Towards a Nondestructive Procedure for Characterization of Molding Compounds

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ABSTRACT

The applicability of using ultrasonics for characterizing and fingerprinting molding compounds is investigated in this study. The goal is to compile a database of ultrasonic parameters for several commercial molding compound and investigate if different molding compounds have distinct "signatures". The suitability of using ultrasonic parameters to estimate the void fraction is also investigated using calibration specimens with varying amounts of voids (simulated with 100 μm glass spheres). Results presented demonstrate that different classes of molding compounds can be distinguished from each other from their frequency dependent attenuation curves and acoustic impedance. In general, the biphenyl resins exhibit a lower attenuation, higher velocity and higher impedance than the creosol-novolac resin. Post-mold cure (PMC) does not change the attenuation curves significantly, and the molding compounds retain distinctly different behaviors. The attenuation measured for even the least attenuative molding compound (MP7320) at 30 MHz is significantly higher (~10 dB) than that at 10 MHz. The slope of the frequency dependent scattering loss correlated well with the volume fraction of the simulated voids. Thus, ultrasonics is demonstrated to be an attractive technique to monitor the material parameters of molding compounds nondestructively and over a small area.

INTRODUCTION

From a reliability perspective, it is important to ensure that an encapsulant has been uniformly cured to the right extent and that it has attained the designed properties. It is also necessary to know whether the properties, and hence propensity for failure, varies from part-to-part or lot-to-lot due to non-optimal processing. Hence, there is a need to monitor and measure the mechanical properties of the molding compound, usually after the device has been fabricated. Ultrasonics is a candidate technique that has been used extensively to interrogate the mechanical properties of metals, ceramics and polymers with relative success [1,2].

The characteristics of molding compounds that are candidates for measurement by ultrasonics include a) type of resin system (biphenyl, creosol novolac etc.), b) filler material (type, volume fraction or loading, and size), c) the degree of cure which affects the extent of crosslinking and d) void fraction and size. Various studies have investigated the relationship between ultrasonic and material parameters. For example, The compressional wave velocity was observed to increase by 60% from the uncured state to the completely cured state in a DER 332/ Jeffamine T403 system [3]. In a study of DGEBA (diglycidyl ether of bisphenol A, Epon828) with diaminobutane as curing agent, Matsukawa and Nagai [4] observed that the amount of curing agent used and the degree of polymerization affected the frequency dependent attenuation and velocity. They found that ultrasonic waves of different frequencies suffered different degrees of attenuation, with the higher frequencies showing greater attenuation. In another study of

DGEBA with N-aminoethylpeparzine (AEP) as the curing agent, the frequency dependent attenuation and phase velocity was studied during the curing reaction [5]. The slope of attenuation coefficient as a function of frequency showed a peak during the middle stage of the curing reaction. In addition, the attenuation coefficient was found to vary linearly with frequency at all stages of the curing, and this slope was strongly dependent on the degree of cure. Results of these, and other experiments, suggest that it should be possible to probe the molding compound nondestructively using ultrasound for information about the cure state. However, it was not investigated whether different types of commercial molding compounds can be distinguished from each other by ultrasonic parameters.

Void fraction has also been estimated nondestructively from the frequency dependent scattering loss of ultrasound in specimens [1,2]. In particular, it has been shown that the slope of the frequency dependent scattering loss of ultrasound is related to the volume fraction of porosity with material constants such as the velocity of compressional waves in the material and the scattering parameter of the pores [2]. Thus, scattering phenomena can be used as an indicator of the void fraction or void content in a molding compound.

The overall objective of this study is to be able to quantitatively determine molding compound characteristics such as void content, cure state and resin type *in situ*, preferably over a small area. To this end, the initial task is to generate a database of ultrasonic properties for commercial molding compounds. One of the obstacles to "finger printing" different molding compounds based on the ultrasonic properties is the relatively complicated composition of commercial molding compounds and the lack of detailed information about the precise composition.

As a first step, the existence of differences in the ultrasonic properties of commercial molding compound is investigated in this paper. Ultrasonic velocity and attenuation coefficients are measured for some common, commercial molding compounds to construct a database. The effect of post-mold cure on the attenuation is also discussed with reference to two types of molding compounds. The issue of resin type is addressed by including epoxidized orthocreosol-novolac (OCN) and bisphenol A specimens in the test matrix. Finally, the applicability of using the slope of the frequency dependent attenuation coefficient to estimate the volume fraction of voids is investigated by means of a simple experiment with calibration specimens.

EXPERIMENTAL PROCEDURE

Molding Compound Characterization

Flat coupons having parallel sides to within 50 μm were used for ultrasonic characterization of molding compounds in an unpackaged state. These specimens were immersed in a water bath mounted on a leveling stage that could be tilted in two mutually perpendicular directions. The whole assembly was placed in the C-mode Scanning Acoustic Microscope (C-sam) and the stage adjusted so that the front surface echo was maximized. This ensured that the ultrasonic beam was incident normal to the specimen. The experimental setup is depicted schematically in Figure 1.

