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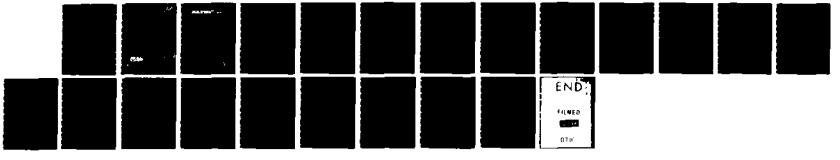

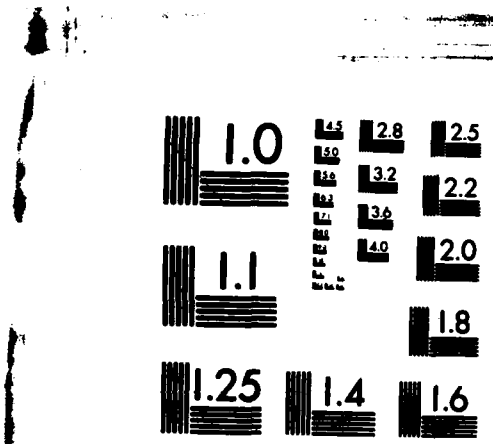

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NONLINEAR ULTRASONIC CHARACTERIZATION OF OXYGEN IMPURITIES IN TITANIUM

FINAL REPORT
R82-995862
MARCH 1982

Naval Research Laboratory

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UNITED TECHNOLOGIES RESEARCH CENTER
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Nonlinear Ultrasonic Characterization of Oxygen Impurities in Titanium

Final Technical Report

Prepared for

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FOREWORD

This final report documents research performed by United Technologies Research Center, East Hartford, Connecticut, under Contract Number N00014-81-C-2426. The effort was sponsored by the Naval Research Laboratory, Washington, D. C. with Mr. H. H. Chaskelis as technical monitor. Dr. H. I. Ringermacher and Dr. R. S. Williams were technically responsible for the work.

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Consultations with Dr. Akira Hikata of Brown University, Providence, R. I., proved to be most helpful and enlightening.

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The efforts of Florence P. Wendell in conducting essential and concise library searches were highly appreciated.

Nonlinear Ultrasonic Characterization of Oxygen Impurities in Titanium

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Nonlinear Ultrasonic Characterization of Oxygen Impurities in Titanium

SUMMARY

Titanium specimens containing oxygen impurities ranging from 0.1 to 0.3 percent by weight have been examined using nonlinear ultrasonic techniques. The amplitude of the second harmonic ultrasonic response has been observed to deviate from the expected perfect square law behavior. This deviation is consistent with theory that relates dislocations and impurity content to ultrasonic second and third harmonic generation in materials and, in the present work, shows an excellent correlation to oxygen impurity content. The results of these tests are presented and directions for future work outlined.

RESULTS AND RECOMMENDATIONS

Nonlinear second harmonic ultrasonic techniques were applied to determine the presence of oxygen impurities present in five titanium specimens provided by the Naval Research Laboratory. The results obtained demonstrate an excellent correlation between second harmonic amplitude deviation from expected square law behavior and the oxygen impurity content.

Consequently, it is recommended that ultrasonic nonlinear techniques for the detection of oxygen in titanium be investigated further with attention given to the following specific tasks:

1. Verification of the original effect, with measurements taken at several different points on the titanium specimens.

2. Measurements on a set of titanium specimens having varying oxygen content but the same micrograin structure.

3. Direct measurement of third harmonic generation as a potentially more sensitive indicator of oxygen contamination.

INTRODUCTION

The presence of oxygen in titanium alloys is known to alter their material properties. Oxygen concentrations as low as 0.1 wt% dramatically increase the strength and hardness of these alloys (Ref. 1). At the same time, however, the alloy ductility is decreased (Ref. 2). These effects are particularly important at weld sites where oxygen embrittlement can pose a serious problem when contamination occurs. The level of contamination will determine the severity of embrittlement. It is, therefore, useful to seek a simple, nondestructive, field-worthy technique with high sensitivity to the presence of oxygen impurities in these alloys.

