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Acousto-Ultrasonic Evaluation of Ceramic Matrix Composite Materials

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ACOUSTO-ULTRASONIC EVALUATION OF CERAMIC MATRIX MATERIALS

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ABSTRACT

Acousto-Ultrasonic (AU) nondestructive evaluation of ceramic composite specimens with a lithium-almino-silicate glass matrix reinforced with unidirectional silicon carbide (NICALON) fibers has been conducted to evaluate their reserve of strength. Ceramic composite specimens with different amount of damage were prepared by four-point cyclic fatigue loading of the specimens at 500 °C for a different number of cycles. The reserve of strength of the specimens was measured as the maximum bending stress recorded during four-point bending test with the load monotonically increased until failure occurs. It was observed that the reserve of strength did not correlate with the number of fatigue cycles. However, it was also observed that higher values of the SWF measurements correspond to higher values of the reserve of strength test data. Therefore, these results show that the acousto-ultrasonic approach has the potential of being used to monitor damage and to estimate the reserve of strength of ceramic composites.

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INTRODUCTION

Quality manufacturing with low production costs is, of course, the ultimate goal for all industrial sectors [1-3]. For advanced composites, because of their intrinsic nature and characteristics, maintaining this balance is especially difficult. Ceramics have been recognized as potential candidates for use in many structural applications by virtue of their potentially excellent mechanical integrity and chemical stability at high temperature. These structural applications include advanced heat engines, heat exchangers, and components subject to friction and wear. To date, structural ceramics with average strengths well in excess of high-performance ceramic component requirements do exist, but poor material reliability renders them unacceptable for use. The mechanical properties of ceramic components are often degraded by the presence of flaws. Mechanical behavior is very much affected by the size, number, and distribution of internal flaws, such as pores, inclusions, agglomerates, large grains, and many other microstructural irregularities [4,5]. Different models explaining the effect of various types of flaws on fracture behavior have been established and published in the literature. For example, statistical models for a complex matrix-inclusion system, and relations between strength and porosity, are given in the literature [6,7]. The strength reduction caused by large grains have also been studied, as well as the effect of flaw size and flaw-size/grain-size ratio on fracture behavior [8,9]. In view of the inherent complexity that exists in the quantitative evaluation and characterization of internal flaws and other microstructural irregularities, the quantitative prediction of mechanical degradation of ceramics as a result of defects has been difficult. Nonetheless, the development of new NDE techniques to detect and characterize the shape and size of the defects can be a significant quality-control measure to achieve structural reliability [10].

The sensitivity of mechanical properties to defects requires carefully controlled processing and finishing operations to improve reliability. As a consequence, on-line quality control and testing ply a vital role in the manufacturing of ceramics. Although proof testing can evaluate parts under operating conditions and eliminate defective parts, it is basically destructive and may damage those components that pass the test. Furthermore, proof testing can be time consuming and expensive. As a consequence, the need to further develop nondestructive techniques for the evaluation/characterization of advanced ceramics is apparent. Presently, nondestructive evaluation techniques (acoustic, and radiographic techniques as well as computerized tomography scanning systems) are being developed primarily to detect porosity, cracking (surface and internal), inclusions, density variations, and binder/plasticizer/sintering-aid distributions [11-15]. Although much work has been done on the evaluation/characterization of polymeric composites, much work needs still to be done on the nondestructive evaluation/characterization of damage in advanced ceramic composites [11-15]. The purpose of this study is to investigate the applicability of the acousto-ultrasonic stress wave factor technique to the nondestructive evaluation/characterization of damage in ceramic composites.

Analytical ultrasonics implies the measurement of material microstructure and associated factors that govern mechanical properties and dynamic response. It goes beyond flaw detection, flaw imaging and defect characterization and includes assessing the inherent properties of material environments in which the flaws reside.