The acoustic impedance was measured using the C-sam, where the received echoes are analyzed with respect to a calibrated reference. The spatial resolution of the instrument at 15 MHz is 0.2 mm.

The attenuation coefficient is calculated from the amplitude of the reflections from the front and back surfaces of the flat coupons. The acoustic impedance determined earlier is used in calculating the correction factor for evaluating the attenuation coefficient. If the reflection and transmission losses due to the acoustic impedance mismatch between the coupling fluid (water) and the sample are not

incorporated via the correction factor, the estimated attenuation coefficient would be an overestimate of the actual loss suffered in the sample. This correction factor is calculated to be:

$$C_{RT} = \frac{4 Z_1 Z_2}{\left(Z_1 + Z_2\right)^2}$$

where Z_1 is the impedance of the couplant and Z_2 is the impedance of the specimen. The attenuation spectra are calculated from the Fourier spectra as follows. The front and back echoes are digitized with identical system gain and energy settings, and the frequency dependent attenuation coefficient is calculated by

$$\alpha(f) = \left(-20/(2l)\right) \log_{10}\left[\frac{\left|F_{back\ echo}\right|}{C_{RT}\left|F_{front\ echo}\right|}\right]$$

where l is the thickness of the specimen and the $|F|$ stands for the magnitude of the Fourier spectrum. Since the acoustic impedance was found to be approximately the same for different frequency transducers, the Z value used in calculating C_{RT} was assumed to be independent of frequency in the range studied.

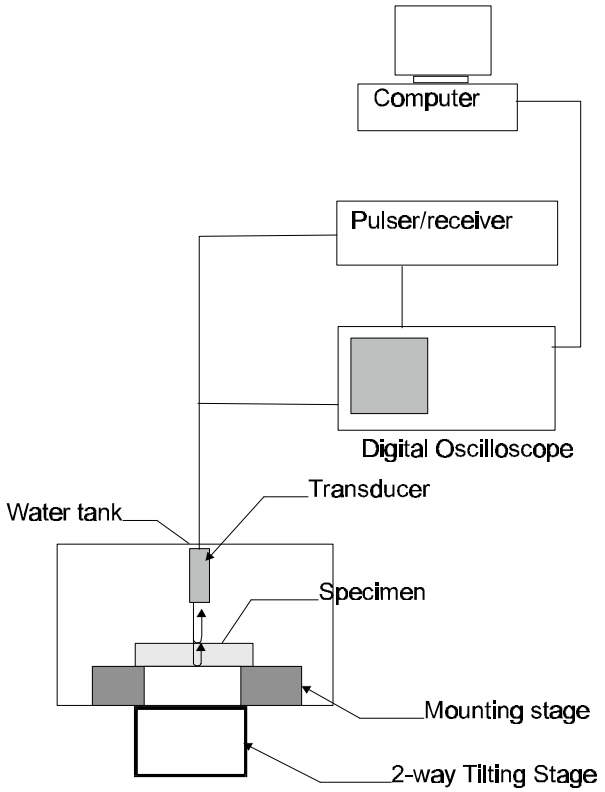

Figure 1. Schematic of experimental setup.

To calculate the velocity of ultrasound in the material, the pulse travel times are measured from the digitized A-scan signal. Since the polarity of the back echo was inverted with respect to the front echo, the measurements were made between corresponding peaks only. Although the sound velocity in the flat coupons could be easily measured using unfocused transducers, a smaller spot size was necessary to make these measurements on the smaller packaged devices (5mm x 5 mm). Using a focused transducer reduces the spot size but may introduce errors in the velocity measurement due to geometrical considerations. To minimize errors due to the use of a focused beam, and at the same time, reduce the

spot size, a mildly focused transducer (F#4) was used. To assess the effect of the focused beam, the velocity in the flat coupons measured with the mildly focused 10 MHz transducer was compared to that measured with an unfocused 10 MHz transducer.

Scattering loss measurements of calibration specimens

To investigate the feasibility of using the slope of the frequency dependent attenuation curve to estimate the volume fraction of filler particles, flat, disk shaped specimens of clear epoxy resin were made with known concentration of 100 μm glass spheres. In these experiments, however, an echo from the back surface of a reference specimen with no glass spheres was used instead of the front surface echo. Thus, the ultrasonic pulse reflected from the backwall of specimens with varying concentrations of glass spheres is normalized on a dB scale with the backwall reflection from an identical specimen with no glass spheres.

