

7-1980

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Recommended Citation

Grayeli, N.; Ilić, D.; Stanke, F.; Kino, Gordon S.; and Shyne, J. C., "Acoustic Measurement of Microstructures in Steels" (1980). *Proceedings of the DARPA/AFML Review of Progress in Quantitative NDE, July 1978–September 1979*. 62.
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Abstract

The measurement of acoustic properties can be used for the nondestructive characterization of the microstructure of materials. We have measured the changes in longitudinal acoustic wave velocity and acoustic attenuation in steel specimens whose microstructure and properties differ widely because of differing compositions and heat treatment. The spatial variation of the relative acoustic velocity in standard Jominy end-quench hardenability test specimens was found to correlate very well with Rockwell C hardness scans, indicating a potentially practical method for measuring the hardening response of heat treated steel. Absolute velocity measurements on steel specimens were found to be subject to random scatter related to minor compositional variations; this limits the utility of absolute velocity measurements for microstructural NDE. Attenuation measurements have also been performed on steel samples with different microstructures. The measurement utilized broadband acoustic pulses corrected for transducer response, liquid buffer/solid specimen reflection, and diffraction effects. Attenuation coefficients were seen to be proportional to frequency squared for martensite and to the fourth power of frequency for pearlite. Higher attenuation was observed for pearlitic than for martensitic microstructures.

Keywords

Nondestructive Evaluation

Disciplines

Materials Science and Engineering

ACOUSTIC MEASUREMENT OF MICROSTRUCTURES IN STEELS

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ABSTRACT

The measurement of acoustic properties can be used for the nondestructive characterization of the microstructure of materials. We have measured the changes in longitudinal acoustic wave velocity and acoustic attenuation in steel specimens whose microstructure and properties differ widely because of differing compositions and heat treatment. The spatial variation of the relative acoustic velocity in standard Jominy end-quench hardenability test specimens was found to correlate very well with Rockwell C hardness scans, indicating a potentially practical method for measuring the hardening response of heat treated steel. Absolute velocity measurements on steel specimens were found to be subject to random scatter related to minor compositional variations; this limits the utility of absolute velocity measurements for microstructural NDE. Attenuation measurements have also been performed on steel samples with different microstructures. The measurement utilized broadband acoustic pulses corrected for transducer response, liquid buffer/solid specimen reflection, and diffraction effects. Attenuation coefficients were seen to be proportional to frequency squared for martensite and to the fourth power of frequency for pearlite. Higher attenuation was observed for pearlitic than for martensitic microstructures.

Acoustic methods can be applied to the nondestructive characterization of the microstructure of materials. This paper reports the initial efforts of our interdisciplinary program to explore microstructural acoustic NDE. The experimental materials were steels.

The properties of materials, especially mechanical properties such as tensile strength, hardness, and impact toughness, are strongly dependent on microstructural features like grain size and shape, the proportions and spatial distribution of the phases present, and macroscale heterogeneities in microstructure resulting from local compositional or thermal-mechanical processing variations. This is true of both metallic and ceramic materials. The microstructural character of materials and their related properties are usually assessed by microscopic examination or by direct measurement of properties; such procedures are often costly, and are necessarily destructive, requiring sacrificial example specimens.

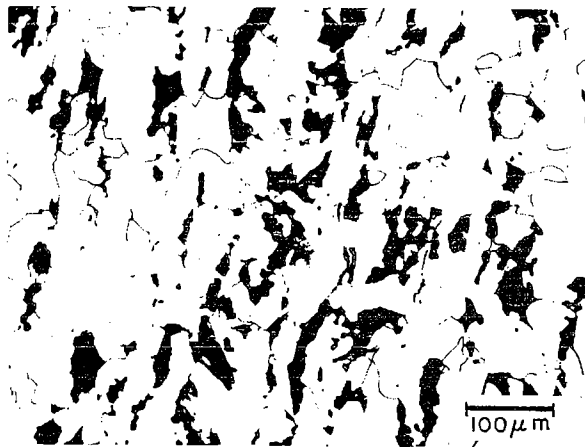
Steels are basically alloys of iron and carbon modified by minor additions of other elements. Steels exhibit remarkable variability of mechanical properties. These useful properties are dependent upon the rather complex microstructure of steel which can be closely controlled by proper selection of composition and heat treatment.

