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## RESONANT BEAM AND ULTRASONIC METHODS FOR EVALUATION OF SINTERED POWDER STEEL COMPACTS

Technical Report

R. H. Brockelman

Date 20 May 1966

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### INVESTIGATION OF RESONANT BEAM AND ULTRASONIC METHODS FOR THE EVALUATION OF SINTERED POWDER STEEL COMPACTS

Technical Report

R. H. Brockelman

<u>DA PROJECT TITLE</u>: Investigation of the Application of Resonant Beam Methods and Ultrasonic Methods of Evaluating Sintered Components

DA PROJECT NO: AW-5-15221-01-AW-M6

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#### ABSTRACT

Results of an investigation designed to determine the practicability of evaluating the mechanical properties of sintered steel powder compacts by ultrasonic and mechanical resonance technique are described. The yield and tensile strengths of sintered prealloyed steel are correlated with sintered density, resonant frequency, sonic and ultrasonic velocity, as well as C-scan ultrasonic transmission recordings. The correlations that have been established demonstrate the effectiveness of sonic and ultrasonic methods for the nondestructive evaluation of sintered powder metals. The experimental procedures are described and the capabilities and limitations of each technique are discussed.

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#### SUBJECT

Sonic Testing of Sintered Steel Powder Compacts.

#### OBJECTIVE

To determine the feasibility of evaluating the mechanical properties of sintered steel powder compacts by sonic techniques.

#### CONCLUSIONS

1. The strength of sintered powder steels generally increases with density; however, a range of strengths is produced at any equivalent density by varying the processing parameters. Therefore, the sintered density is ineffective in accurately predicting the strength of sintered powder metals where the processing parameters are subject to change.

2. The mechanical resonant frequency provides a better correlation of strength than the sintered density in steel powder compacts of uniform density and/or interparticle bonding.

3. When the log of the tensile strength is plotted as a function of the longitudinal resonant frequency, a straight-line relationship is obtained for both the SAE 4630 and SAE 4650 steel powder tensile bars.

4. Localized areas of low density and/or interparticle unbond can alter the strength of a compact considerably and, depending upon their location and size, may not appreciably affect its resonant frequency.

5. A recently developed ultrasonic imaging system depicts local variations in sound transmission in sintered steel powder compacts. These local variations correlate with the rupture points of the tensile bars which did not adhere to the resonant frequency-strength relationship.

6. Point by point, ultrasonic velocity measurement detects the presence of inhomogeneities in sintered steel compacts and coincides with ultrasonic imaging results to pinpoint their location.

7. Immersion pulse-echo techniques are preferable to direct contact methods for ultrasonic velocity measurement. The tensile strength-ultrasonic velocity relationship, for sintered steel tensile bars (ultrasonic velocity measured by the immersion pulse-echo technique), compares favorably with the tensile strength-resonant frequency relationship for the same tensile bars.

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#### CONCLUSIONS - Continued

8. Since the properties of sintered steel compacts vary with direction of measurement, it is necessary that comparative ultrasonic velocity measurements always be made in the same relative direction with respect to the pressing direction. Also, each material must be considered individually when the results of velocity measurements are examined.

9. The use of dry couplants, such as plastic tapes, appears promising for ultrasonic velocity measurement.

#### RECOMMENDATIONS

1. The effect of complex geometry on the resonant beam method should be evaluated so that the practicability of this method can be determined.

2. Improved techniques should be developed for the accurate measurement of ultrasonic velocity in production components, with emphasis placed on investigating materials and techniques for use in a dry couplant method. Methods of making more accurate time measurements should also be explored.

3. The ultrasonic imaging instrument should be utilized for the detection of local variations in production components of complex shape.

#### 1. INTRODUCTION

This work represents an extension and continuation of a preliminnary investigation conducted in 1Y 65 and reported previously. (1) In that investigation, the feasibility of utilizing various nondestructive sonic techniques for the testing and evaluation of sintered iron powder compacts was examined. Two sonic methods, mechanical resonance and ultrasonics, were established as more sensitive than the sintered density to factors that apparently have a direct effect on the strength of sintered iron powder compacts.

The resonance method provides an accurate, rapid measure of the natural resonant frequency. This frequency, or the velocity of sound propagation calculated from the resonant frequency, correlates very closely with the tensile strength of compacts of simple shape and is sensitive to strength variations caused by the change of the processing parameters of powder method of manufacture. The parameters varied were the compacting pressure, sintering time and temperature, and surface condition of the powder prior to compaction. The resonance method averages the entire component properties and, possibly, creates a disadvantage in the testing of components of irregular shape where the distribution of porosity is not always uniform.