Ultrasonic techniques generally satisfy the above requirement. Hardness and internal friction must influence sound velocity and the attenuation - frequency spectra in these materials. At the very least, the presence of oxygen is expected to change the density and, hence, the sound velocity. In addition, oxygen should alter the elastic constants, although observation of this effect can be more complicated.

THEORY

Basis for Nonlinear Methods

For the present task, a novel nonlinear ultrasonic approach was chosen, since it is well known that the motion of dislocations in single crystals can be a source of second harmonic generation (Ref. 3). Hikata has shown (Ref. 4) that the presence of impurities can pin dislocation motion thereby altering both the second and third harmonic content. Grain size in a polycrystalline sample may also have an effect on harmonic generation. While this is essentially an unknown area, Jon, et al., (Ref. 5), have shown that 99.96 percent pure polycrystalline titanium demonstrates a strict square law second harmonic behavior, apparently arising from lattice anharmonicity.

The present nonlinear approach is based on the assumption that oxygen acts upon the dislocations either by pinning or by generating localized stresses and thereby alters the third harmonic content of the observed ultrasonic signal. If perfect square law behavior for second harmonic generation is assumed, then the presence of third harmonics, dependent on oxygen content, will appear as a deviation from square law behavior and vary monotonically as a function of the oxygen concentration in the titanium specimens.

Basis for Linear Methods

For the sake of completeness, the titanium specimens were also characterized using linear ultrasonic techniques. In particular, velocity of sound and attenuation - frequency spectra were investigated. As discussed further, under Experimental Results, correlation between oxygen content and the attenuation - frequency spectrum was observed. However, it was not as well resolved as that observed with the nonlinear methods. Further, sound velocity data, within experimental accuracy showed no dependence on oxygen content.

EXPERIMENTAL TECHNIQUES

Specimens and Transducers

Five, rough surface, titanium plate specimens, with wt.% oxygen contents of 0.075 (specimen A), 0.136 (B), 0.194 (C), 0.238 (D), and 0.290 (E) were provided by the NRL for the present investigations. Prior to performance of the ultrasonic tests, the plates were machine ground flat with opposite parallel faces.

Lithium niobate ($LiNbO_3$) transducers were used for all the nonlinear data collection. A 1.25 cm dia, 5 MHz, 36°-Y cut transducer was used as the transmitter for longitudinal waves. The receiver, bonded on the opposite side of the plate, was a 0.625 cm dia, 10 MHz transducer of the same variety. All the transducers were bonded with Nonaq grease and "wrung in". Contact to the transducers was made through fine wires spot-soldered on the ground side and center of the coaxial plating.

Instrumentation

A block diagram of the ultrasonic system assembled for the nonlinear measurements is shown in Fig. 1. A Matec 515A gated RF amplifier provided 2 μs pulses at up to 2 kW power to the $LiNbO_3$ transducer. Alan 50HT82.5 high power attenuators on the input and output sides permitted precisely controlled attenuation changes for calibration control of the data. To ensure correct Alan attenuator calibration, 3 dB, 50 ohm feedtrhough, fixed attenuators were placed on both sides of the Alans so they were certain to look into 50 ohms in both directions. In this way, the attenuator switch combinations could be made to reproduce performance within 0.25 dB. The input pulse was sent through a 5 MHz lowpass filter to inhibit transmission of 10 MHz rf, and the received signal was passed through a 10 MHz high pass filter to eliminate the 5 MHz fundamental transmission signal while permitting passage of any second harmonic 10 MHz rf generated within the sample as a result of its nonlinear response. A Matec 251 tuned preamp further restricted the bandwidth. A Matec 605 broadband receiver was used for the final gain stage to a recording instrument.