The dependence of steel microstructure on composition and heat treatment is illustrated in Fig. 1. Figures 1(a) and 1(b) compare the microscopic appearance of polished and etched steel specimens containing 0.2 and 0.6 wt. pct. carbon respectively; after annealing at 900°C and slow cooling in air to room temperature both steels consist of ferrite, the white grains, and pearlite, the darker constituent. Ferrite is essentially pure iron; pearlite is a mixture of two different crystalline phases (too finely dispersed to be resolved at the magnification shown), iron carbide (Fe_3C) and ferrite. Since virtually all the carbon resides in the pearlite, the proportion of pearlite is greater in the higher carbon steel. Figure 1(c) shows a steel with 0.4 wt pct carbon with a martensitic microstructure

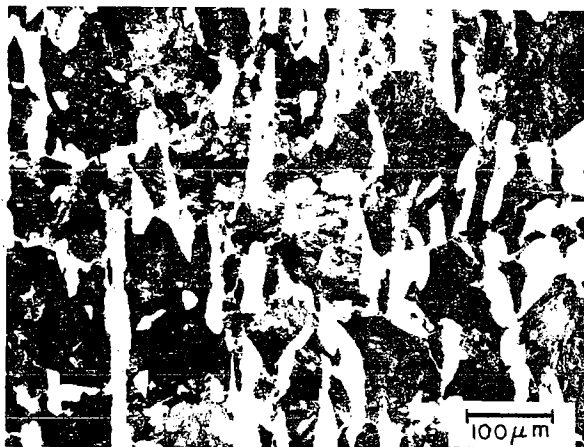
rather than pearlite and ferrite. This steel has been cooled rapidly in water after annealing at 900°C; the fast cooling causes the formation of martensite, a supersaturated solid solution of carbon in iron. Mechanical properties are strongly affected by the proportions of ferrite and pearlite and by the existence of martensite rather than pearlite and ferrite. Changes in microstructure such as these, enable steels to be heat treated to obtain optimum combinations of properties.

ACOUSTIC VELOCITY MEASUREMENT

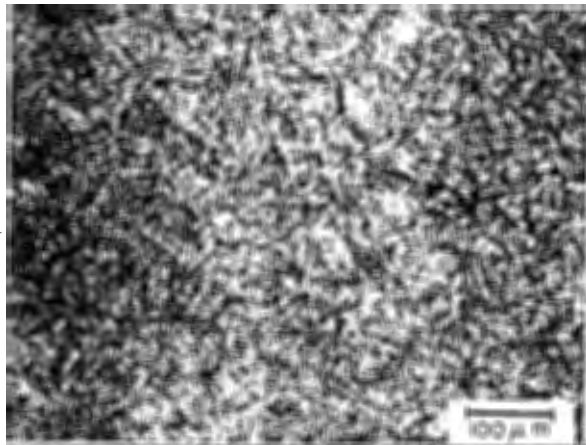
Our measurements of longitudinal acoustic wave velocity in metallic samples were made with a computer-controlled system developed for measuring acoustic velocity fields in solid samples immersed in a liquid buffer. The measurements use a two pulse-echo technique that cancels out the effects of the liquid buffer. The measuring method is illustrated in Fig. 2. Two sets of tone bursts several rf cycles long are transmitted, and their reflections from the specimen are received by a mechanically scanning commercial transducer. The delay between the two pulses is adjusted to overlap the back-face echo of the first pulse with the front-face echo of the second pulse, and this sum is gated out and detected. The product term is then used as an error signal in a phase-lock loop, which adjusts the frequency to keep the phase difference between the two echoes constant. Effectively, the measurement of phase change introduced by propagation through the specimen is converted to that of frequency, which can be performed with great precision. All of the above operations, including the mechanical scanning of the transducer, data collection, reduction, and display are controlled by a PDP 11-34 minicomputer. The resulting system precision is about 1 part in 10^6 of the measured longitudinal acoustic velocity. The accuracy of the absolute velocity measurement is 2 parts in 10^4 , as dictated by the accuracy in measuring the acoustic path length, the specimen thickness.



(a)



(b)



(c)

FIG. 1. Different steel microstructures resulting from difference in composition and heat treatment. 1(a): pearlite and ferrite in normalized 0.2 wt pct C steel; 1(b): pearlite and ferrite in normalized 0.6 wt pct C steel; 1(c): martensite in water quenched 0.4 wt pct C steel.