Acousto-ultrasonics is an analytical ultrasonic NDE technique which measures the relative efficiency of energy transmission in the specimen. An ultrasonic pulse is injected with a transmitting transducer mounted on the surface of the specimen. A larger amount of damage (i.e., flaws, changes in the microstructure, etc.) in the specimen produces a higher signal attenuation, resulting in lower stress wave factor (SWF) readings. Traditionally, the SWF has been evaluated as the number of oscillations higher than a chosen threshold in the ring down oscillations in the output signal from the receiving transducer. The stress wave factor does not yet have a standard definition. In this study, a stress wave factor is any stress wave parameter in any domain, such as the time and frequency domains, that help to characterize the acousto-ultrasonic signal. In references [16-17] the reader can find in detail all the stress wave factors used in this study. The SWF has already been correlated with the mechanical strength of composite materials by Vary and Lark [18], Williams and Lampert [19], Kautz [20], and Govada, et al. [21]. SWF measurements have also been correlated, by the author of this proposed study, with damage in wire rope [22], with swelling of wood products [23], with the adhesive bond strength between rubber and steel [24,25], and with the adhesive bond strength of connections in wood structures [26]. A good review of analytical ultrasonics in materials research and testing is given in references [27-29]. As mentioned, the purpose of this study is to investigate the applicability of the acousto-ultrasonic stress wave factor techniques to the nondestructive evaluation/characterization of damage in ceramic composites.

EXPERIMENTAL PROCEDURE

To determine the feasibility of using the acousto-ultrasonic technique to nondestructively evaluate high temperature fatigue damage in ceramic composites, specimens with a lithium-almino-silicate glass matrix reinforced with silicon carbide (NICALON) fibers were manufactured using the slurry infiltration method [8]. The specimens with a fiber volume fraction of approximately 45% had a matrix material composition of 15% of Li_2O, 20% of Al_2O_3, and 65% of SiO_2. Specimens with dimensions of 6.35 mm x 9.525 mm x 63.5 mm (0.25" x 0.375" x 2.5") were manufactured and polished with a 320 grade wheel. To obtain specimens with different amount of damage the manufactured specimens were submitted to different numbers of four-point bending fatigue cycles at 500^o C (932^o F). Damage due to cyclic fatigue loading was applied on a MTS fatigue testing machine at a rate of 5 Hz. The applied sinusoidal force had a minimum value of 25 lbs (111 Newtons) and a maximum value of 175 lbs (778 Newtons). The major span and the minor span distances in the four-point bending test fixture are 2.05 in (52 mm) and 1.09 in (27.8 mm), respectively. Six groups with different number of fatigue cycles were prepared. Table 1 shows the number of specimens per group, the number of cycles per group, and the corresponding percentage of cycles to failure. The minimum load was arbitrarily chosen so that the specimen would remain properly aligned within the loading fixture. The maximum load of 175 lbs (778 N) was chosen by observing that three initial specimens, tested under these fatigue loading conditions, failed within 3% of each other, the lowest at 129,140 and the highest at 131,950 cycles of fatigue life. Therefore, for these loading conditions, the fatigue life (nf) was assumed to be 129,000 cycles. Five Hertz was chosen as the cyclic loading frequency because it allowed the specimens to be fatigued at a fairly rapid pace without introducing dynamic loading effects.

The specimens were separated into six groups based on the number of fatigue cycles. The number of fatigue cycles per group was determined by taking incremental percentages of the number of cycles to fatigue failure (nf). Table 1 shows the number of specimens in each group, the number of fatigue cycles for each group, and the corresponding reserve of strength of each specimen. A number of specimens failed well below nf while the specimens were being fatigued for groups 2 through 6. Table 2 shows the specimens that failed during the fatiguing process and the number of cycles at which they failed. Specimens failed prior to achieving the desire fatigue life at values as low as 0.8% to as hight as 73.8% of nf. Therefore, the concept of fatigue life (nf) for ceramic composites is not ideal and for the remainder of this study the specimens will be referred to as having a particular number of fatigue cycles and not as having a particular percentage of nf. The specimens were maintained at 500° C during the fatiguing process by using a circular oven that surrounded the loading fixture. A thermocouple was attached to the center of each specimen. The temperature of the specimen was maintained within 5° C of 500° C throughout the fatiguing procedure. Insulation was placed above and below the oven to maintain a constant temperature within the loading cell. The MTS testing machine also had a water jacket above the fixture to prevent the load cell transducer from being affected by the heat from the oven.

To determine the reserve of strength of each specimen a four-point bending test was performed were the load was monotonically increased until failure of the specimen occurred. An ATS Series 1100 Twin Screw Universal testing machine was used with a constant cross head speed of 0.3175 cm/min. (0.125 in./min.). The reserve of strength, defined as the maximum load supported by the specimen until failure occurs, is shown in Table 2. The reserve of strength of specimen 1-1 is not reported in Table 2 because during testing it was observed that the specimen was not properly seated on the test fixture.