Longitudinal ultrasonic velocity can also be correlated with tensile strength. However, since the properties of sintered metal powders tend to vary with direction of measurement, it is necessary that comparative ultrasonic velocity measurements always be made in the same relative direction with respect to the pressing direction. Ultrasonic velocity measurements have the advantage of being localized since the area under test is determined by the size of the transducer. This makes it possible to examine critical sections or complex parts that may be impossible to evaluate by resonance techniques.

The subsequent reported effort was conducted to determine the practicability of evaluating the mechanical properties of sintered steel powder by the same sonic methods. Sintered steel would normally be selected for powder metal applications where high strength is of major importance. However, for the preliminary investigation, iron powder was used to eliminate effects that variations of composition and cooling rate could have on the mechanical properties of steel.

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#### 2. TEST PRINCIPLES

The fact that mechanical properties of sintered metal products are not generally equivalent to those obtained by conventional metallurgical processes can be mainly attributed to the predominant effects of inherent porosity. Presently, measurement of the density or the overall volume of the inherent porosity represents the principal nondestructive criterion of sintered metal powder quality. However, the nature or character of this porosity, which includes the shape and distribution of the pores, is a variable dependent upon the processing parameters and can also significantly affect the mechanical properties of sintered metal powders. In the previous investigation of sintered iron powder, the tensile strength was observed to be closely related to the shape of the pores; it increased as the pores approached more spherical shapes with increased time and temperature of the sintering treatment.

The use of sonic techniques as potential test methods for sintered powder metals is based on the theoretical relationship between the velocity of sound propagation in a material and its elastic modulus. (2) A number of theoretical and experimental investigations have shown that the elastic modulus of a porous material is related to the shape and distribution of porosity in addition to the total pore volume. (3), (4), (5) As the pores approach more regular shapes, the elastic modulus increases. Since sonic velocity is a function of the elastic modulus, any change in the modulus should be reflected in sonic measurements. Therefore, the dependency of the elastic modulus on the character of the inherent porosity, which, to a large extent, determines the mechanical properties of a sintered compact by its amount, shape, and distribution, indicates an interrelation between sonic velocity and mechanical properties.

#### PROCEDURE

#### a. Test Specimens

ASTM standard, flat, powder metal, tension test bars 3-1/2 inches long were prepared from both SAE 4630 and SAE 4650 prealloyed steel powder. All the tensile bars were densified by pressing at 20 tsi and sintered in an endothermic gas atmosphere for one hour at  $2050^{\circ}$ F. By re-pressing the tensile bars at 25, 33 and 46 tsi and resintering in an endothermic gas atmosphere for periods of 1/2 and two hours at a temperature of  $2050^{\circ}$  F, test specimens with a variety of densities and strengths were produced. Density was determined for each specimen by the standard volume and weight measurement technique. The maximum sintered densities achieved by this processing were somewhat lower than had been desired. This was undoubtedly because of the poor compressibility of pre-alloyed steel powder as compared to mixing alloying ingredients with the

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#### 3. PROCEDURE - Continued

powder and relying on homogenization by diffusion during sintering. Pre-alloyed steel powder was used in order to eliminate the effects that incomplete alloying of premixed powders could have on strength properties.

#### b. Resonant Frequency

The fundamental longitudinal and transverse resonant frequencies of each tensile bar were measured with the use of commercially available equipment based on the floating-beam method. With this instrument, the specimen is supported on fine wires and excited by piezoelectric or electromagnetic transducers. A frequency spectrum is then scanned. At the resonant frequency of the specimen, the amplitude of vibration becomes a maximum, and this frequency is displayed by a high-precision counter. The method is simple and free from experimental uncertainties for specimens of simple shape.

The fundamental longitudinal resonant frequency is related to the thin-rod sonic velocity by Vo =  $2 \times 1 \times f$ , where Vo = thin-rod velocity in inches per second, l = length of sample in inches, and f = the longitudinal resonant frequency in cycles per second. Although the sonic velocity is of basic importance, it is apparent from this relationship that, if the length is constant, the velocity is directly related to the resonant frequencies. Therefore, the resonant frequencies may be compared directly with the relative properties.