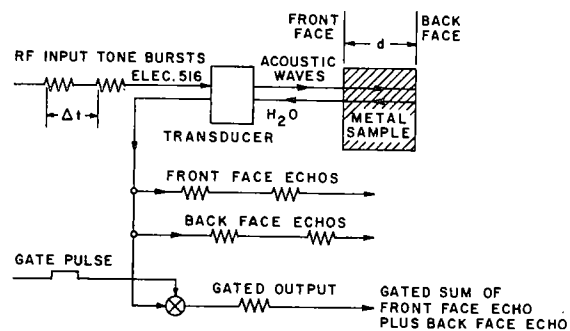


FIG. 2. Schematic of the precision acoustic velocity measuring system.

ABSOLUTE VELOCITY MEASUREMENTS

Specimens of 5 plain carbon steels were prepared by machining 1 cm thick flat plates oriented both longitudinally and transversely relative to the steel bar stock. Since the acoustic wave propagation direction was through the 1 cm plate thickness, acoustic velocity could be measured both parallel to and transverse to the rolling direction for each steel composition to detect the influence of any preferred orientation or crystal texturing. The compositions of the steels are shown in Table I. All specimens were given identical heat treatment; they were heated to 900°C and air cooled (normalized). This resulted in pearlite/ferrite microstructures, the pearlite varying from 12 to 100 volume pct over the range of carbon contents. After machining, the flat specimens were lapped to assure that their flat surfaces were parallel to within 2.5 μm.

TABLE I

Composition of Plain Carbon Steels used for Acoustic Velocity Measurements

Steel Type (AISI)	C	Mn	Si	P	S
1010	0.10	0.45	0.03	0.007	0.032
1020	0.20	0.52	0.21	0.009	0.028
1035	0.33	0.72	0.17	0.018	0.025
1060	0.55	0.80	0.19	0.012	0.023
1095	0.93	0.50	0.22	0.009	0.030

The resulting velocity measurements exhibited random scatter that totally obscured the variation in velocity anticipated from the variation in microstructure, Fig. 3. One can easily calculate an expected acoustic velocity from the known densities of pure iron ferrite¹ and Fe₃C,² and known elastic constant data for ferrite/Fe₃C mixtures.³ Increasing carbon should cause a decrease in the longitudinal acoustic velocity linearly proportional to carbon content; velocity should decrease

0.85 pct per wt. pct carbon in the steel, all other factors being identical. The scatter observed in the measured velocity was not caused by variations in preferred orientation; had there been any significant degree of preferred orientation, the longitudinal and transverse velocities would differ much more than the slight variations, which can be attributed to sample thickness variations, seen in Fig. 3. We believe that the random variations in such elements as manganese, silicon, sulfur and others always present in at least trace amounts, cause variations in density and elastic modulus and thus introduce random variations in acoustic velocity equal to or greater than the systematic effect of carbon in changing the microstructure. For example, we have calculated that the range of manganese contents in our specimens (see Table I) causes density variations large enough to obscure all the above change attributable to variations in microstructure. Therefore we conclude that absolute velocity measurements are not practical means for characterizing steel microstructure, because random compositional variations will confuse any attempt to calibrate absolute acoustic velocity with microstructure.

used to map out the microstructural pattern. Jominy end-quench test bars provide another example of steel objects containing microstructural gradients. We successfully used relative velocity measurements to survey quantitatively the microstructure of end quench test bars of several different alloy steel compositions.

The Jominy Test or End-Quench Test is the standard metallurgical quality control procedure for measuring the hardening response of heat treatable steels. The end-quench test specimen is a one-inch diameter round bar, four inches long. The bar is heated to about 850°C. It is then placed in a fixture and cooled by a jet of cold water impinged upon one end as shown in Fig. 4. This results in uniaxial heat flow toward the water-quenched end, and reproducible cooling rates that decrease with increasing distance away from the rapidly-cooled quenched end. The fast cooling rate at the quenched end causes the formation of hard, strong martensite; the slower cooled end transforms to softer, weaker pearlite or a pearlite and ferrite mixture, depending on composition. At intermediate locations mixed martensite/pearlite microstructures result from the intermediate cooling rates. The position of the transition from martensite to pearlite is a measure of the hardening response. The usual way of assessing the microstructural gradient along the length of an end-quench bar is to survey the Rockwell C hardness measured on flats ground along the side of the end quench test bar. This is possible because there is a pronounced hardness gradient caused by the microstructural gradient. Although surveying hardness is a great deal easier and less time consuming than directly observing the microstructure under a microscope, the hardness surveys are tedious because up to 60 individual manual hardness measurements may be required to survey a single test bar, a procedure taking about one hour.

