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Ultrasonic Characterization Of Epoxy Composites Reinforced By Glass Powder

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Abstract :

The aim of this work is to find relationships between the selected parameters of an ultrasonic waves and the glass powder content in an epoxy composites . Epoxy composites materials with 10% , 20% , 30% , 40% , 50% and 60% glass powder volume fraction were all examined by means of pulse-echo ultrasonic . The experimental results showed relationships between , ultrasonic echo amplitude response , ultrasonic attenuation , ultrasonic velocity and glass powder content in the investigated composite materials. This study has also assessed the ability of pulse-echo technique to carry out such testing . The described method can be applied to the post-production quality control of a finished composite products and has great importance for products with a high failure-free requirements .

Keywords : Ultrasonic , epoxy , glass powder , composites .

توصيف فوق صوتي لمتراكبات الإيبوكسي معززة بمسحوق الزجاج

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الخلاصة:

الهدف من هذا العمل هو إيجاد علاقة بين متغيرات مختارة للموجة فوق الصوتية ومحتوى مسحوق الزجاج في متراكبات الزجاج / الإيبوكسي . مواد متراكبات الزجاج / الإيبوكسي وبنسب حجمية من الزجاج 10 % ، 20 % ، 30 %، 40 % ، 50 % ، 60 % تم إختبارها بواسطة صدى – النبضة فوق الصوتية . النتائج المختبرية أظهرت علاقة بين إستجابة سعة الصدى فوق الصوتية ، الاضمحلال فوق الصوتي ، السرعة فوق الصوتية ومحتوى مسحوق الزجاج في المواد المتراكبة التي تضمنتها الدراسة ، كذلك هذه الدراسة أظهرت أمكانية صدى – النبضة لإنجاز اختبارات من هذا النوع . الطريقة بهذا الوصف يمكن تطبيقها لأغراض السيطرة النوعية لما بعد الإنتاج على منتجات المتراكبات المصنعة وهي ذات أهمية كبيرة لمتطلبات المنتجات الخالية من العيوب .

1. Introduction:

Non-Destructive Evaluation Techniques , NDE , such as ultrasonic techniques have long been used to obtain data about the presence of cracks in materials. The information contained in the ultrasonic signals can be analyzed in terms of ultrasonic velocity , ultrasonic wave attenuation or structural noise . The methodology consists in the use of existing relationships between the elastic properties and density of the material and the ultrasonic velocities .[1]

It is also known that propagation of ultrasonic waves is sensitive to the variations in the microstructure and mechanical properties . Therefore , establishing a relationship between the micro structural and ultrasonic evaluation results could be very useful for improving the process parameters and controlling the quality of the products , .[2]

Addition of fillers into polymer materials is a common industrial practice. Fillers can change properties of composites and create new materials. Filler materials , filler volume fraction , filler size and shape , size distribution and interactions between matrix and filler , and finally interactions between solid particles affect mechanical and rheological properties of filled polymer composites.[3]

Epoxy resin are the most suitable polymer for composite mixtures , and extensive research has been carried out on their rheological behavior [3,4] , and their mechanical properties.[5,6] Highly cross-linked thermosetting polymers , such as epoxy resins and unsaturated polyesters , are an important class of synthetic materials . Their main distinction from other types of polymers lies in their density cross-linked molecular structure. [7]

Interrogation of specimen by ultrasonic pulses yields information relating to material acoustic properties and specimen dimensions.[8] The velocities of longitudinal and transverse wave were considered as representative quantities of the ultrasonic behavior by using pulse-echo measurements. The velocity of longitudinal waves is determined more simply and with greater accuracy than the velocity of transverse waves because these waves produced by special probes , show high attenuation when passing through the particle filled polymers.[4]

Less information is available on application of ultrasonic testing for reinforcement content determination in particulate composite materials.

The prime aim of the presently work is to explore the capability of ultrasonic pulse-echo amplitude technique to characterize certain properties of a glass powder/epoxy composites and mainly to evaluate the effect of glass powder content in these composites .