#### c. Ultrasonic Velocity

Ultrasonic velocity measurements were made by both the throughtransmission and pulse-echo techniques. In the through-transmission technique, crystal transducers are coupled to the opposite faces of the sample and the time for the pulse to be transmitted through the section between the transducers is measured by a calibrated sweep delay of an oscilloscope. The bulk sonic velocity can then be calculated from  $V_L = T/t$ , where  $V_L$  = bulk velocity in inches per second, T = thickness of sample between transducers in inches, and t = pulse travel time in seconds. In the pulse-echo technique, a crystal transducer is coupled to the surface of the sample and the time interval between successive reflected pulses is measured by a calibrated sweep delay of an oscilloscope. The bulk sonic velocity can then be calculated from  $V_L = 2T/t$ , where  $V_L =$  bulk velocity in inches per second, T = sample thickness in inches, t = measured time interval between reflected pulses in seconds.

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#### 3. PROCEDURE - Continued

Water immersion, oil contact, and plastic contact techniques were investigated to ascertain relative sensitivity and simplicity of velocity measurement on porous metals.

#### d. Ultrasonic Attenuation

Immersion pulse-echo techniques were used to measure at 5 megacycles the ultrasonic attenuation of longitudinal waves at points along the length of the steel test specimens. These measurements were both parallel and perpendicular to the compacting direction. Ultrasonic attenuation was found to vary considerably along the length of the test specimens. This variation indicated nonuniform interparticle bonding or nonuniform distribution of internal cavities.

Ultrasonic imaging equipment was utilized to produce C-scan recordings of the powder steel tensile bars. This equipment produces a representation of the component which indicates the amount of ultrasonic energy transmitted through the material as lighter or darker points. This imaging method is easier to use and the results easier to interpret than the immersion pulse-echo technique for depicting local variations in density and/or interparticle bonding. The recordings were visually related to the mechanical rupture point of each tensile bar.

#### 4. RESULTS AND DISCUSSION

#### a. Resonant Frequency and Ultrasonic Imaging

The yield and tensile strengths as functions of the sintered density for the SAE 4630 and SAE 4650 steel powder tensile bars are shown in Figures 1 and 2. Both the yield and tensile strengths increase with sintered density for the specimens subjected to the same sintering time, and generally increase with sintering time for specimens of equivalent density. If different sintering temperatures had been introduced into the processing procedure, an even wider range of strengths at equivalent density would have been obtained. This demonstrates why the sintered density is ineffective in accurately predicting the strength of sintered metals where the processing parameters are subject to change.

The yield and tensile strengths are plotted against the longitudinal fundamental resonant frequency of the same tensile bars as shown in Figures 3 and 4. The correlations are better than those of the sintered density and strength shown in Figures 1 and 2. The transverse resonant frequency mode yielded similar results and could also be used in the evaluation of tensile bars.

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#### 4. RESULTS AND DISCUSSION - Continued

If the tensile strength-resonant frequency data are plotted semilogarithmically, with the tensile strength on the log scale, as shown in Figures 5 and 6, linear relationships are obtained. These data are similar to the test results obtained on the sintered iron powder compacts. For the sintered iron compacts, a linear relationship was observed between the velocity of sound propagation, calculated from longitudinal resonant frequency, and the log of the tensile strength.

The fact that the strengths of a number of the test bars (numbered in Figures 5 and 6) fall conspicuously below the straight-line relationships can be attributed to localized areas of low density and/or interparticle unbound. Since the resonant frequency is a function of the entire component properties, a weak area of an otherwise uniform compact could alter the strength of the compact considerably and, depending upon its location and size, not appreciably affect its resonant frequency. Thus, the importance of a method capable of detecting nonuniformities in sintered compacts is realized. These nonuniformities, which probably originate during the pressing process, were not evident in the previous investigation in which all the sintered iron compacts conformed closely to the resonant velocitystrength relationship. This result is anticipated when the relative ease of compacting iron powder as compared to prealloyed steel powder is considered.

Local variations in sound transmission were shown in the C-scan recordings of a number of the tensile bars. The broken tensile bars and their corresponding recordings for the tensile bars (numbered in Figures 5 and 6) whose strengths are noticeably lower than those predicted by resonance measurements are shown in Figure 7. Local variations in sound transmission correlate with the rupture points of the tensile bars.