A schematic diagram of the acousto-ultrasonic measurement system is shown in Figure 2 were both the transmitting and the receiving transducers are mounter on the same side of the test specimen. Other specimen-transducer configuration used in this study, namely straight through and offset configurations, are shown in Figure 3. The pulsing transducer was the Panametrics V306 wide band transducer with a central frequency of 2.25 MHz and a beam width of 1.27 cm (0.5") and the receiving transducer was the Panametrics A306S narrow band transducer with a central frequency of 2.25 MHz and a beam width of 1.27 cm (0.5"). Transducers with a center frequency of 5 MHz and 7.5 MHz were also used in a attempt to improve the correlation between the SWF and the reserve of strength.[16]. It was observed that the best results were obtained with the 2.5 MHz transducers reported here. A dry silicone couplant disk with a thickens of 0.254 mm (0.01") was used between the transducers and the specimens. The center-to-center spacing between pulsing and receiving transducers was 1.98 cm (.781") and a contact pressure between the transducers and the specimens of 470 kPa (68.18 psi) was experimentally found to be adequate for saturation.

The transmitting transducer was excited by an ultrasonic pulser/receiver (Panametric, Model 5055 PRM) which was set at a pulsing rate of 200 pulses/s with the energy set at 1. The output signal from the receiving transducer was amplified 40 dB in an amplifier (Panametrics, Model 5678) with a passband filter between 0.5 MHz and 40 MHz. A synchronous trigger as well as the received signal were also sent to an AST computer equipped with an analog to digital converter (Sonotek, Model STR*8100) board. The Digiscope software package developed by Sonotek, Inc. was used to display the signal on the AST monitor and to store the signal on floppy diskette. To stabilize the signal and to further reduce noise effects, Digiscope was set to average sixteen AU waveform signals into every waveform that was saved. Each acousto-ultrasonic signal was saved using 1024 points. Once the acousto-ultrasonic signals were stored on diskette, they were quantified, using the stress wave factor approach, by the methods described in reference [16]. This was accomplished using the waveform measurement and analysis program (WAVEMAP) [17]. The WAVEMAP program stored these parameters in a file which can be imported into a LOTUS 1-2-3 spreadsheet. In LOTUS 1-2-3, a least squares fit regression was performed to obtain correlations between the SWF parameters and the reserve strength test data shown in Table 1.

EXPERIMENTAL RESULTS AND CONCLUDING REMARKS

Acousto-ultrasonic stress wave factor measurements have been conducted on ceramic composites with lithium-almino-silicate glass matrix reinforced with silicon carbide (NICALON) fibers. Stress wave factor measurements were recorded and the results correlated with the reserve of strength test data obtained during four-point bending static tests were the load was monotonically increased until failure occurred.

The stress wave factor does not yet have a standard definition. In this study the stress wave factor is assumed to be any useful ultrasonic parameter in any domain such as the time and frequency domains. A variety of used stress wave factors is provided by Reis et al. [16,17], and only a typical set of experimental results is presented there.

Figure 4 shows the number of fatigue cycles (i. e., damage)as a function of the reserve of strength (i. e., the maximum bending stress registered during the four-point bending test of specimens until failure occurs, see table 2). The lack of correlation precludes the number of fatigue cycles to be used as an indicator of the amount of damage. This is probably due to the existence of different defects in the virgin specimens due to variations during the manufacturing process. The fact that eight specimens failed during the fatigue procedure, seven failing below 40 percent of the estimated nf, gives an other indication that the initial states (i. e., defects) of the specimens were significantly different. Therefore, it may be concluded that knowing the loading history of the specimens is insufficient to determine the reserve of strength in ceramic composites. The defects in the virgin specimens must be taken into consideration when predicting the reserve of strength of ceramic composites.

Figures 5 and 6 show the average threshold crossings and the average rectified area in the time domain, respectively, as a function of the reserve of strength of the test specimens using the straight-through transducer configuration. In the frequency domain, Figure 7 represent the average zeroth moment of the power spectral density (area under the curve), also chosen as a stress wave factor, as a function of the reserve of strength for same side transducer configuration. The results shown in Figure 7 can prove essential in cases when only one side of the part being tested is accessible for inspection. Figure 8 show the average threshold crossings in the time domain as a function of the reserve of strength of the test specimens using the offset transducer configuration. In Figures 5-8, for each of the nineteen data points, the average SWF represents the average of six measurements.

In Figures 5-8 the average stress wave factor value was regressed on the reserve of strength and the results are provided on each figure. It was observed that higher values of the SWF measurements correspond to higher values of the shear strength test data.