All the linear ultrasonic data was taken on a similar Matec system (Fig. 2) that was microprocessor controlled to vary frequency over a programmed range, and accumulate and plot attenuation data.

Procedures

It was important, in taking the nonlinear data, to ensure that the observed nonlinear behavior was not generated in the electronic equipment. To avoid this, the receiver amplifier was always operated at or near a fixed point on its response

FIG. 1

ULTRASONIC SYSTEM FOR ANALYSIS OF MATERIAL NONLINEARITY

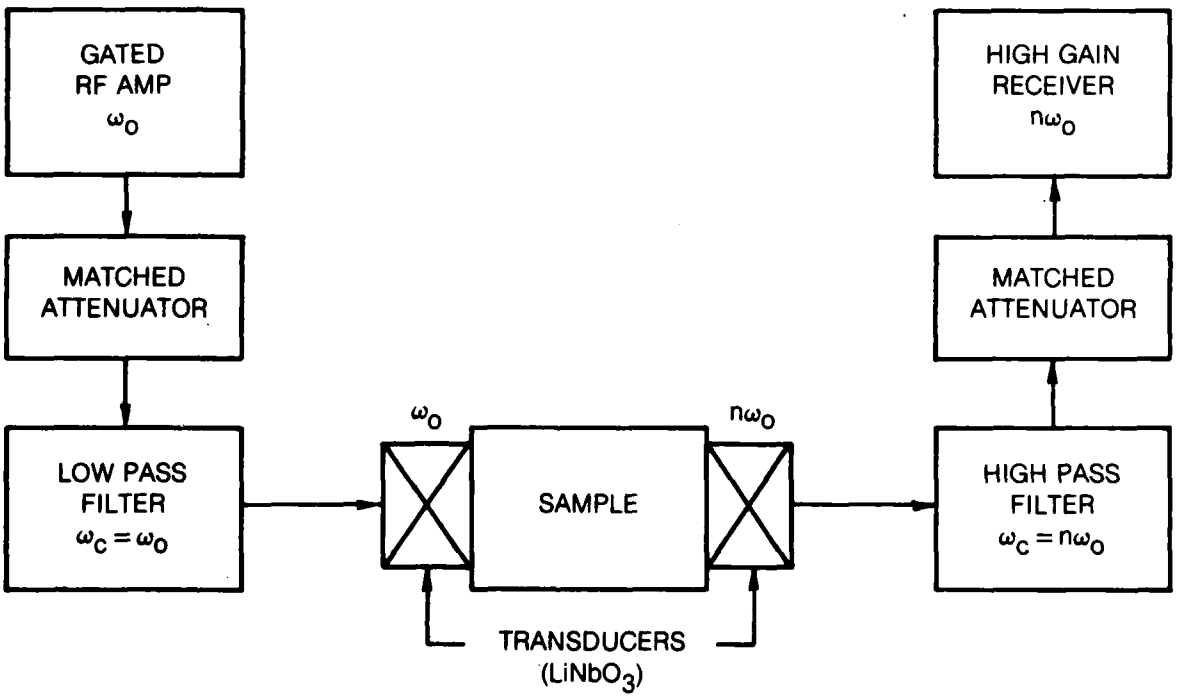

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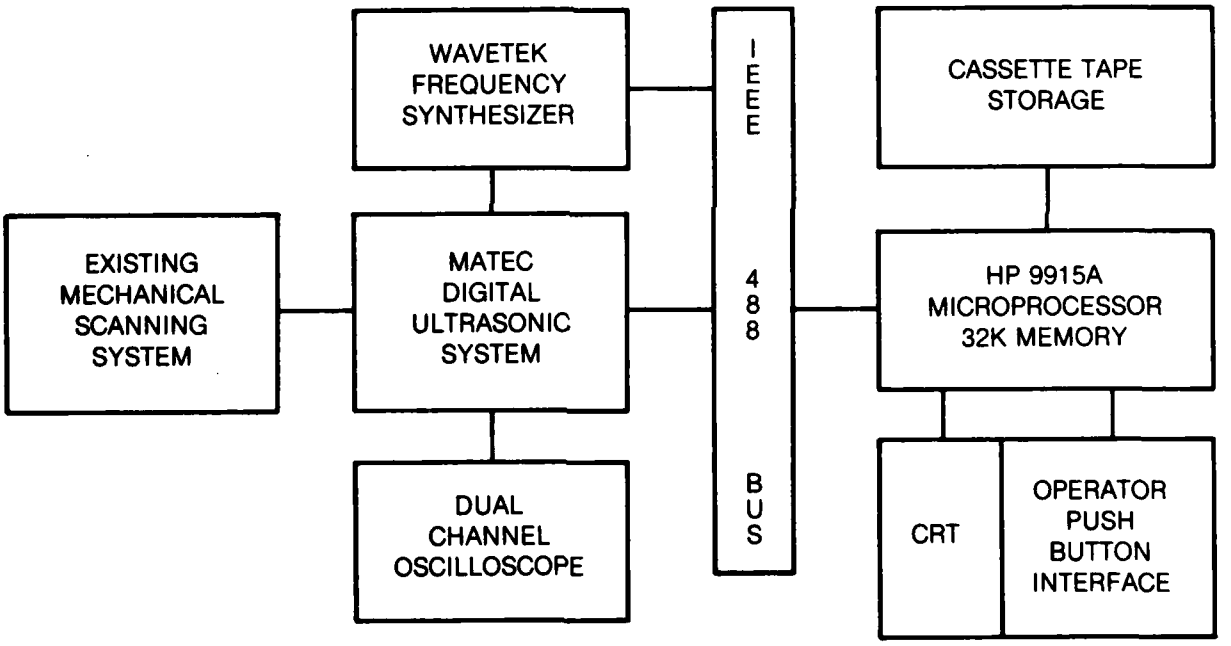
FIG. 2

ULTRASONIC SYSTEM FOR AUTOMATED MEASUREMENT OF ATTENUATION



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curve, for all input signal levels. It was assumed that the second harmonic amplitude A_2 is precisely proportional to the square of the fundamental A_1:

$$A_2 = KA_1^{2.000} \tag{1}$$

Starting at the highest power level to the input transducer, for every 1.0 dB of attenuation inserted at the input side, 2.0 dB was withdrawn on the output side. This, in effect, fixed the exponent at 2.000. In this manner, a perfect square law response would show as a constant signal out independent of signal in. Matching the attenuators as just described, and using lithium niobate, which is known to have an extremely linear response at high power levels, further helped to ensure valid results. System linearity was constantly monitored with each new power level, and maintained to within 1.5 percent.

A single, arbitrary point on the surface of each specimen was used for taking the power data rather than averaging many points. The consistency of the resulting data suggests that this technique represents an absolute method of comparison between independent samples.

EXPERIMENTAL RESULTS

Nonlinear Method

Data was obtained over an input amplitude range of 16 dB with a maximum input voltage to the transducer of approximately 400 volts peak-to-peak. The input amplitude was decreased from its maximum value in 2.0 dB steps, while output attenuation was simultaneously removed in 4.0 dB steps. To first-order, the output amplitude remained constant, thus indicating an approximate square law output behavior with input amplitude change. Since the deviation was small, a statistical method that disregards the quantitative nature of the deviation within each sample data set was employed. An average output amplitude A_2 was calculated for each sample using the power levels evaluated. The deviation of each amplitude level from the average output amplitude was taken to be a statistical representation of the deviation from perfect square law behavior and a mean square deviation, ΔA_2, was calculated for each sample.