2. Material and Experimental Methods :

2-1 Materials :

The thermosetting matrix used in this study was epoxy resin . Strong coat EN100 Hardener and strong coat EN100 Base Grev (Resin) , all manufactured by Ayla construction chemicals under licence from DCP , England . Batch No. 2717 . The epoxy resin cured at room temperature with 40% by weight of hardener according to the manufacturer specification. The densities of the epoxy resin and hardener are 1.52 and 1.12 g/cm^3 respectively . Glass powder with particle size 150 µm was used as filler . The density of the glass was 4.35 gm/cm^3.

2-2 Samples preparation :

Six samples of the epoxy composites were fabricated as circular shape of 50 mm diameter and 10 mm thickness.

The epoxy and the glass powder were mixed mechanically by hand , after this homogenization process , the hardener were added , then the mixture was mixed again and powered into circular mold .

The volume fractions of glass powder were 10% , 20% , 30% , 40% , 50% and 60% The casted samples were cured under ambient conditions .

2-3 Ultrasonic Equipment and Measurement Procedures :

Fig.(1): Krautkramer Ultrasonic flaw detector USM2 type and test samples.

Figure.(1) , shows a photograph of the German USM2 ultrasonic pulse-echo measuring system used . The system consist of Krautkramer ultrasonic pulser-receiver flaw detector and a set of normal probes were used as transmitting receiving transducers of sound waves , producing ultrasound of 1 , 2 and 4 MHz, , respectively . A simple machine oil was used as transducer/specimen interface couplant . Constant load was applied on probes through the wide study .

The sound velocity C^*_1 of the longitudinal waves of each specimen was evaluated using the relationship [4] :

$$C^*_1 = C_1 (dx/dy) \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(1)$$

Where C_1 is the sound velocity of the reference block , dx is the real specimen thickness , and dy is the equivalent thickness of the specimen , which is measured on the screen of the oscilloscope. The main velocity values were calculated by repeating the measurement three times on each specimen .

Computation of apparent attenuation coefficient with longitudinal and shear wave velocities are very important for characterization of particulate composites using ultrasonic testing . ASTM standard E664-95 [5] is used for computing the apparent attenuation in these particulate composite samples . The apparent attenuation is computed by the Equation (2) [6,9] considering first and second back-wall echos :

$$\text{Attenuation} = \frac{\Delta\, dB}{2\, N\, T} \qquad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots(2)$$

Where ΔdB : amplitude difference between 1^{st} and n^{th} signal .

T : Specimen thickness in mm .

N: Number of signals .

The first back wall echo in the far zone was maximized to reach the 80%FSH (Full screen height) of energy dB_1 , after multiple reflection the 2^{nd} signal was also maximized by additional energy dB_2 , to reach the reference of the first signal (80 FSH) on the oscilloscope screen , then :

$$\Delta dB = dB_2 - dB_1$$

There measurements were done by each probe on each specimen , and the average value was calculated .

The speed of wave propagation and energy loss by interactions with material microstructure are key factors in ultrasonic determination of material properties . Relatively small variations of velocity and attenuation can indicate significant property variations.[10]

Mechanical properties of the glass/epoxy composites could be correlated with metallographic observations. Moreover , the results of mechanical and ultrasonic test can be associated with metallographic observations.

The ultrasonic non-destructive evaluation techniques employed may prove a useful addition for qualifying the homogeneity of glass powder distribution.

3. Results and Discussion :

Ultrasonic response estimation by pulse echo technique depends on the amplitude of the ultrasonic beam reflected back to the probe which is indicated as an echo height on the screen. This height is proportional to the reflected energy received from the back wall surface. The amplitude of the echo signal was recorded in dB , keeping the signal height at 80% FSH (Full Screen Height) as a reference through out the whole study .

Figure.(2) , showing the oscilloscope screen trace using 1 , 2 & 4 MHz probe frequency at 20% volume fraction interval of the test samples . Due to the lower signal to noise ratio using 1 MHz probe (masking the backwall echo due to high scattering) , all the results of 1 MHz probe were neglected from the whole study .

