# Ultrasonics: A Technique of Material Characterization

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## 1. Introduction

The material science and characterization is a field concerned with inventing new materials and improving previously known materials by developing a deeper understanding of properties under different physical conditions. The properties of materials depend upon their composition, structure, synthesis and processing. Many properties of materials depend strongly on the structure, even if the composition of the material remains same. This is why the structure-property or microstructure property relationships in materials are extremely important.

On the basis of different physical properties, the materials are classified mainly into five categories: (a) metals and alloys, (b) semi-metals and semiconductors, (c) ceramics, glasses and glass-ceramics, (d) polymers, and (e) composite materials. Functional classification of materials includes aerospace, biomedical, electronic, energy and environmental, magnetic, and optical (photonic) materials. The structural classification of materials are of two types as (a) crystalline (single crystal and polycrystalline), and (b) amorphous.

The selection of a material and the potential to be manufactured economically and safely into useful product is a complicated process. It requires the complete knowledge of constituent material not only after production but also in processing. Increased competition and need of higher productivity and better products from material producing industries are creating more stringent requirements for process and quality control. This demands the characterization of materials. The topic material characterization essentially includes the evaluation of elastic behaviour, material microstructure and morphological features, associated mechanical properties etc. The destructive, semi-destructive and non-destructive testing (DT & NDT) techniques are available for the complete characterization. These characterization techniques are the basic tool for the quality control and quality assurance of the material or component or product.

Ultrasonics, which is a sub category of acoustics deals with acoustics beyond the audio limit. The application of ultrasonics falls into two categories as high frequency- low intensity and low frequency – high intensity. The low intensity application carries the purpose of simply transmitting energy through the medium in order to obtain the information about the medium or to convey information through the medium. High intensity application deliberately affects the propagation medium or its contents. So, the low intensity and high

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intensity application of ultrasonic wave belongs in non-destructive and destructive techniques of characterization respectively.

The quantities, ultrasonic velocity and attenuation are the important parameters, which are required for the ultrasonic non-destructive technique of material characterization. The ultrasonic velocity is related to the elastic constants and density of material. Hence, it gives the information about the mechanical, anisotropic and elastic properties of medium through it passes. It is also important in low temperature physics because it is involved in the evaluation of Debye average velocity and Debye temperature. Ultrasonic velocity in nanofluid depends on the concentration of nano-particles of material dispersed in polymer matrix, thus it is not only important at bulk scale but also at nanoscale. When the ultrasonic wave propagates through the medium, its some part of energy is attenuated through the different mechanism like thermal loss, scattering, absorption, electron-phonon interaction, phonon-phonon interaction, and magnon-phonon interaction etc., called as ultrasonic attenuation. The coefficient of ultrasonic attenuation correlates several physical properties like elastic constants, guruneisen parameter, thermal conductivity, thermal relaxation time, acoustic coupling constant, thermal energy density, specific heat, particle size, density, Debye average velocity, and concentration etc. Thus, the material can be characterized with the knowledge of ultrasonic parameters under different physical conditions.

Normally, the ultrasonic NDT of material characterization are used for the determination of (a) elastic constants (Shear modulus, Bulk modulus, Young modulus and lame modulus), (b) microstructure (grain size, texture, density etc.), (c) discontinuity (porosity, creep damage, fatigue damage etc.), and mechanical properties (tensile strength, shear strength, hardness etc.). The new work in this field also provides the characterization of advanced and smart materials like GMR etc. Now a day, the synthesis and characterization of nanomaterials and nanofluids are also in touch of ultrasonic NDT&E.

In this chapter, ultrasonic material property characterization has been considered. Initially, it covers information about the ultrasonic wave, its mode of propagation and characteristic properties. After this, a brief study of ultrasonic velocity and attenuation in solid has been discussed, which covers the theoretical evaluation and experimental measurements of these ultrasonic parameters. Later on, the characterization of different material (metals, alloys, platinum group metals, nanomaterials, nanofluid, semiconductor etc) has been discussed on the basis of these ultrasonic quantities and related parameters.