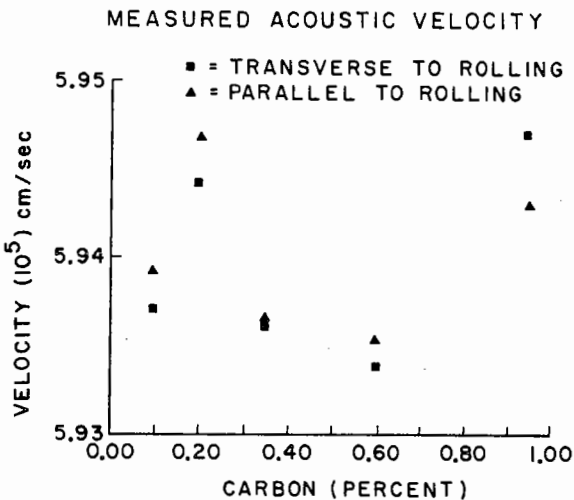


FIG. 3. Measured acoustic velocity in plain carbon steels of varying carbon content. The random scatter caused by minor fluctuations in alloying elements obscures the systematic variation in velocity related to the microstructure.

RELATIVE VELOCITY MEASUREMENTS

By relative velocity measurements we mean measurements of acoustic velocity at different locations within a given object. If the material composition is uniform within the object (as is usually the case), variations in acoustic velocity can be unequivocally related to microstructural variations from one location to another. There are many situations where microstructure is not uniform and the pattern of microstructural variation is important, e.g., in an induction hardened steel shaft heat treated to be martensitic on the outer surface but pearlitic in its interior. Velocity variation within a given object, i.e., relative velocity, could be

DIAGRAM OF A JOMINY TEST IN ACTION

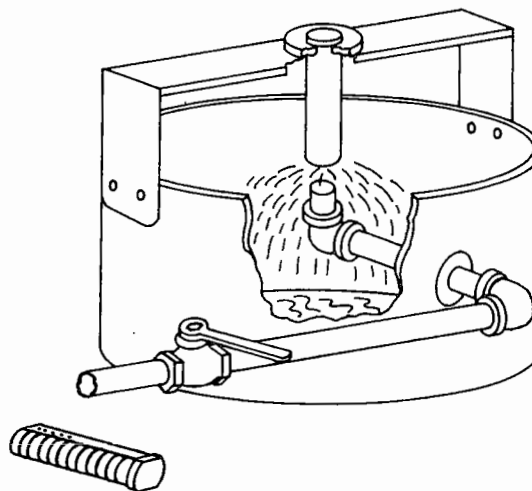


FIG. 4. End-quench test.

Figure 5 compares a conventional hardness survey with a longitudinal velocity scan of an end-quench test bar of AISI type 4140, a common, low alloy, heat-treatable steel. The longitudinal acoustic wave velocity was measured by the method described above

and illustrated in Fig. 2. The acoustic path was transverse to the axis of the test bar. Parallel flats were ground on opposite sides the full length of the test bar; the acoustic path was thus along bar diameters through the thickness of the bar between the ground flats. The relative acoustic velocity is plotted as $\Delta V/V_0$, the fractional change in velocity at any point relative to V_0 the velocity at the slower cooled, pearlitic end of the test bar. As seen in the plot the velocity decreased by about 0.7 pct at the martensitic, quenched end. The accuracy of the relative acoustic velocity measurement is limited by the uniformity of the thickness and the deviation from perfect parallelism of the ground flats. This is estimated to be 1 or 2 parts in 10^4 which is about 2% of the actual range of velocities measured. Thus the sensitivity and discrimination of the velocity measurement is equal to or better than that of the hardness measurements.

Figure 6 is a cross plot showing the relative velocity change $\Delta V/V_0$, versus the hardness. The correlation is virtually linear except for the extreme values. However, it is the intermediate values that are most important, because these occur at the region of transition from martensite to pearlite, the location critical to the measurement of hardening response.

The computer controlled automatic acoustic velocity scan requires only 4 minutes from start to finish with the data automatically plotted as $\Delta V/V_0$ vs position in the bar. This contrasts favorably with the hour or so required for a manual hardness survey. To our knowledge, this is the first time an end-quench test has been performed using a velocity scan rather than a hardness scan.