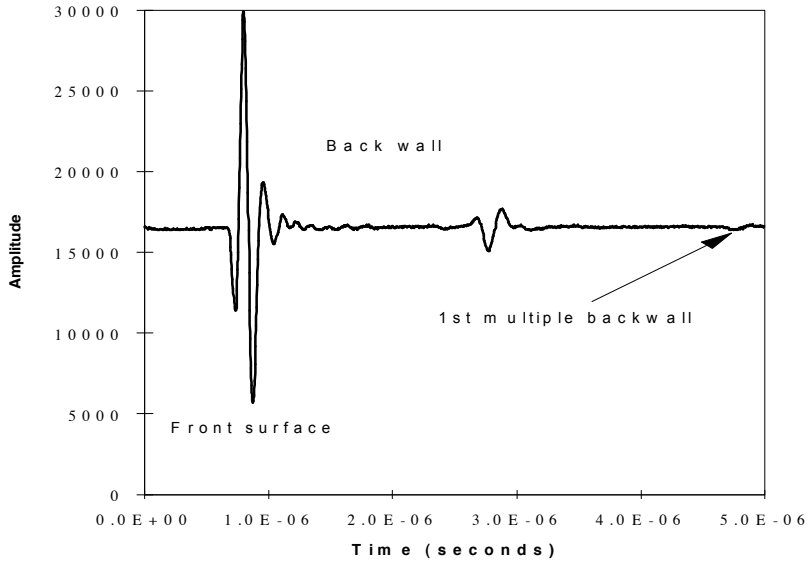

Figure 2. Time domain signals of the front and backwall echoes for the flat coupon 6300HA.

Table 1. Velocity and Impedance measured on the flat coupons

Specimen ID	Resin type	Velocity 5 MHz Unfocused	Velocity 10 MHz Unfocused	Velocity10 MHz F#4 focused	Z@15 MHz
SUMITOMO 6300 w/ PMC	OCN	3.23	3.23	3.23	4.88
SUMITOMO 6300 w/o PMC	OCN	3.39	3.42	3.41	4.97
SUMITOMO 6300HW	OCN	3.30	3.33	3.30	5.22
SUMITOMO 6300HA	OCN	3.26	3.28	3.29	4.77
SUMITOMO 7320CR	BP	3.88	3.89	3.89	6.80
NITTO MP8000CH	OCN	4.07	4.11	4.08	7.50
NITTO MP190ML	OCN	3.48	3.48	3.51	5.82
TOSHIBA KE250MT	OCN	3.41	3.48	3.51	5.99
TOSHIBA KE250MH	OCN	3.37	3.42	3.45	4.76

RESULTS AND DISCUSSION

Velocity and Acoustic Impedance Measurements on Molding Compounds

Measurements on flat coupons: Measurements were made on flat coupons of several molding compounds from three manufacturers to estimate the differences ultrasonically. The compressional wave velocities measured with 5 and 10 MHz unfocused transducers and the 10 MHz mildly focused transducer are presented in Table 1. The results obtained with the two 10 MHz transducers suggests that, for materials having velocities in the 3 to 4 mm/μsec range and the F#4 transducer used, the errors due to the use of a mildly focused beam are negligible. These measurements were made with pulsed broadband transducers, and the higher frequencies in the echo from the back wall were severely attenuated indicating the need for measuring the attenuation as a function of frequency. For illustration, the front and back echoes from specimen 6300HA are examined. Figure 2 shows the time traces of the two echoes and Figure 3 shows the magnitude spectra of the two signals. From a cursory comparison of the signals reflected from the front surface and from the back wall, it is seen that the attenuation is significant, and that there is a polarity reversal due to the interface condition at the back wall. In addition, it is also seen that the higher frequencies (sharper peaks) are almost completely attenuated in the back wall echo. This behavior is easier to observe in the frequency domain data shown in Figure 3. The peak in the spectra (via fast Fourier Transform) of the front wall signal is at around 7 MHz while the backwall signal shows a peak at around 6 MHz and the higher frequencies beyond 8 MHz are completely attenuated. The tendency to absorb higher frequencies more than the lower frequencies results in an apparent "downshifting" of the transducer frequency response.

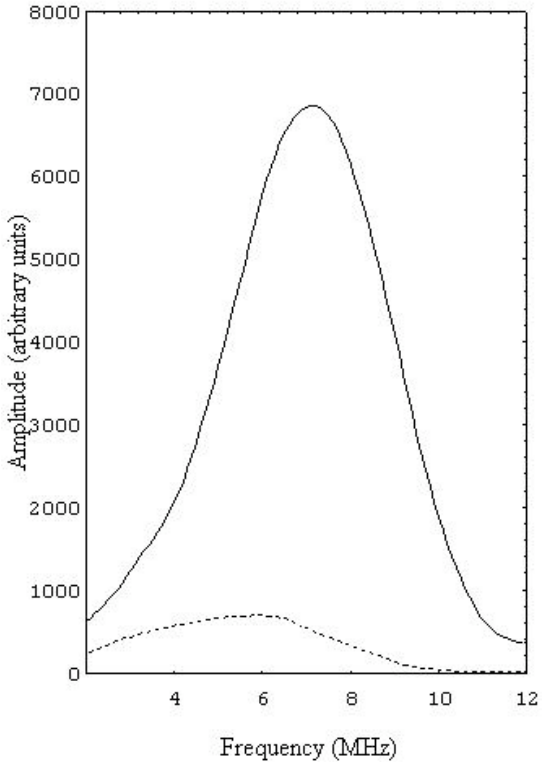

Figure 3. Amplitude spectrum of the front and backwall signal from 6300HA with a 7 MHz transducer.