From **figure.(3)** it is shown that the echo amplitude response increases from 10% volume fraction to 40% volume fraction and then decrease from 40 to 60% volume fraction.

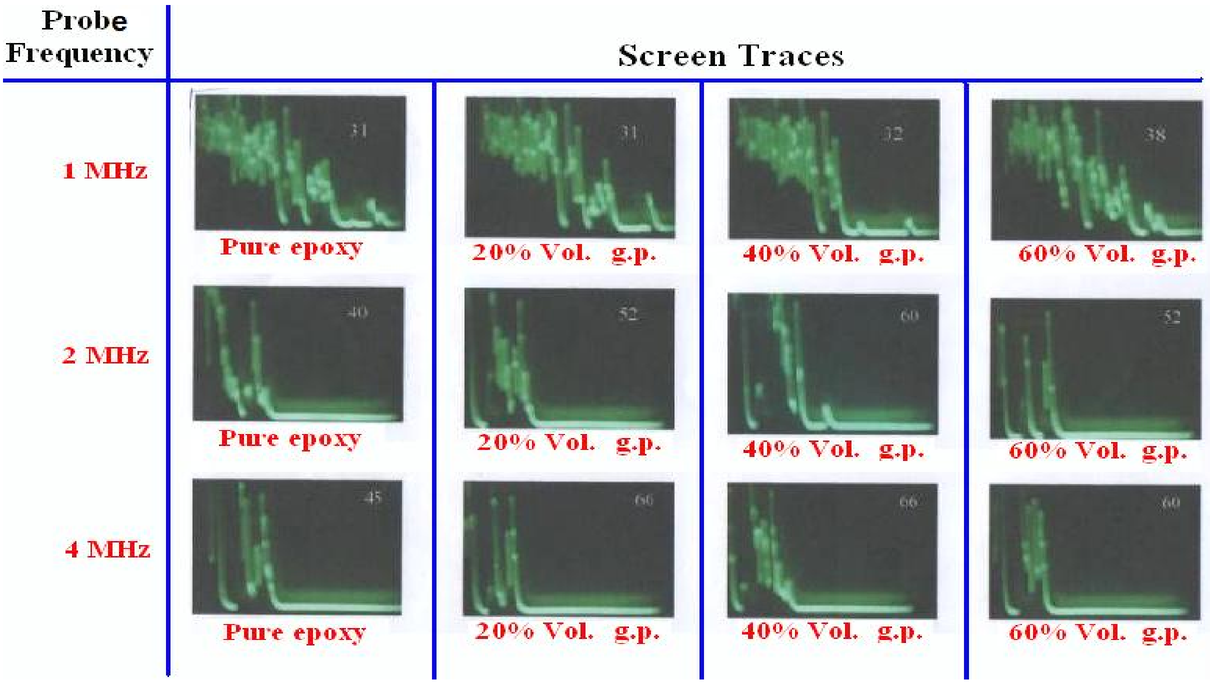
Figure.(4) shows the ultrasonic longitudinal velocity in the particulate composites with varying volume fraction at frequencies of 2 and 4 MHz . Particles volume fraction variation appears to have a little effect on the ultrasonic velocity. The longitudinal velocity in composites is lower when test was performed at 4 MHz frequency compared to the value computed using 2 MHz . This can be attributed to the fact that the wavelength of ultrasonic waves decreases with an increase of frequency . Scattering of ultrasonic waves at particles is dependent upon the ratio between wavelength and the particle size . Scattering of ultrasonic wave does not occur at each and every particle-particle interface rather than between clusters of particles. Due to the above said reasons , the longitudinal velocity of the composite computed at lower frequencies will be more than the longitudinal velocities computed at higher frequencies , as the wavelength decreases (increase in probe frequency) , wave starts interacting with the particles more extensively and results in increasing scattering . Due to this reason , the wave takes longer time to transfer through the composite sample and thus decreasing the ultrasonic velocity.