In addition to the 4140 steel end-quench test we have also performed similar acoustic velocity scans on end-quench test bars of types 52100, 4615, 8640, and 1095 steels with similarly encouraging results. We find these results persuasive that acoustic velocity measurements can be used to map out microstructural variations within steel objects.

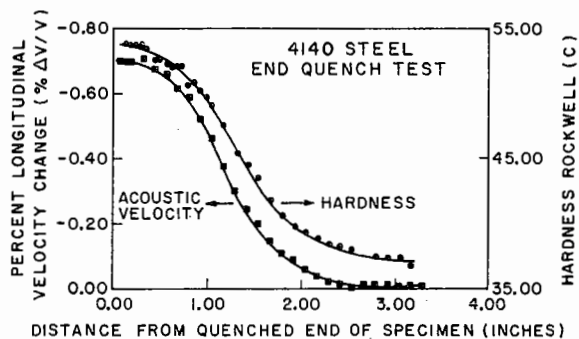


FIG. 5. End-quench test results measured in type 4140 steel. The two curves compare the microstructure gradient as surveyed acoustically and by conventional hardness measurements.

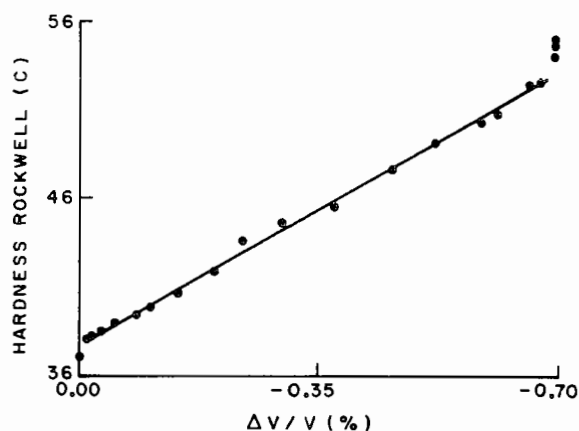


FIG. 6. Relative acoustic velocity vs hardness Rockwell C correlated for the data shown in Fig. 5.

ACOUSTIC ATTENUATION

In principle, attenuation measurements are more attractive than velocity measurements as the basis for acoustic NDE microstructural characterization. Where velocity is weakly a function of microstructure, acoustic attenuation is strongly affected by microstructure because of scattering at grain boundaries, second phase particles and other microstructural features.⁴ Moreover, the strong frequency dependence of the acoustic attenuation coefficient can provide additional information related to microstructure.

This paper reports only our earliest, preliminary efforts to characterize microstructure by means of acoustic attenuation measurements.

A computer interfaced system has been developed for quickly and easily measuring the attenuation of acoustic waves traveling through metal samples. The same samples used for the velocity measurements can be used for attenuation measurement. A commercial ultrasonic transducer launches longitudinal waves through a water bath at normal incidence to the sample and then receives the echoes. Normal incidence is attained by swiveling the transducer to maximize the amplitude of the front face echo. A narrowband pulse (i.e., a tone burst with at least 10 rf cycles) must be used for this alignment because the various components of a broadband pulse are not equally affected by non-normal incidence and therefore the maximum is difficult to determine. Computer controlled digital stepping motors can also be used to move the transducer for spatial scanning.

The three-echo method is used to determine the attenuation, as described by Papadakis.⁴ This method uses the measured amplitudes of three echoes to solve the equations for these echoes for any of the three unknowns in the system. In our experiment, these unknowns are the pulse-echo frequency response of the system, the reflection coefficient at the sample-water interface, and the attenuation through the sample. Broadband pulses are used to obtain

information over the frequency range of an octave or more. The front face echo and first two back face echoes are digitized and then stored in a computer. Since the dynamic range of these echoes can be 40 dB or more, many samples of each echo are averaged to improve the signal-to-noise ratio. The echoes are gated and separated in the computer, and the moduli of their Fourier transforms are computed by an FFT program. Figure 7 shows three gated, averaged pulses with their frequency spectra superimposed for a typical sample. The spectral components of the three echoes at each frequency are compared in the three-echo method to calculate the attenuation through the sample, correcting for transducer response and also for reflection and diffraction losses. Figure 8 illustrates the importance of including the diffraction correction for the echoes shown in Fig. 7. The scales are logarithmic, log dB/cm versus log f , so that the slopes of the linear plots give the powers of the frequency dependence. These curves are plotted by a computer and an analog plotter by simply connecting 128 attenuation data points within the passband. No curve fitting techniques have been used. One measurement produces a large amount of attenuation information about the sample.