## 2. Ultrasonic wave

As a sub category of acoustics, ultrasonics deals with the acoustics above the human hearing range (the audio frequency limit) of 20 kHz. Unlike audible sound waves, the ultrasonic waves are not sensed by human ear due to the limitations on the reception of vibrations of high frequency and energies by the membrane. Ultrasonic wave exhibits all the characteristic properties of sound. Ultrasonic vibrations travel in the form of wave, similar to the way light travels. However, unlike light waves, which can travel in vacuum, ultrasonic wave requires elastic medium such as a liquid or a solid. The wavelength of this wave changes from one medium to another medium due to the elastic properties and induced particle vibrations in the medium. This wave can be reflected off with very small surfaces due to having much shorter wavelength. It is the property that makes ultrasound useful for the non-destructive characterization/testing of materials. The knowledge of generation/detection of ultrasonic wave and its characteristics is important for its precise and suitable application.

### 2.1 Sources of ultrasonic wave

The ultrasonic wave (UW) can be generated with the mechanical, electrostatic, electrodynamic, electromagnetic, magnetostrictive effect, piezoelectric effect, and laser methods.

Mechanical method or Galton Whistle method is an initial method for the generation of ultrasonic wave. This uses mechanical shock or friction for the generation of wave in frequency range of 100 kHz to 1 MHz. A high frequency of ultrasonic wave (10 to 200 MHz) can be generated using electrostatic method. The magneto inductive effect is used in electrodynamic method for the production of ultrasound. The mechanical deformation in ferromagnetic material in presence of magnetic field is called as magnetostriction. This phenomenon is most pronounced in metals such as nickel, iron, cobalt and their alloys. Magnetostriction effect is used for generation of ultrasonic wave in magnetostrictive effect method. In this method, inverse Piezoelectric effect is used for generation of ultrasound is the Piezoelectric effect method. In this surface of suitable material, its some portion of energy is absorbed at the surface with in the skin depth and rest get reflected. The absorbed energy produces tangential stress and then bulk strain through transient surface heating; as a result UW is produced in concerned medium.

## 2.2 Transducers for ultrasonic wave

The device that converts one form of energy to another form is called as transducer. An ultrasonic transducer converts electrical energy to mechanical energy, in the form of sound, and vice versa. The main components are the active element, backing, and wear plate (Fig.1).





#### a. The Active Element

The active element, which is piezo or ferroelectric material, converts electrical energy such as an excitation pulse from a flaw detector into ultrasonic energy. The most commonly used materials are polarized ceramics which can be cut in a variety of manners to produce different wave modes. New materials such as piezo polymers and composites are also being employed for applications where they provide benefit to transducer and system performance.

## b. Backing

The backing is usually a highly attenuative, high density material that is used to control the vibration of the transducer by absorbing the energy radiating from the back face of the active element. When the acoustic impedance of the backing matches with the acoustic impedance of the active element, the result will be a heavily damped transducer that displays good range resolution but may be lower in signal amplitude. If there is a mismatch in acoustic impedance between the element and the backing, more sound energy will be reflected forward into the test material. The end result is a transducer that is lower in resolution due to a longer waveform duration, but may be higher in signal amplitude or greater in sensitivity.

## c. Wear Plate

The basic purpose of the transducer wear plate is to protect the transducer element from the testing environment. In the case of contact transducers, the wear plate must be a durable and corrosion resistant material in order to withstand the wear caused by use on materials such as steel. For immersion, angle beam, and delay line transducers, the wear plate has the additional purpose of serving as an acoustic transformer between the high acoustic impedance of the active element and the water.

Now a days, following type of transducers are in use for different applications.

- 1. Normal beam or single element or delay line transducer
- 2. Dual element transducer
- 3. Angle beam transducer
- 4. Immersion transducer
- 5. Mechanical focous transducer
- 6. Electronic time delay focouing or array transducer
- 7. Capacitive transducer

In most of applications piezoelectric transducers are used for generating and receiving the ultrasonic waves.