The results of this investigation also show that the acousto-ultrasonic approach has great promise for evaluating damage in ceramic composites. Several stress wave factors, particularly the peak amplitude and the rectified area in the time domain as well as the zeroth moment of area in the frequency domain, show relative good correlation with reserve of strength. The results indicate that the approach could be used for quality control, eliminating the need for a costly destructive testing program. In addition, there is potential application of this approach for evaluation of damage in ceramic composite parts already in the field. Along with pattern recognition methods, the AU approach should provide an accept/reject criteria for quality control of ceramic composites eliminating the need of costly destructive testing methods. For more effective and practical applications of the technique a more detailed investigation should be undertaken by studding ceramic composites with seeded defects.

Table 1. Ceramic Composite Specimens With Different Reserve of Strength

Group Number	Number of Specimens	Percentage of (nf)	Number of Cycles	Specimen Name	Maximum Bending Stress	
					(kPSI)	(MPa)
1	5	0	0	1-1	-------	--------
				1-2	63.92	440.73
				1-3	72.31	498.55
				1-4	65.26	449.97
				1-5	70.15	483.66
2	3	20	25,800	2-1	41.85	288.56
				2-2	46.38	319.78
				2-3	56.52	389.66
3	3	40	51,600	3-1	56.90	392.32
				3-2	48.44	333.97
				3-3	62.05	427.79
4	3	60	77,400	4-1	40.67	280.40
				4-2	32.93	227.02
				4-3	62.77	432.75
5	3	80	104,320	5-1	54,84	378.13
				5-2	45,94	316.76
				5-3	42.73	294.59
6	3	90	116,100	6-1	58.68	404.55
				6-2	50 83	350.46
				6-3	45.94	316.76

Table 2. Composite Specimens with Premature Failure During the Fatigue Procedure

Number of Specimens	Number of Cycles For Fatigue Failure	Percentage of (nf)-%
1	32,680	25.3
1	6,380	4.9
1	1,060	0.8
1	340*	-------
1	95,240	73.8
1	14,680	11.4
1	43,710	33.9
1	35,100	27.2

*Hydraulic machine skipped during load adjustment

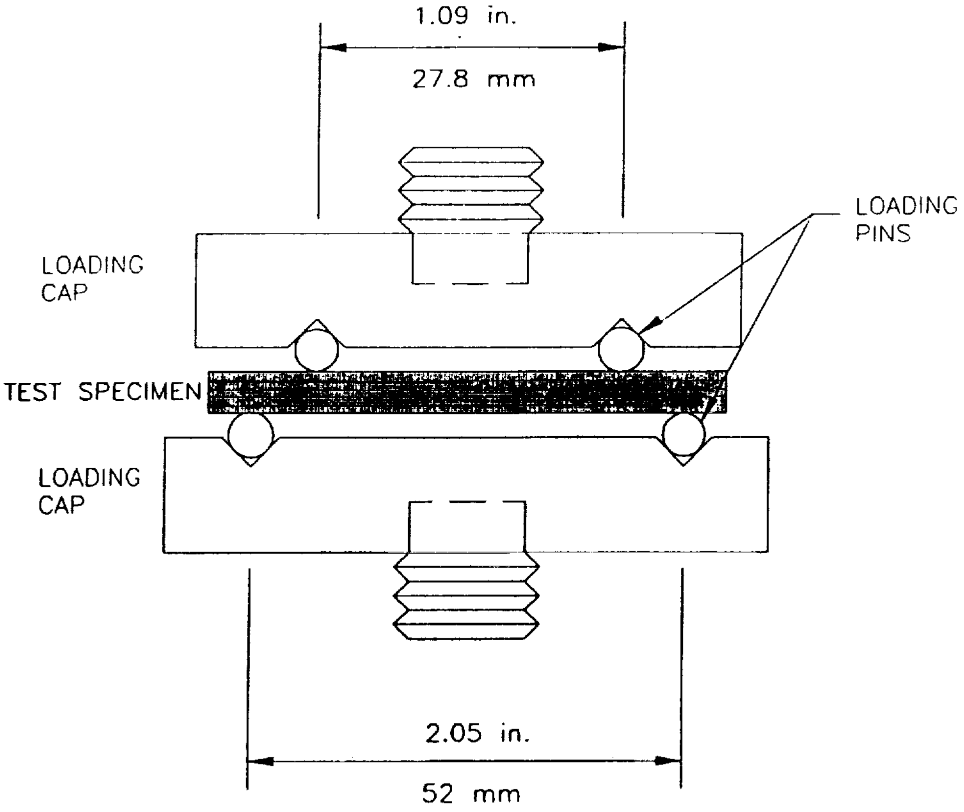

Figure 1. Four-Point Bending Test Fixture.