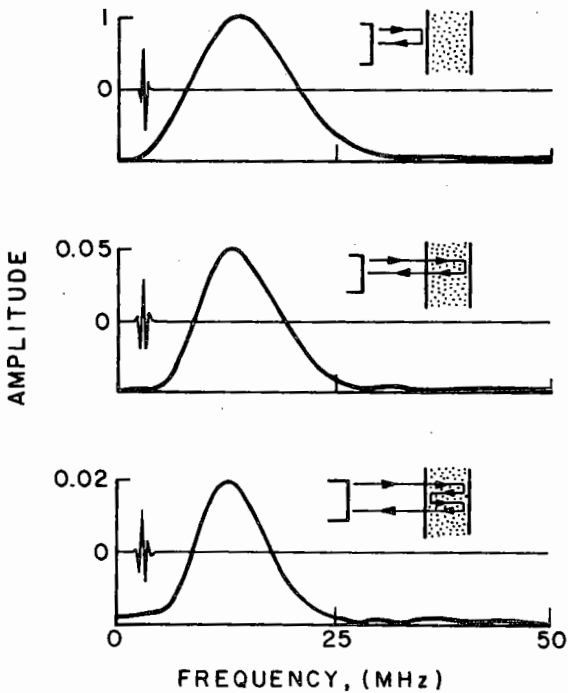


FIG. 7. Three echoes of the same broadband pulse (plotted in both the time and frequency domains) used to obtain the attenuation coefficient in the specimen for the range of frequencies in the pulse.

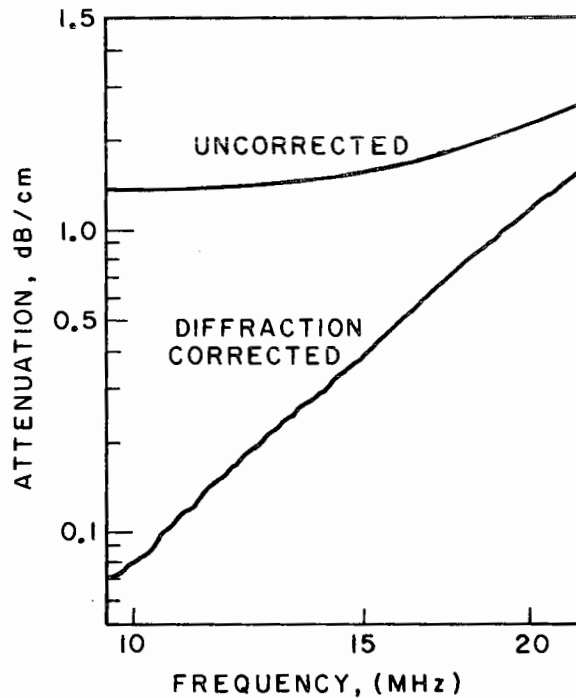


FIG. 8. Attenuation vs frequency for a steel sample illustrating the importance of corrections for diffraction.

Figure 9 shows the attenuation in two different steel samples with identical composition but

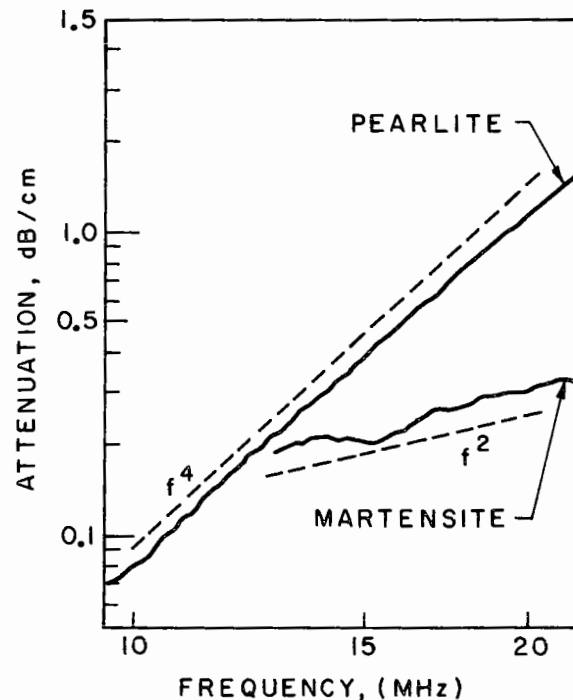


FIG. 9. Attenuation vs frequency for two specimens of the same steel heat treated differently to have martensite in one but pearlite in the other.