The mean square deviation was then translated into an exponent deviation, ΔN, using:

$$\Delta N = N \frac{\Delta A_2}{A_2 \mathrm{Ln} A_2}$$

Here we set $N = 2.000$.

The nonlinear data results are presented in Fig. 3, with the exponent deviation from perfect square-law behavior, ΔN, plotted against oxygen concentration. As can be seen, excellent correlation exists between a linear increase in the exponent deviation and the oxygen contamination with the exception of sample B.

Experimental error in the data arises primarily from the $\pm$ 0.25 dB attenuator calibration error. Over a 16 dB input amplitude range, this amounts to an average error of 8 percent or less; i.e., within the circular regions plotted in Fig. 3.

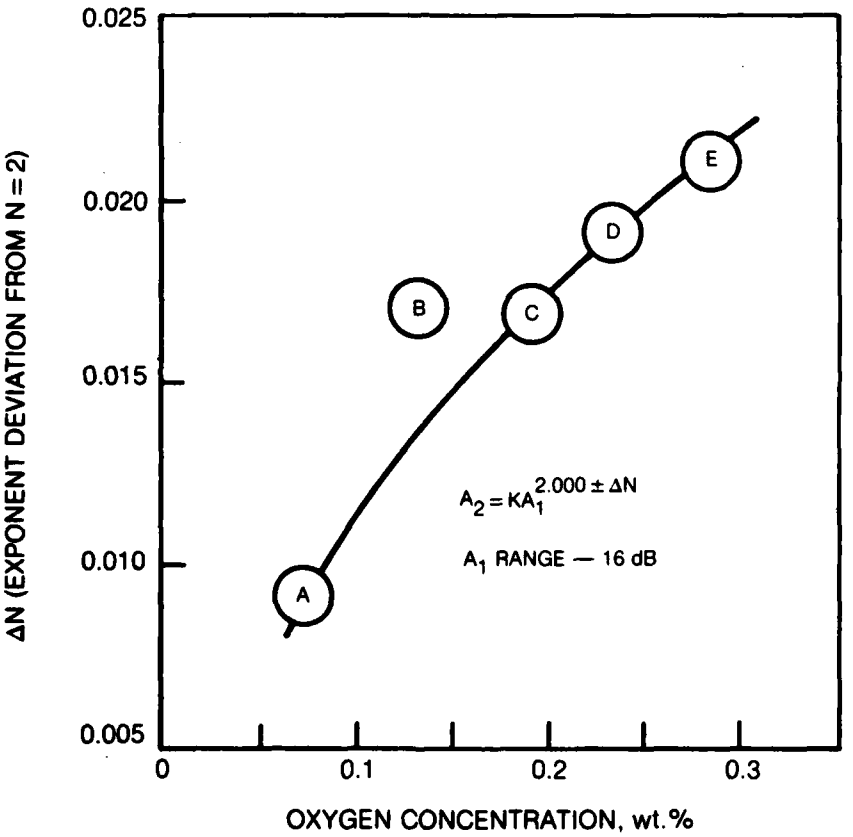
Linear Methods

Velocity of Sound Measurements

Velocity of sound data was obtained on all five titanium specimens using direct pulse-echo transmission methods. Results are shown in Table I. There are essentially no differences in the measured velocities within the $\pm$ 1 percent experimental error. Hsu and Conrad has previously reported (Ref. 6) an increasing sound velocity with oxygen content in titanium which they attributed to changing density and elastic

FIG. 3

SQUARE LAW DEVIATION AS A FUNCTION OF OXYGEN CONCENTRATION



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constants. However, their work involved specimens with oxygen contents up to 0.9 wt.%, while the present data extends only to 0.3 wt.%. The present velocity values agree with their low concentration values to within their data scatter.

