15th International Conference on Infrared and Millimeter Waves, Orlando, FL, Dec. 1990

5. R.H. Giles, T.M. Horgan, and J. Waldman, "Silicon-Based Anechoics at Terahertz Frequencies", <u>Proc. of the 17th Int. Conf. on Infrared and Millimeter Wave</u>, Los Angeles, CA, Dec. 1992.

6. R.H. Giles and T.M. Horgan: "Silicone-Based Wedged-Surface Radiation Absorbing Material", U.S. Patent Pending

7. G.T. Ruck, D.E. Barrick, W.D. Stuart and C.K. Kirchbaum, <u>Radar Cross Section Handbook</u>, Volumne 2, Plenum Press 1970, Section 8.3.2.1.1.3

8. R.H. Giles, A.P. Ferdinand, M.J. Coulombe, J. Waldman, ULRF and W. Nixon, W. Reinhold, AFSTC: "Submillimeter Wavelength Modeling of Dielectric Materials in Polarimetric Radar Approaches", <u>Proceedings of the NATO Advanced Research Workshop on Direct and Inverse Methods in Radar Polarimetry</u>, Bad Windsheim, Federal Republic of Germany, September 1988.

9. R.M.A. Azzam and N.M. Bashara, <u>Ellipsometry and Polarized Light</u>, North-Holland 1979, Section 4.3

10. M.A. Ordal, R.J. Bell, R.W. Alexander, L.L.Long and M.R. Querry; "Optical Properties of Fourteen Metals in the infrared and far-infrared: Al, Co, Cu, Au, Fe, Pb, Mo, Ni, Pd, Pt, Ag, Ti, V, and W." <u>Applied Optics</u>, Vol. 24, No. 24, pp 4493-4499, Dec. 1985 Note on reference: *Theoretical evaluation of the frequency dependent complex refractive index for aluminum was possible using the Drude model with parameters suggested by Ordal et al. The reflectivity of Al was determined to have a value of 0.99735 at 0.584 THz.*

11. S. Janz, D.A. Boyd, and R.F. Ellis: "Reflectance Characteristics in the Submillimeter and Millimeter Wavelength Region of a Vacuum Compatible Absorber", <u>International Journal of Infrared and Millimeter Waves</u>, Vol. 8, No. 6, 1987, pp. 627-635

12. B.T. Dewitt: "Analysis and Measurement of Electromagnetic Scattering by Pyramidal and Wedge Absorbers", Ph.D. Dissertation, Ohio State University, 1986

13. B.E. Prewer, B. Milner: "Radiation Absorber and Method of Making It", U.S. Patent # 4,942,402 , July 17, 1990