different microstructure. The curve for pearlite shows a frequency dependence very close to f^4 indicating that Rayleigh scattering is the dominant mechanism for attenuation. The curve for martensite exhibits lower values of attenuation, with a frequency dependence nearer to f^2 , indicating a different mechanism for attenuation from that in martensite. These results are comparable to previous results by Papadakis.⁵

CONCLUSIONS

1. Absolute acoustic velocity measurements are subject to random variations that limit their utility for microstructural NDE.
2. Relative velocity change measurements can be used to map microstructure with precision in a given piece of steel.
3. Acoustic attenuation measurements are very sensitive to microstructure, and they have good potential for practical, microstructural NDE.

ACKNOWLEDGEMENTS

This work was sponsored by the Air Force Office of Scientific Research under Grant AFOSR78-3726. The experimental steels were provided by the U.S. Steel Corporation.

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SUMMARY DISCUSSION
(N. Grayeli)

John Schuldies (Airesearch): Was the jominy test bar sectioned up into disks, essentially, and the velocity measurement made on the disk?

N. Grayeli: No.

John Schuldies: Was it a radial measurement, then?

N. Grayeli: Two parallel surfaces are made on the jominy bar, and acoustic wave is sent into the sample, perpendicular to the bar axis, and we scan along the sample.

John Schuldies: So, it is a radial velocity measurement time of flight through diameter of the bars?

N. Grayeli: I really don't understand what you mean.

Gordon Kino (Stanford University): It's a flat bar. You scan along the length of the bar through its thickness.

N. Grayeli: Sure.

Unidentified Speaker: You are measuring across that bar.

Gordon Kino: It's a standard kind of thing.

Don Yuhas (Sanoscan): Did I read that attenuation curve right on pearlite and martensite? The number I got was something like one db per centimeter at 20 megahertz.

N. Grayeli: Maybe I can ask my colleague.

Gordon Kino: We have increased the frequency to 50 megahertz because of this and we were quite surprised. As you look back at Papadakis' work, indeed he saw these low attenuations.

Unidentified Speaker: The structure with 100 percent of pearlite is very good two-phase material. Did you measure anything of the frequency-dependent velocity at any higher frequency? 15 and 20 megahertz?

N. Grayeli: No, we didn't.

Unidentified Speaker: It's to be expected?

N. Grayeli: We don't know.

John Duke (Virginia Tech): You know, if you measure -- I was interested in -- the gentleman didn't follow up on his question. In the jominy bar, in quenching it in that particular fashion it's unlikely that you probably have uniformity of hardness throughout the diameter perpendicular to the faces that you ground. The measurement that you're making I think is probably characterizing the character of the material better than in fact the hardness test is because it's a bulk measurement. The hardness test, of course, is a surface measurement, and you, of course, would expect if you were to plate something with aluminum, and the hardness of the aluminum would be independent, necessarily, of the material which is deposited. So, I think, you know, you could make a lot more out of this than comparing it to hardness measurements.

N. Grayeli: Yes, it is a good comment in general, but in the case of a jominy bar, the structure is uniform throughout the diameter perpendicular to the faces on which we ground.

J. White (Westinghouse): Did you look at grain size in terms of the relationship on frequency dependence of attenuation?

N. Grayeli: That's our next step.

J. White: It's quite a strong relationship.

N. Grayeli: I think so.

(continued)

N. Grayeli (continued discussion)

Albert Birks (Battelle Northwest): In that respect there's a rather definitive Japanese study looking at grain size. And if you don't look at the substructure within the grains, the correlation is not very good.

N. Grayeli: Yes, it is true, but the effect of substructure is not very much, and I think to get a better correlation we have to consider that part which comes from substructure inside the grain.

P. Holler: One question. How do you calculate the influence of manganese on the velocity?

N. Grayeli: We use the data from the Barrett and Massalski (structure of metals) to calculate the velocity change by addition of Mn and other alloying elements. The change in lattice constant, caused by Mn will change the density and the density change, will change the velocity in the materials.

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