TABLE I

SUMMARY OF SPECIMEN PROPERTIES

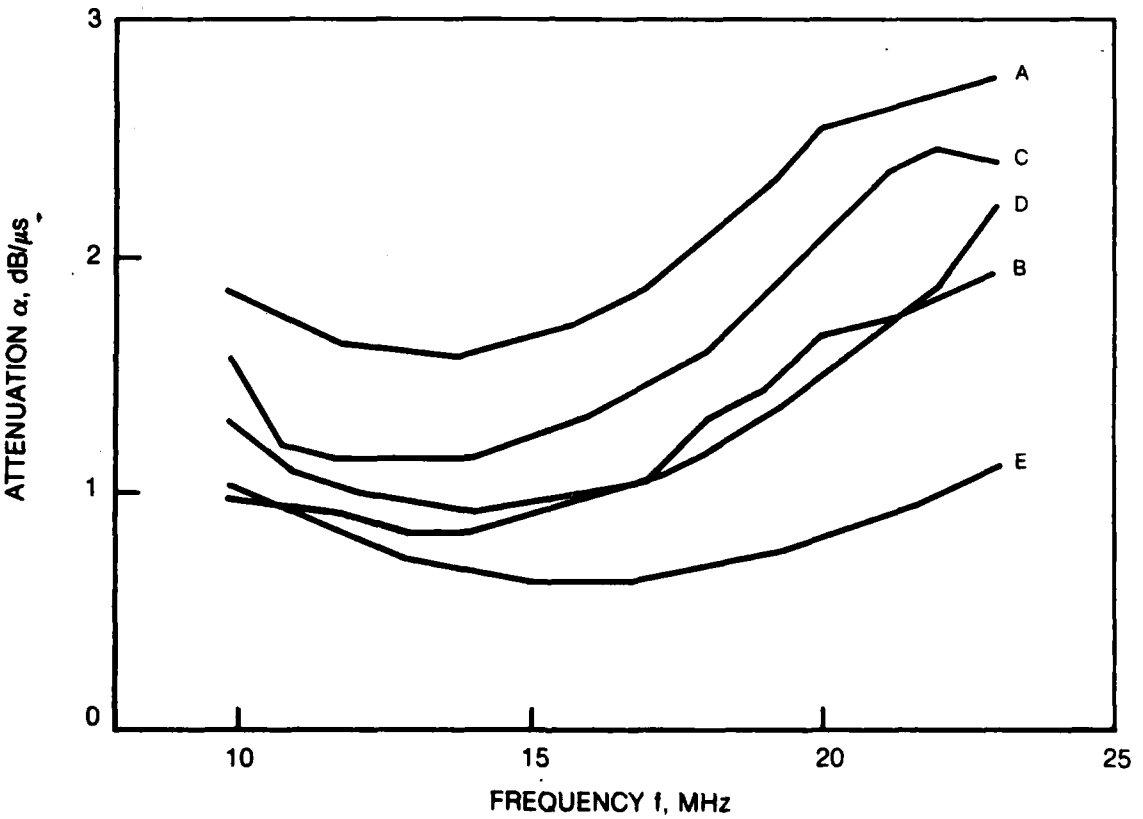
Specimen	Oxygen Content wt.%	Long Sound Velocity cm/sec
A	0.075	$(6.13 \pm 0.06) \times 10^5$
B	0.136	6.11 ± 0.06
C	0.194	6.02 ± 0.06
D	0.238	6.05 ± 0.06
E	0.290	6.11 ± 0.06

Attenuation-Frequency Spectra

Attenuation data in polycrystalline specimens is generally quite susceptible to errors arising from variations in the grain structure. These can include phase cancellation effects, which predominate when the ultrasonic wavelength λ is comparable to grain dimension D, and diffusion effects arising when $\lambda << D$. The major contributors to attenuation in the present regime ($\lambda >> D$) arise from lattice hysteresis and Rayleigh scattering.