## 2.3 Characteristics of ultrasonic wave

For the appropriate choice of ultrasonic wave with suitable frequency and intensity, the knowledge of some essential parameters related to transducer is important. The characteristic parameters of ultrasonic wave are:

1. Sound Field (Near field and far field): The sound field of a transducer is divided in two two zones; the near field region or Fresnel zone and far field region or Fraunhofer zone. In the near field region the ultrasonic beam converges and in the far field it diverges. The near field is the region directly in front of transducer where echo amplitudes goes through a series of maxima and minima and ends at the last maximum, at the distance N (N =  $D^2v/4C = D^2/4\lambda$ ; N: near field distance, D: Element diameter, v: frequency, c: material sound velocity, and  $\lambda$ : wavelength) from the transducer (Fig.2). The intensity variation along and across the axial distance up to near field region is approximately constant and after which it decreases. The beam boundary defines the limits of the beam to the point where the disturbance ceases to exist or falls below the threshold value. The beam intensity at the boundary is reduces to one half (6dB) of the intensity at the beam axis (Fig.2).



Fig. 2. (a) sound field of transducer, (b) amplitude verses frequency of UW, (c) intensity of UW verses axial distance from transducer.

- 2. Focal zone: The starting and ending points of the focal zone on axis of transducer are located where pulse echo signal amplitude drops to -6dB of the amplitude at the focal point. If  $Z_B$  and  $Z_E$  are the beginning and end of the focal zone from the transducer then, then focal zone will be difference of them. The length of focal zone (F<sub>z</sub>) is equal to  $NS_F^2[2/(1+0.5S_F)]$ ; where  $S_F$ : Normalized focal length=F/N, F: focal length, N: near field distance.
- 3. Beam diameter: It is a parameter, which defines the transducers sensitivity. Smaller the beam diameter, the greater amount of energy is reflected by the flaw. At -6dB drop of intensity, the beam diameter (BD) at the focus is equal to  $0.2568DS_F$  or 1.02FC/vD. For the flat transducer, normalized focal length have value one. The Fig.3 represents the clear picture of focal zone and beam diameter.



Fig. 3. Focal zone of transducer and beam diameter

4. Beam spread or half angle: The spreading of ultrasonic beam always take place as the wave travel from the transducer. In the near field, the beam has a complex shape that narrows, while in the far field it diverges. The divergence angle or beam spread angle  $(\theta)$  is equal to  $\sin^{-1}(K\lambda/D)$  or  $\sin^{-1}(KC/vD)$ . Where K is a constant which depends on shape of transducer, edge of beam and method used to determine the beam spread.It is clear that beam spread from a transducer can be reduced by selecting a transducer with higher frequency or larger element diameter or both. Fig.4 shows a simplistic understanding of beam spread angle.



Fig. 4. Ultrasonic beam divergence and angle of divergence

## 2.4 Detection of ultrasonic wave

There are various methods for the detection of UW. The methods are based on the principle of piezoelectric, electrostatic and magnetostriction effects. The classical methods like mechanical and optical methods are also used for the detection of UW. Normally the devices based on piezoelectric effect are used commercially for the detection of UW, these devices comes in electrical method of detection.

The prime division of detection in electrical method are the Interferometer or continuous wave (CW) and Pulse technique (PT) methods. In CW method, the UW generated by the

source is passed through the concerned medium/specimen, which is reflected from the reflecting plate. The reflecting plate is adjusted towards the source such that the current in the oscillator of the source changes periodically in maxima or minima. The maximum in current corresponds to the half of wavelength interval due to the formation of standing wave between source and plate. This method is preferred in low frequency region for the measurements of ultrasonic parameters in liquids.

The Pulse technique is utilized for detection or measurement of transit time in both liquids and solids. It uses piezoelectric transducer with and without delay lines for the production and detection of UW. In this method short duration electric pulses generates the UW with the broadband piezoelectric transducer. The generated longitudinal or shear wave are transmitted to the specimen. The reflected wave or echo by the medium are detected by the transducer on the principle of direct piezoelectric effect and echo pattern is obtained. Using this pattern, the exact transit time needed for a signal to travel between the front and back surface of the specimen or concerned medium is determined, that is used for determination of ultrasonic velocity and attenuation. The different pulse techniques for precise measurements or detection of UW are Sing around, Pulse superposition, Pulse echo overlap, Cross-correlation, Phase slope and Pulse transmission method. Hydrophones are also piezoelectric transducer that generates electrical signal when subjected to pressure change or UW under water. It can detect UW in air, but will be less sensitive due to its design as having a good acoustic impedance match with water.