Table 2. Approximate compositions of some molding compounds

Compound	Filler loading	Low stress additives	Resin type
Sumitomo 7320	high (~ 80%)	low	Biphenyl
Nitto MP8000CH	medium-high	medium	OCN
Nitto MP190ML	medium	high	OCN
Sumitomo 6300HA	low (~ 60%)	high	OCN

The frequency dependent attenuation characteristics of a molding compound have important bearing on their analytical (quantitative) characterization. This is because attenuation is sensitive to microstructural factors such as the distribution of voids and filler, presence of low stress additives etc., and can be used as an indicator. To explore these issues, the frequency dependent attenuation behavior of several molding compounds are investigated. The complete characterization of molding compounds is a nontrivial task further complicated by the "uncertainty" surrounding the exact formulation of these molding compounds. The intent in this paper is to quantitatively estimate the differences in *commercial* molding compounds rather than to measure the microstructural components explicitly. Results for molding compounds from two different manufacturers, Nitto and Sumitomo, are presented below. The polymer systems discussed include biphenyl and OCN resins. The biphenyl resins are characterized by lower values of viscosity, moisture absorption, glass transition temperature (T_g) and shrinkage than the creosol-novolacs. In general, the biphenyl resins are reported to have a lower CTE (both below and above T_g) and higher moduli and strengths than the creosol-novolac system [6].

The attenuation spectra for different compounds are presented in Figure 4. Of these, only the 7320 is a biphenyl resin (see Table 2), while the rest are creosol-novolac based. The biphenyl resin system has the lowest attenuation compared to the others, and this behavior was also observed in other biphenyl based molding compounds such as MP7150. Among OCN resins, the molding compound 6300HA has the highest attenuation and the steepest slope, while MP8000CH has the lowest attenuation and the shallowest slope among all. The absolute attenuation seems to correspond fairly well to the quantity of low stress additives. Also, the OCN resins exhibit an attenuation of around 1.3 dB/mm at 4 MHz but the differences become more dramatic at 8 MHz, where 6300HA has almost twice the attenuation of MP8000CH. This indicates that higher frequency inspection could reveal smaller differences in the composition of the molding compound.

Although inspection of IC packages is usually performed in the 15-30 MHz frequency range, the thickness of the flat specimens precluded making the attenuation measurements with a higher frequency transducer. For example, when a transducer with a peak frequency of about 32 MHz was used, the peak in the spectrum of the backwall echo was found to be only about 10 MHz. This problem, which is inherent in pulsed ultrasonic systems, can be overcome by using a tone burst or continuous wave measurement technique that insonifies the specimen with ultrasound at a single frequency, such as the scanning laser acoustic microscope [8]. To measure the attenuation by this technique, one of the specimens (7320) was thinned down such that sections of 3 different thickness' were produced. The

relative attenuation between these sections was measured using the Scanning Laser Acoustic Microscope using 10 and 30 MHz transducers. Ultrasound was passed through the specimen and the intensity of the transmitted sound averaged over a rectangular area was measured. A calibrated electrical attenuator that could be varied in steps of 1 dB, was used to attenuate the averaged signal to match the intensity transmitted through each different thickness. This technique obviates the need to take into account reflection losses as long as the surface finish between the different sections remains the same. The material's attenuation (at a single frequency) is given by the ratio of the externally imposed attenuation to the thickness difference between two sections. Using this method the attenuation of the biphenyl compound (7320) was calculated to be about 3 dB/mm and 13 dB/mm at 10 and 30 MHz, respectively, and the results are plotted in Figure 5, along with the frequency dependent data determined earlier. The attenuation at 30 MHz for even the least attenuative resin is significantly higher than at 10 MHz, which could be a potential hindrance for inspection at higher frequencies.