#### b. Ultrasonic Velocity and Imaging

Immersion pulse-echo techniques were investigated and found to be preferable to direct contact methods for ultrasonic velocity measurement. Optimum results were obtained by submerging in water only the transducer and the surface of the specimen facing the transducer. Ultrasonic velocities were measured by this method through the thickness, point by point, along the length of an SAE 4630 steel powder tensile bar. Velocity variations (Figure 8) are evident along the length of the bar, indicating areas of nonuniform distribution of

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#### 4. RESULTS AND DISCUSSION - Continued

internal cavities and/or interparticle bonding. The broken tensile bar and its C-scan recording are also shown for purposes of comparison. Both ultrasonic velocity and transmission measurements indicate the presence of inhomogeneities. Furthermore, their locations coincide with the point of lowest ultrasonic velocity, which, in turn, correlates with the rupture point of the bar. Ultrasonic velocity measurements were also made by this method through the thickness of each SAE 4650 steel tensile bar at the center of its gage length, where the smallest reduced section is located. No attempt was made to obtain, point by point, velocities on these bars. The tensile strength-ultrasonic velocity data plotted semilogarithmically with the tensile strength on the log scale are shown in Figure 9. A straight-line relationship is obtained which compares favorably with Figure 6, the tensile strengthresonant frequency relationship for the same tensile bars. The tensile bars that depart from this relationship are the same bars shown in Figure 6 that ruptured at detectable inhomogeneities outside their smallest reduced cross-sectional area.

The through-transmission contact technique was successfully applied to the measurement of velocity through the length of four-inch steel powder bars. This technique affords a method of measuring velocity without depending upon a multiple of reflected pulses. This is of importance in testing some sintered metal powder compacts where excessive scattering of the pores renders pulse-echo techniques usable.

#### c. Anisotropic Ultrasonic Velocity and Microstructure

The ultrasonic velocity and transverse resonant frequency were higher when measured in the pressing direction, which is contrary to what was observed in the sintered iron powder. A microstructural examination of the pore structure of the sintered steel specimens revealed the reasons for their anisotropic behavior and the apparent disagreement as to what was observed in the sintered iron. The microstructure of a SAE 4650 steel powder compact is shown in Figure 10. Apparently the prealloyed SAE 4650 steel powder is nearly spherical in shape and remains practically undeformed after the pressing operations because of its rigid structure. The powder particles are bonded in the pressing direction, which accounts for the higher velocity and frequency in this direction, with porosity aligned nearly parallel to the pressing direction around the sperical powder particles. The rigid powder structure and the limited ability of a powder compact to transmit pressure in a lateral direction produce this type of structure (Figure 10).

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#### 4. RESULTS AND DISCUSSION - Continued

In contrast the porosity of the sintered iron compacts, after compaction, was aligned perpendicular to the pressing direction around the powder particles. These particles were elongated normal to the pressing direction. Anisotropy was larger in the SAE 4650 steel compacts than in the SAE 4630 steel compacts.

The microstructure of an SAE 4630 steel powder compact is shown in Figure 11. This type of structure could have been produced by pressing powder which was either spherical in shape and deformed by the pressing operations or initially of irregular shape. Although the structure appears more uniform than that of the SAE 4650 steel compact, its alignment of porosity nearly parallel to the pressing direction around irregularly shaped powder particles also gives it anisotropic features. These results emphasize the necessity for making comparative ultrasonic velocity measurements in the same relative direction with respect to the pressing direction, as well as the necessity for considering each material individually, when the results of such measurements are examined.

#### d. Ultrasonic Coupling Methods

Various ultrasonic coupling methods for sintered metals were examined when these methods were determined to exert an influence on the accuracy and simplicity of velocity measurement and also to affect the properties of a component so as to impede its future usefulness. Any wet-coupling media, such as oil or water, tend to infiltrate the pores of a sintered powder compact. The consequences of this infiltration are numerous. For example, an oil couplant used in a direct contact test is almost immediately exhausted by the capillary action of the pores. A water couplant, used in the immersion techniques, can fill the pores and internally corrode an iron or steel compact. Also, it is known that the presence of either media in the pores alters the ultrasonic velocity. However, quantitative effects were not established. Two methods of preparing the surface of a compact to prevent the penetration of a wet-coupling media were used in the previous investigation to establish a relationship between ultrasonic and resonant velocity. Grinding the specimen faces or coating them with a chemically inert silicone grease impedes the infiltration of oil or water couplants. However, these methods may be impractical from an economic viewpoint or impossible to use when complex components are to be tested. Therefore, the use of a dry couplant, which eliminates the infiltration problem, was briefly examined. A vinyl plastic tape with a pressure-sensitive adhesive on one face was attached directly to the crystal-transducer face. This composite was then calibrated on a piece of wrought steel for use as the standard velocity of steel. Calibration was necessary to determine the exact time required for an ultrasonic pulse to transverse the thickness of the tape. This time is subtracted from the total measured time to give the time that is used with the specimen thickness to calculate velocity. The results obtained by this method were encouraging and

#### 4. RESULTS AND DISCUSSION - Continued

justify additional effort in this direction. Other materials, which have good coupling characteristics and wear properties, should be investigated for possible use as dry couplants.