Figure.(5) shows that the attenuation coefficient values increases from 10% volume fraction to 30% volume fraction and then decrease from 30% volume fraction to 60% volume fraction . The increase of attenuation coefficient in the range 10-30% volume fraction due to the increase in particles number , thus increasing the scattering of the travelled ultrasonic energy in addition to the energy loses by absorption in the epoxy matrix . In this range the percentage of the epoxy is more than the percentage of the particles in the matrix , thus a large portion of the energy loses due to matrix absorption . The volume fraction in this range are termed as dilute suspensions as the particles are widely dispersed in the epoxy matrix.[11] However, after 30% volume fraction on using 2 and 4 MHz probes , the particles start getting close to each other increasing the possibility of contact between particles , and decrease the amount of epoxy in the matrix and thus decreasing the absorption on ultrasonic energy . Therefore , the ultrasonic signal has a higher probability of traveling through the particles rather than interacting with the epoxy matrix lying around. Thus the attenuation coefficient decreases in the range 30-60% volume fraction . Hence , it is concluded that attenuation of ultrasonic signals in these particulate composites is caused by wave absorption in epoxy matrix at low volume fraction of 10-30% and by scattering between particles at high volume fractions of 30-60% . At higher volume fraction greater than 60% no consistent signal was obtained due to the high attenuation in the samples . Therefore , the results obtained are not reported .

4. Conclusions:

Depending on the results obtained in this study , the main conclusions can be summarized as follows :

1. Particles volume fraction appears to have a little effect on the ultrasonic velocity.
2. The increase in attenuation coefficient in the range 10-30% volume fraction due to the increase in particle number , thus increasing the scattering of the travelled ultrasonic energy in addition to the energy loses by absorption in the epoxy matrix .
3. Attenuation of ultrasonic signals is caused by wave absorption epoxy matrix at low volume fraction of 10-30% , and by scattering between particles at high volume fraction 30-60%.
4. Greater than 60% volume fraction of glass powder there is no observable signal was obtained .
5. The test shows the unreliability of using the 1 MHz probe . Better investigation could be achieved using the 2 MHz probe compared to the 4 MHz probe .
6. The investigation shows the reliability of the pulse-echo amplitude technique to characterize the composites .



g.p.(glass powder)

Fig.(2): Oscilloscope screen Traces of selected samples , using normal probes of different frequencies