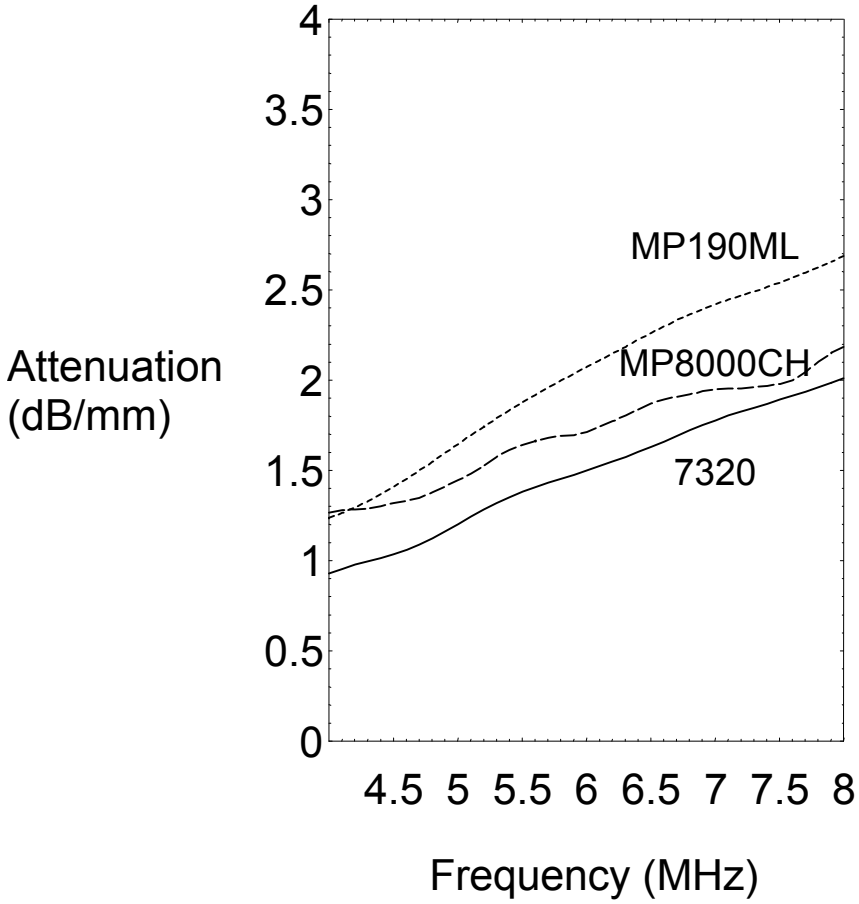

Figure 4. Frequency dependent attenuation characteristics of some commercial molding compounds.

Measurements on packaged devices: The compressional wave velocities measured for the devices are tabulated in Table 3. For these measurements, the front surface echo was used along with an echo from an internal interface at a known depth. To enable the precise placement of the ultrasonic beam, the devices were first imaged with a 10 MHz F#4 transducer in the C-sam. In comparing the velocities, it is seen that the velocity scales with the filler content. The velocity of MP180S is comparable across 3

different packages, while MP190M and MP 195 possess a distinctly higher velocity than the MP180S compound, but are close to each other. The data suggests that molding compounds with similar compositions, but different designations, have similar velocities. To quantify the difference between the different molding compounds in terms of the ingredients would require calibration specimens where only one parameter is varied at a time.

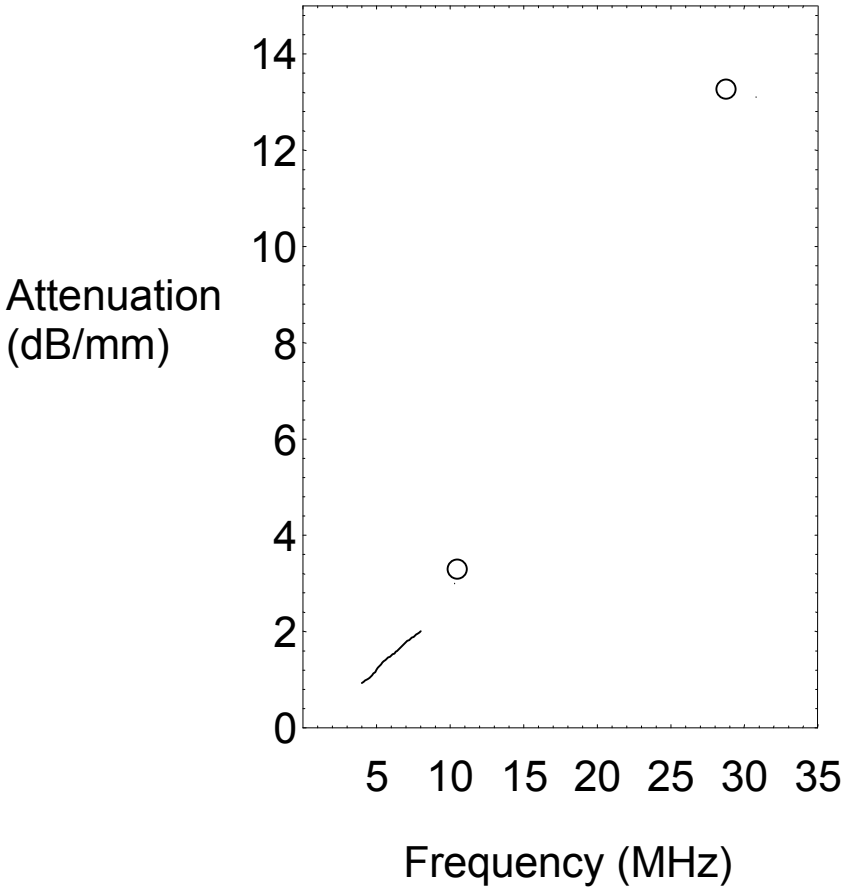

Figure 5. Attenuation coefficient measured at higher frequency (10 and 30 MHz) using the SLAM compared with the frequency dependent attenuation measured for MP7320.

The impedance data are slightly lower than those measured for the flat coupon, which is attributed to the rougher surface finish of the devices. A rougher surface finish will scatter sound away from the transducer and result in lower intensity of sound, causing the measured value of the impedance to be artificially low. Therefore, comparisons of the acoustic impedance are made only for the flat coupons which had a uniform surface finish. In general, the biphenyl resins exhibit a higher acoustic impedance than the OCN resins.

Effect of Post Mold Cure

In this study we examined the frequency dependent attenuation behavior in specimens that were, and those that were not, subject to PMC. It should be remembered that, the molding compounds investigated

in this study, cure very rapidly. The curing reaction is expected to be nearly complete even in the specimens that were not subject to PMC, and the primary purpose of PMC is to relieve thermal stresses and warpage rather than change the cure state dramatically. Therefore, a strong change in the attenuation is not expected in the molding compounds before and after PMC.