#### **APPENDICES**

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A - Illustrations (11)

B - Literature Cited

C - Distribution

APPENDIX A

#### ILLUSTRATIONS

- Figure 1 Yield and Tensile Strengths as Functions of the Sintered Density for the 4630 Steel Powder Tensile Bars.
- Figure 2 Yield and Tensile Strengths as Functions of the Sintered Density for the 4650 Steel Powder Tensile Bars.
- Figure 3 Yield and Tensile Strengths as Functions of the Longitudinal Resonant Frequency for the 4630 Steel Powder Tensile Bars.
- Figure 4 Yield and Tensile Strengths as Functions of the Longitudinal Resonant Frequency for the 4650 Steel Powder Tensile Bars.
- Figure 5 Relation Between Log Tensile Strength and Longitudinal Resonant Frequency for 4630 Steel Powder Tensile Bars.
- Figure 6 Relation Between Log Tensile Strength and Longitudinal Resonant Frequency for 4650 Steel Powder Tensile Bars.
- Figure 7 Correlation Between Ultrasonic Transmission and Point of Rupture of Sintered Steel Tensile Bars. (Numbers refer to specimens singled out in Figures 5 and 6).
- Figure 8 Correlation Between Ultrasonic Transmission, Velocity, and Point of Rupture of 4630 Sintered Steel Tensile Bar.
- Figure 9 Relation Between Log Tensile Strength and Ultrasonic Bulk Velocity, Measured by the Immersion Pulse Echo Technique on 4650 Steel Powder Tensile Bars.
- Figure 10 Photomicrograph of Sintered 4650 Steel Powder Compact. Pressed at 20 tsi (arrow indicates pressing direction), Sintered for One Hour at 2050° F, Repressed at 25 tsi, Resintered for 1/2 Hour at 2050° F, Sintered Density -5.96 g/cc, Unetched, 100X.
- Figure 11 Photomicrograph of Sintered 4630 Steel Powder Compact. Pressed at 20 tsi (arrow indicates pressing direction), Sintered for One Hour at 2050° F, Repressed at 25 tsi, Resintered for 1/2 Hour at 2050° F, Sintered Density -6.05 g/cc, Unetched, 100X.

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APPENDIX A

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APPENDIX A





FIGURE 2 - Yield and Tensile Strengths as Functions of the Sintered Density for the 4650 Steel Powder Tensile Bars.





FIGURE 4 - Yield and Tensile Strengths as Functions of the Longitudinal Resonant Frequency for the 4650 Steel Powder Tensile Bars.

REPORT SA-TR19-1521 55-50 -45-40-. . TENSILE STRENGTH, 1000 PSI-35\_ 0 0< 30-SEE FIGURE 1 FOR KEY TO SYMBOLS 25 20 21,250 21,750 22,750 23,250 20,250 20,750 22,250 19,750 LONGITUDINAL RESONANT FREQUENCY, CYCLES PER SEC



APPENDIX A







Figure 7. Correspondence Between Point of Rupture of Sintered Powder Steel Tensile Bars (Nos. 1, 2, and 3, in Figures 5 and 6) and C-Scan Ultrasonic Transmission Patterns.

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Figure 10. Photomicrograph of Sintered 4650 Steel Powder Compact. Pressed at 20 tsi (Arrow indicates pressing direction), Sintered for 1 Hour at 2050°F, Repressed at 25 tsi, Resintered for 1/2 Hour at 2050°F, Sintered Density -5.96 g/cc, Unetched, 100X.



Figure 11. Photomicrograph of Sintered 4630 Steel Powder Compact. Pressed at 20 tsi (Arrow indicates pressing direction), Sintered for 1 Hour at 2050°F, Repressed at 25 tsi, Resintered for 1/2 Hour at 2050°F, Sintered Density -6.05 g/cc, Unetched, 100X. APPENDIX B

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