The five specimens were examined over a frequency range of 10-20 MHz, with the results presented in Fig. 4. The attenuation levels are seen to vary considerably in the five specimens over the entire frequency range. To suppress any frequency independent effects, the slope of attenuation versus frequency was evaluated and is presented in Fig. 5. Data above 20 MHz is not reliable due to poor signal-to-noise levels. Below 20 MHz, some correlation can be seen between the slope of attenuation and the oxygen content. The slope decreases with increasing oxygen concentration. A, B, and C are not well resolved, while D and E show increasingly significant slope changes with frequency.

ATTENUATION AS A FUNCTION OF FREQUENCY FOR SAMPLES A-E



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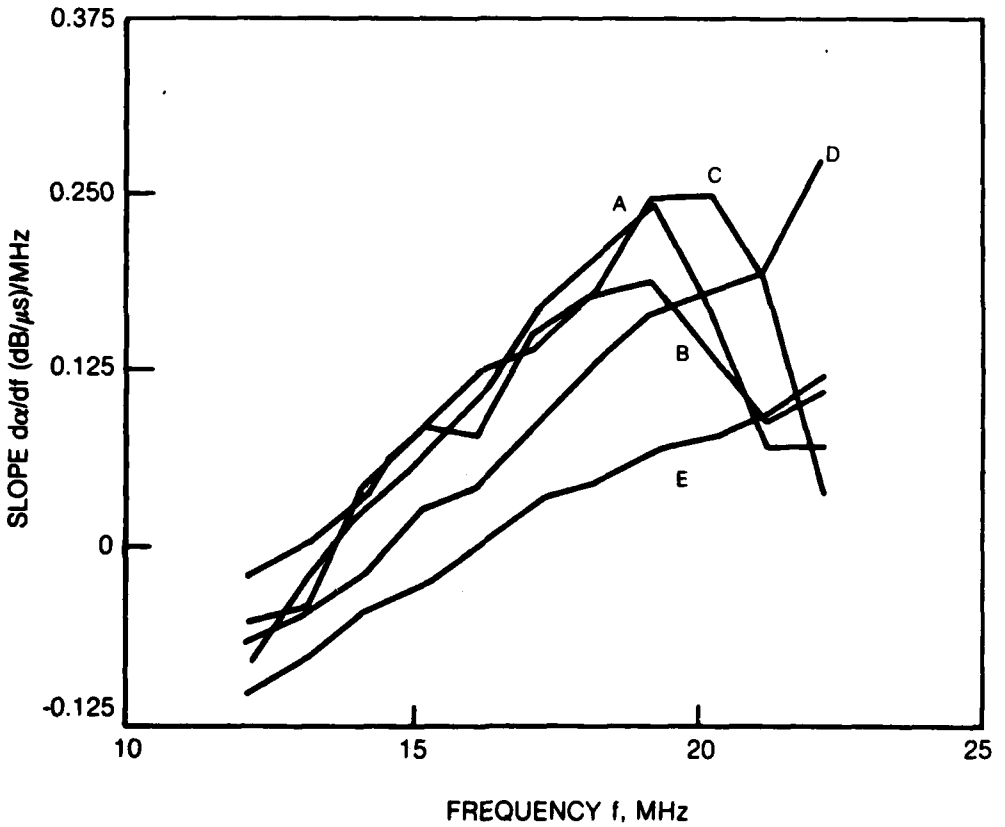
SLOPE OF ATTENUATION AS A FUNCTION OF FREQUENCY FOR SAMPLES A-E



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CONCLUDING REMARKS

Linear techniques, although demonstrating some correlation, appear to be more sensitive to measurement artifact errors arising from material inhomogeneities and, hence, demand greater care in data measurement and interpretation.

Nonlinear ultrasonic techniques showed an excellent correlation between deviation from the ideal, perfect material, second harmonic amplitude square-law behavior and the oxygen impurity concentration.

Furthermore, the excellent correlation obtained using a randomly chosen point for the data on each specimen suggests this technique shows promise in giving results independent of the above-mentioned artifact errors.

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