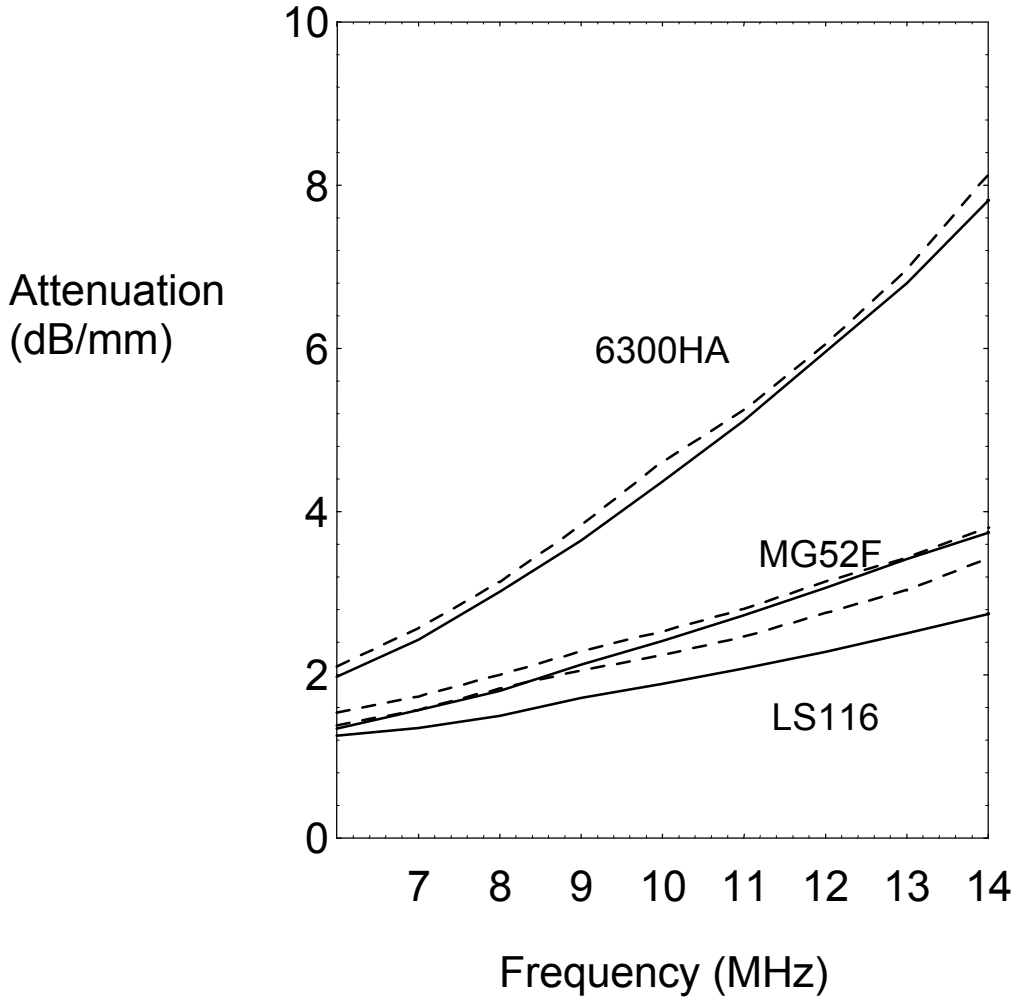

Figure 6. Effect of PMC on the frequency dependent attenuation for 6300HA and LS116F.

The frequency dependent attenuation curves for two molding compounds, Sumitomo 6300H and Plascon LS116F, are shown in Figure 6. These data were obtained with a 15 MHz, broadband transducer. As anticipated, PMC has only a minor effect on the frequency dependent attenuation for both molding compounds, confirming that cure was nearly complete even before PMC. The attenuation curve for 6300 HA corresponds extremely well with the data obtained from the flat specimens. Also, the two molding compounds retain their distinctly different curves even after PMC. Because the flat coupons and the PMC/no-PMC specimens were obtained from separate sources, the good correlation indicates that the data reported indeed reflect material characteristics and that the experimental technique used is robust.

Further work needs to be done to provide quantitative measures of the cure state. The effect of varying material parameters such as filler loading, cure state and low stress additives on the ultrasonic parameters such as attenuation and velocity needs to be determined before the inverse problem (deducing material parameters from the ultrasonic measurements) can be solved. As a first step in this direction, the

effect of volume fraction of 100 μm glass spheres on the slope of frequency dependent attenuation is examined in this study using calibration specimens.