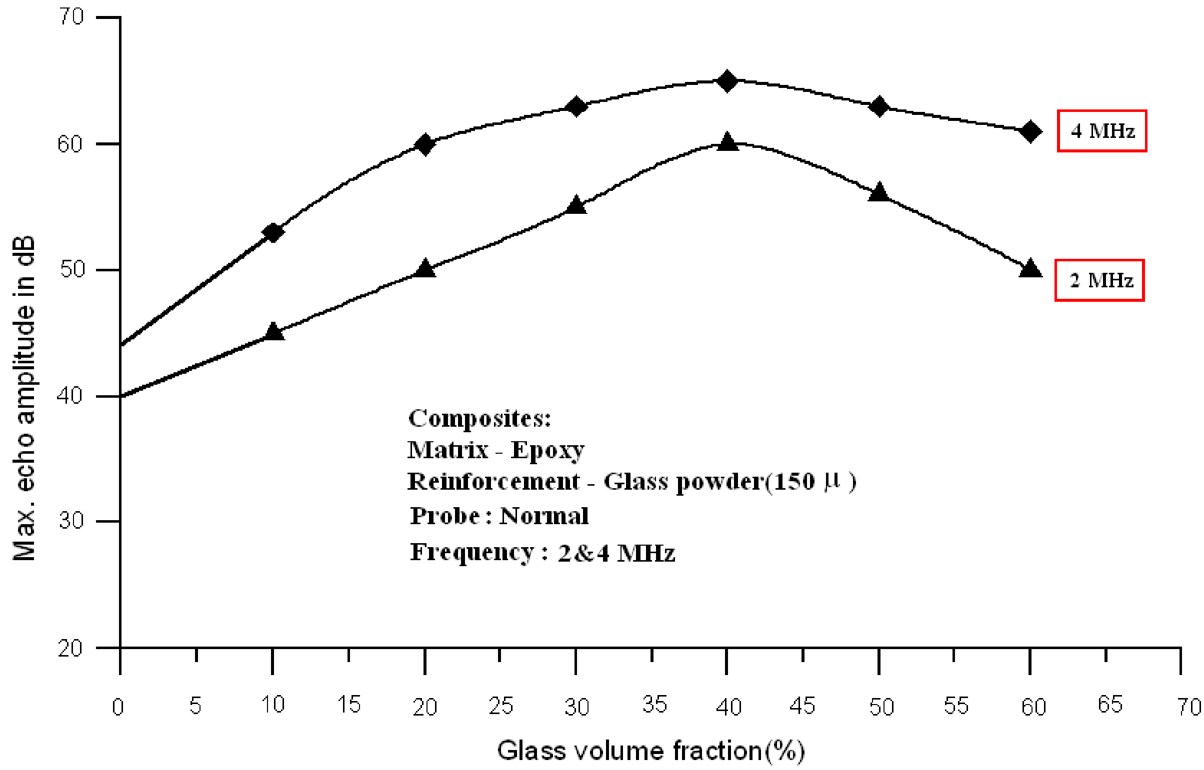


Fig.(3): The effect of reinforcement volume fraction (%)on the max. echo amplitude using normal probes of different frequency