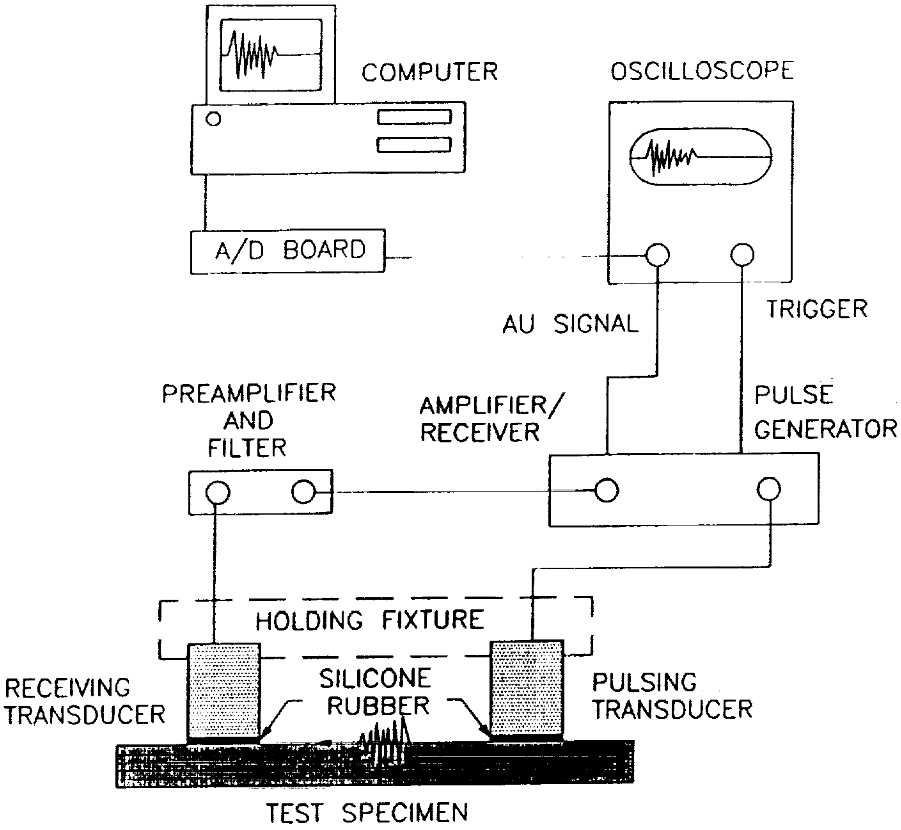

Figure 2. Typical Acousto-Ultrasonic Data Acquisition System with both the Transmitting and Receiving Transducers on the Same-Side of the Test Specimen.