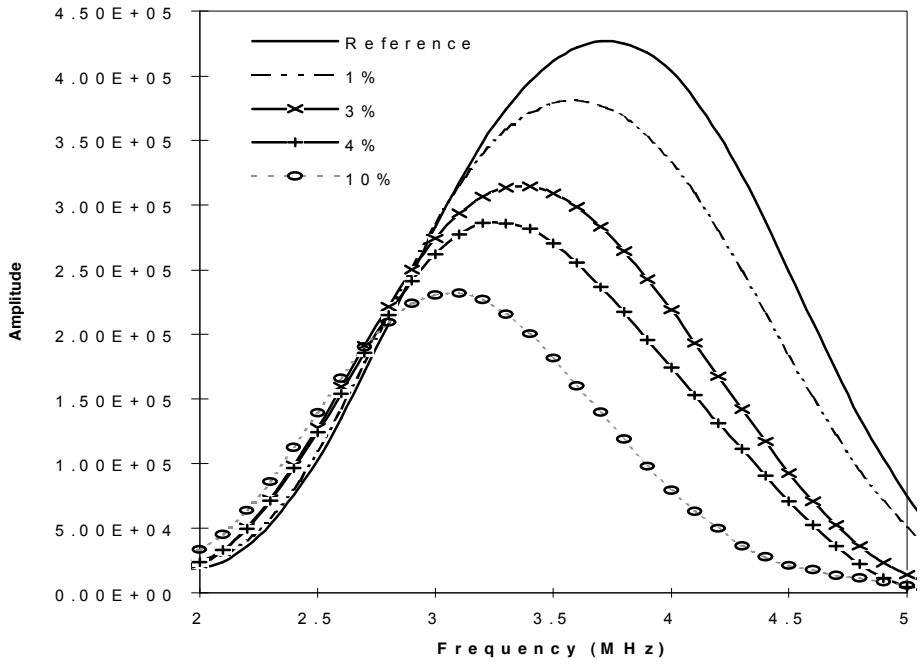

Figure 7. Magnitudes of the frequency spectra for different volume fractions of 100 μm glass spheres in clear epoxy resin.

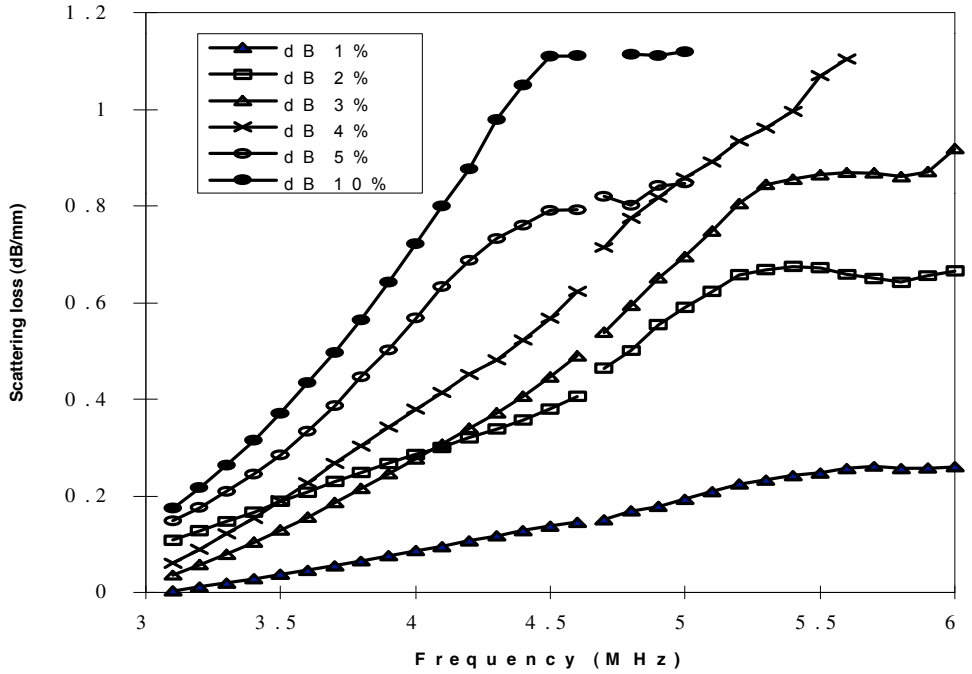

Figure 8. Scattering loss coefficients for the case of 100 μm glass spheres in clear epoxy resin.

Study on Calibration Specimens

The FFT spectra of the signals for a few volume fractions of 100 um solid glass spheres are shown in Figure 7 for a 3.5 MHz transducer. It is seen that as the volume fraction increases, the peaks shift to lower frequencies (also observed in molding compounds). The frequency dependent attenuation curves for the 100 μm spheres are shown in Figure 8. Only the data in the useful range of the transducers, where the amplitudes are sufficiently high, are shown. The data shown in Figures 8 show remarkable continuity even though they were obtained using different transducers. Since the reference signal includes the effect of absorption losses, the loss measured in these experiments is solely due to that caused by scattering only. In contrast, the loss measured for the molding compounds includes the effect of both absorption and scattering. Thus, the slope of the frequency dependent attenuation curves in Figure 8, arising from scattering of sound by the spheres, can be used to determine the volume fraction of the scattering objects. In accordance with scattering theories developed for a dilute distribution of scatterers [6,7] two features of the curves are analyzed, namely a) the slope of frequency dependent scattering loss b) the frequency at which the scattering loss loses its linear dependence with frequency (characterized by a plateau at higher frequencies).