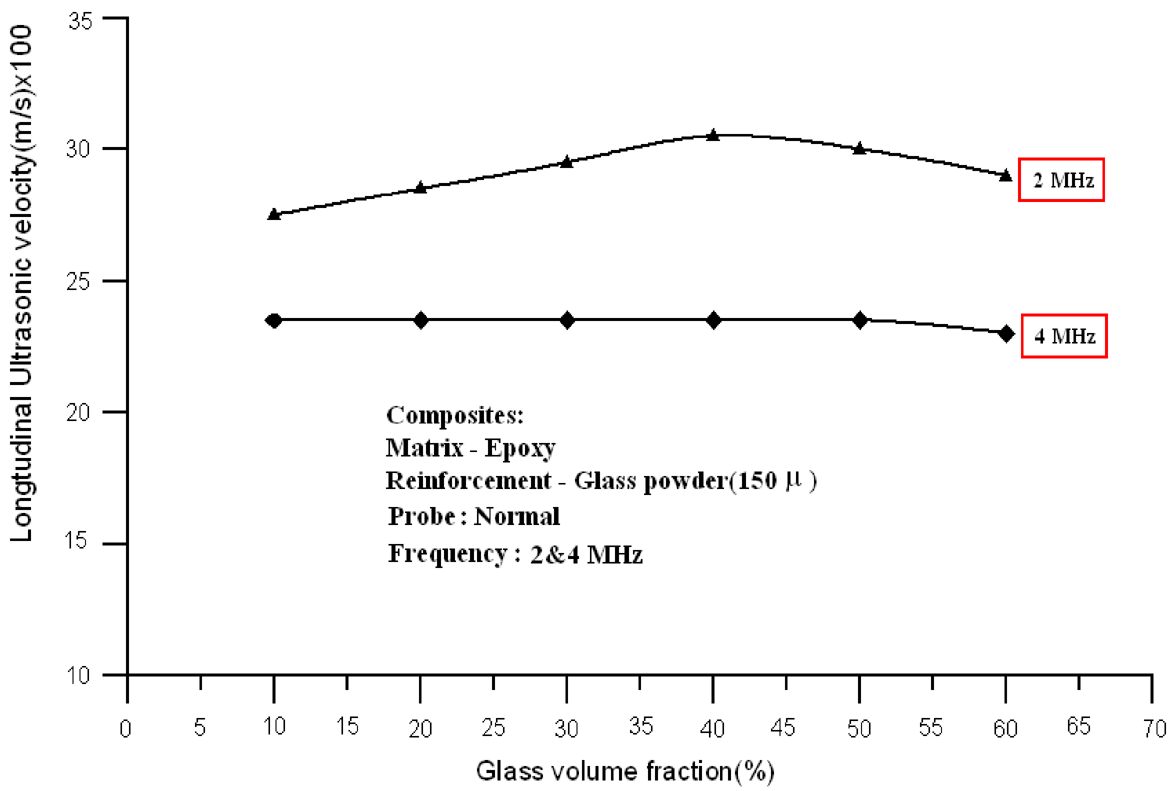


Fig. (4) : The effect of reinforcement volume fraction (%)on the ultrasonic velocity using normal probes of different frequency

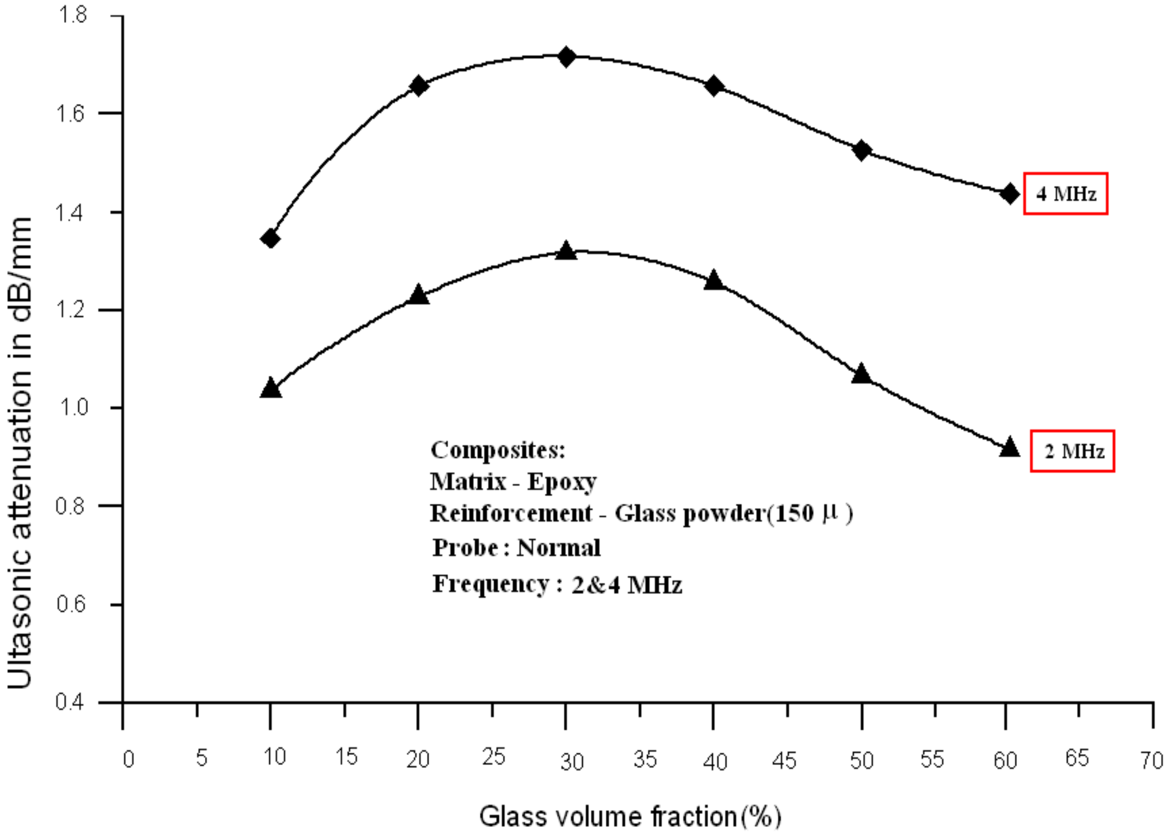


Fig. (5) : The effect of reinforcement volume fraction (%)on the ultrasonic attenuation using normal probes of different frequency

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