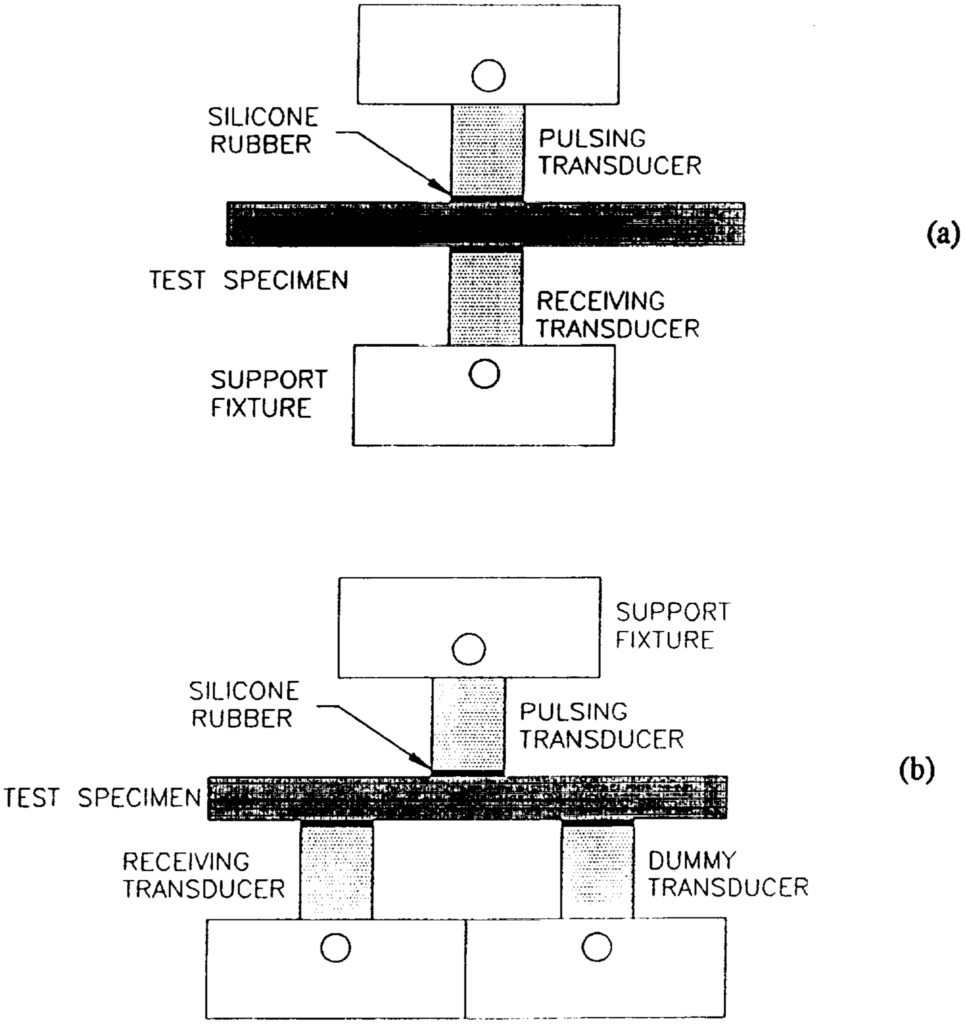

Figure 3. Other Typical Specimen-Transducers Configuration: (a) Straight-Through; (b) Offset.

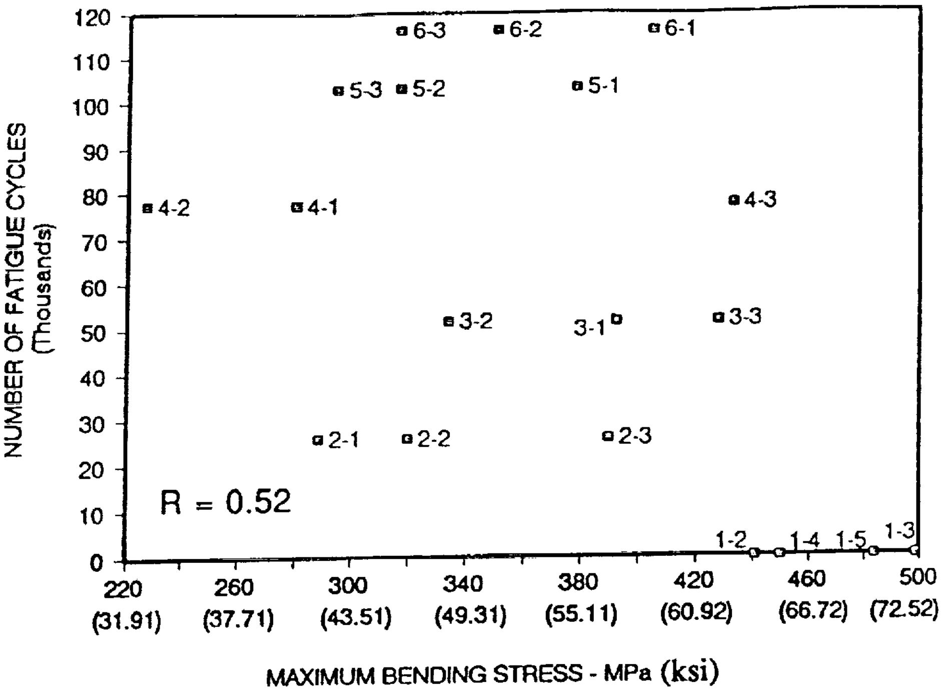

Figure 4. Number of Fatigue Cycles versus Maximum Bending Stress.