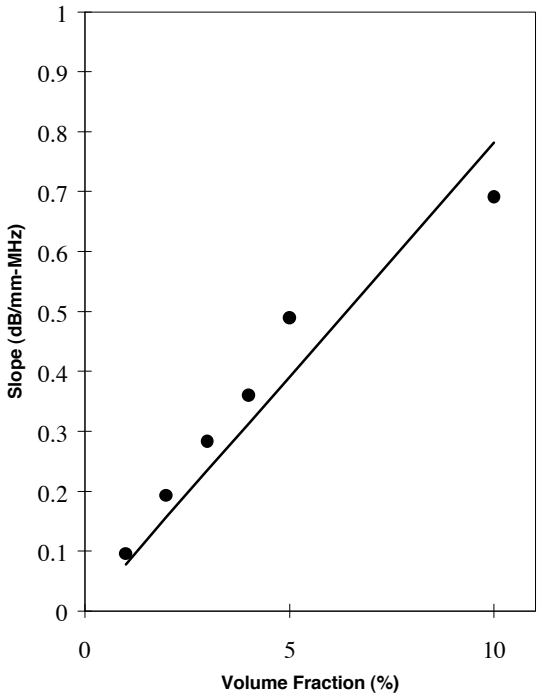

Figure 9. Correlation between the slope of the frequency dependent attenuation curve and the volume fraction of the glass spheres.

The volume fraction of the particles and the average particle size, respectively, are given by the relationships $c = (V_l / 2\pi K)(d\alpha / df)$ and $a = (V_l / 2\pi)(1/f_p)$ where V_l is compressional wave velocity, K is a scattering constant that depends on the material system, $d\alpha/df$ is the slope of the linear portion of the curve, and f_p corresponds to the point where the attenuation attains a plateau. The slopes of the linear portion in Figure 8, plotted in Figure 9 for the different volume fractions, correspond very well to the

volume fraction. This indicates that a similar procedure can be used to study voids in the molding compounds, and a quantitative estimate of the void fraction may be obtained for different locations of the part. The size of the beads estimated using a measured compressional wave speed of 3.2 mm/μsec and using a value of 5MHz for f_p, is 100 μm, which is in good agreement with the sphere sizes used in this study (95-120 μm).

Table 3. Ultrasonic properties measured on actual packages

SAMPLE ID	Molding compound	Compressional wave velocity (mm/μsec)	Filler content	Resin type	Acoustic Impedance
A	NITTO MP180S	3.30±0.004	low silica	OCN	5.44
B	NITTO MP7400	4.37±0.008	high silica	Biphenyl	7.97
C	SUMITOMO EME6300HG	3.29±0.016		OCN	5.72
D	NITTO MP190M	3.59±0.011	low silica	OCN	5.53
D*	NITTO MP180S	3.38±0.013	low silica	OCN	5.68
E	NITTO MP8000	4.00±0.014	medium to high silica	OCN	5.52
F	NITTO MP7150	4.12±0.04	high silica	Biphenyl	6.24
G	NITTO MP195	3.608±0.014	low silica	OCN	6.24
H	NITTO MP180S	3.36±0.008	low silica	OCN	5.45
I	NITTO MP180S	3.281±0.012	low silica	OCN	5.31

CONCLUSIONS

Different classes of molding compounds can be distinguished from each other nondestructively using ultrasonics in terms of their frequency dependent attenuation and acoustic impedance. For the molding compounds investigated in this study, it was found that the frequency dependent attenuation is lowest for the biphenyl based compounds, while different OCN resins have different slopes and higher values of attenuation. The velocity of ultrasound in the molding compounds is function of filler loading, and the attenuation appears to be controlled to a greater extent by the amount of low stress additives (flexibilizers). The attenuation coefficients were only weakly controlled by PMC, and the curves of different molding compounds remain distinctly different after PMC.

Solving the inverse problem , namely, estimating the void content and cure state from the ultrasonic data, requires further work on calibration specimens with carefully controlled compositions. For the relatively simple case of a resin with 100 μm spheres, the slope of the frequency dependent attenuation correlates well with the volume fraction. These results are encouraging for estimating void fractions of a few percent (larger voids), but needs to be extended for the case of relatively small particle size (~20μm) and high filler loadings encountered in practice (>60% by volume). Now that it has been established that ultrasonics is an attractive technique to characterize molding compounds, further efforts will be focused at better understanding the contribution of each ingredient on the ultrasonic properties. The database will be useful in identifying anomalous materials resulting from either improper curing, inhomogeneous filler loading, porosity or PMC by providing baseline properties.

ACKNOWLEDGEMENTS

Stimulation for this study was provided in part by Janet Semmens of Sonoscan who noted some interesting variations in acoustic impedance measurements among molding compounds used for

integrated circuits. Samples for this study have been accumulated from a wide variety of sources and special acknowledgment is owed to Walter Motta of SGS Thompson and Bradley Smith of Allegro Systems. Funding for the study is provided by the IR&D Program of Sonoscan, Inc.

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