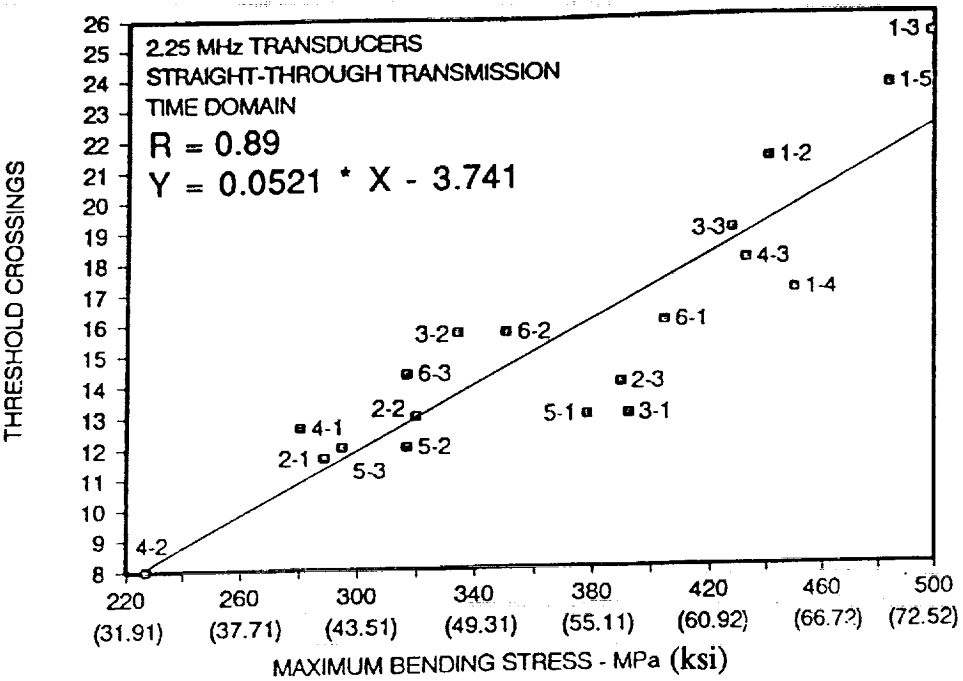

Figure 5. Average Threshold Crossings versus Maximum Bending Stress for Straight-Through Transmission.

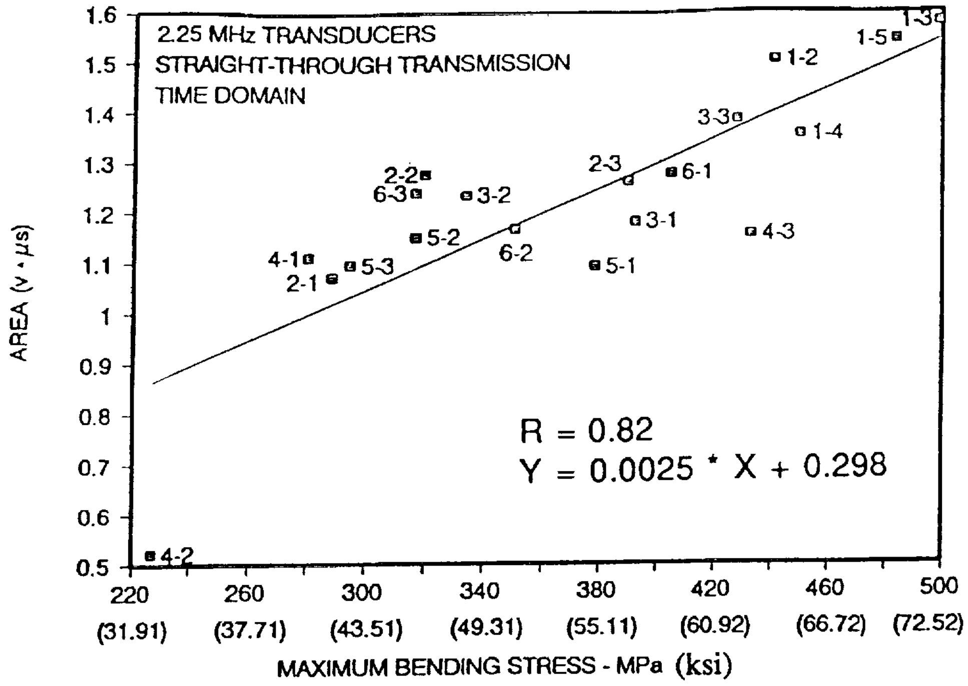

Figure 6. Average Rectified Area versus Maximum Bending Stress for Straight-Through Transmission.

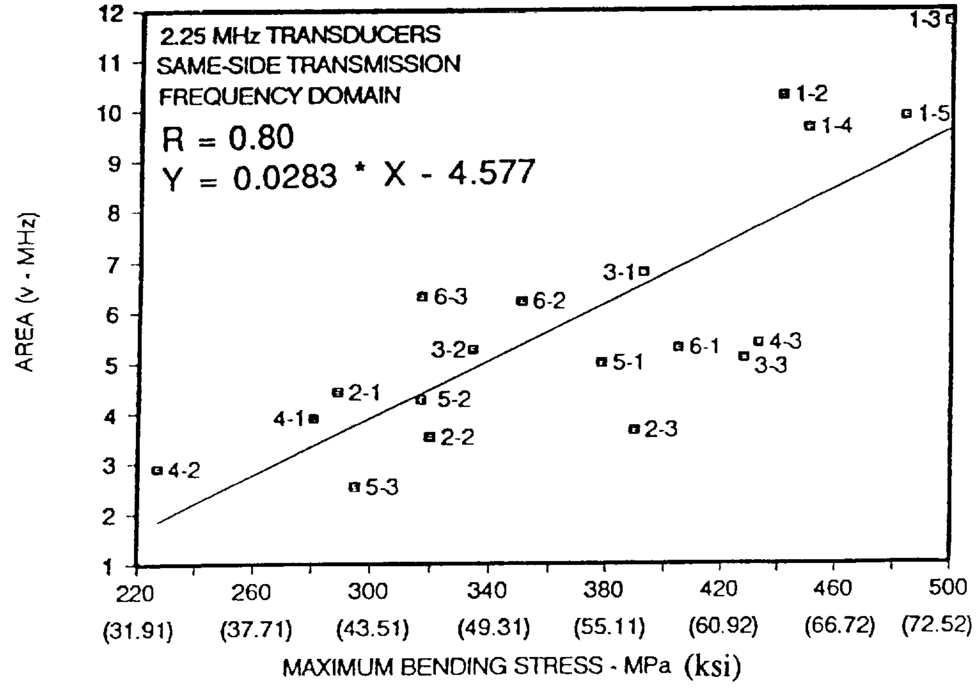

Figure 7. Average Area Under the Power Spectral Density Curve Versus Maximum Bending Stress for Same-Side Transmission.

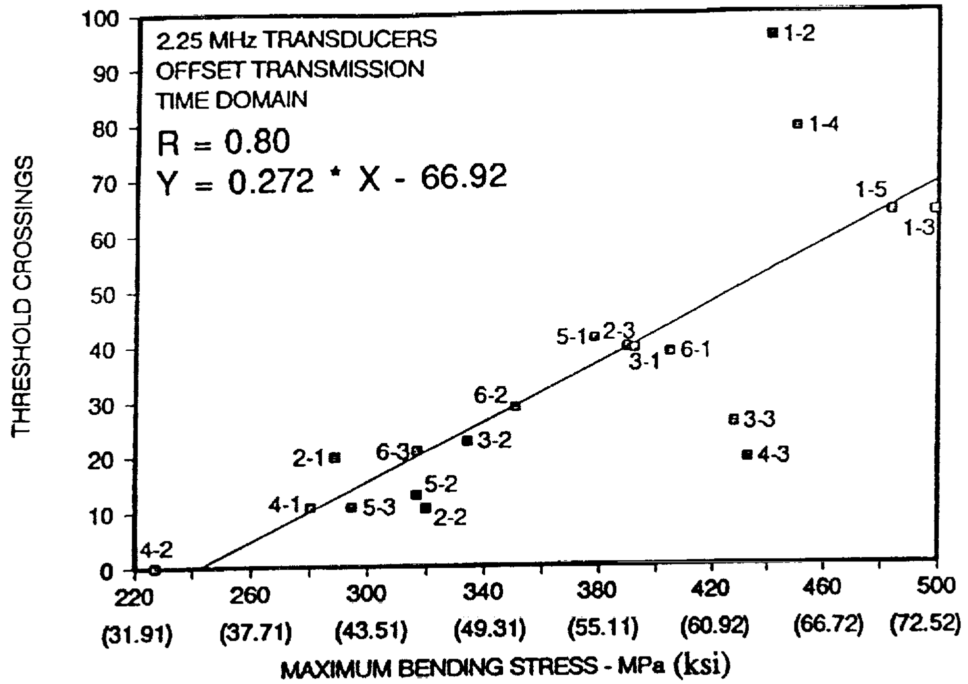

Figure 8. Average Threshold Crossings Versus Maximum Bending Stress for Offset Transmission.

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