# 3. Material characterization techniques (NDT & DT)

The two major classification of material characterization technique are non-destructive testing (NDT) and destructive testing (DT). Under destructive technique (such as: tensile testing, creep testing, impact testing, torsion testing, hardness testing etc.) of characterization the tested material or product can not be used again. The destruction of test object usually makes this type of test more costly. Non-destructive testing technique is a specific procedure whereby the service ability of materials or components is not impaired by testing process. The various methods like visual testing, liquid penetrant testing, magnetic particle testing, eddy current testing, radiographic testing, ultrasonic testing, leak testing, thermography and neutron radiography are the NDT technique of material characterization. Among the various non-destructive testing and evaluation (NDT&E) plays a key role in material characterization.Ultrasonic properties provide important diagnostic for microstructural properties as well as deformation processes in a material, controlling material behaviour based on the physical mechanism to predict future performance of the materials.

# 4. Classification of ultrasonic application and testing

The ultrasonic testing involves both the low intensity and high intensity ultrasonic wave for the characterization, that belongs in non-destructive and destructive techniques of characterization respectively. Uses of high intensity and low frequency ultrasonic wave includes medical therapy and surgery, atomization of liquids, machining of materials, cleaning and wielding of plastics and metals, disruption of biological cells, and homogenization of materials. The low intensity and high frequency ultrasonic waves are applied for medical diagnosis, acoustical holography, material characterization etc. The low intensity ultrasoud measurements provides a good diagnosis of material property and process control in industrial apllication (Alers, 1965; Green, 1973; Lowrance, 1975; Renolds, 1978; Teagle, 1983; Smith, 1987; Varry, 1987; Thompson, 1996; Jayakumar, 1998; Kumar, 2001; Raj, 2003; Roth, 2003; Blodgett, 2005).

## 5. Ultrasonic NDT as a material characterization

There are four mode of propagation by which an ultrasonic wave can propagate in a medium, as: longitudinal or compressnal wave, transverse or shear wave, surface or Rayleigh wave and plate or lamb wave. The most common methods of ultrasonic examination utilize the longitudinal waves or shear waves.

Ultrasonic velocity or attenuation are the parameters that correlate to structural inhomogenities or flaw size atomistic (interstitials), elastic parameters, precipitates, dislocations, ordering of molecules in liquid crystals, phase transformations, porosity and cracks, concentration of different components of alloys or mixed crystal system, vacancies in lattice sites, size of the nanoparticles in nano-structured materials, electrical resistivity, specific heat, thermal conductivity and other thermophysical properties of the materials depending upon the different physical conditions like temperature, pressure, crystallographic orientation, magnetization etc. Thus, ultrasonic study of a material provides information about elastic constants, microstructure, discountinuty, and mechanical properties under different condition.

#### 5.1 Ultrasonic velocity

On the basis of mode of propagation there are four types of ultrasonic velocities, as longitudinal, shear, surface and lamb wave velocity. Longitudinal and shear wave velocities are more important for the material characterization because they are well related to elastic constants and density. However, it is independent of frequency of wave and dimension of the given material. The mechanical behaviour and anisotropic properties of the material can be well defined on the knowledge of ultrasonic velocity. The mathematical formulations and measurement techniques for ultrasonic velocity are detailed in following heads.

#### 5.1 A Ultrasonic velocity, related parameters and its theoretical evaluation

The mechanical properties of the solids differ from those of fluids in two important respects. Firstly, greater binding forces exist between their constituent atoms so that they support shear stress. Secondly, anisotropy may occur, especially in single crystal, in which the atoms form regular lattice. The velocity of ultrasonic wave of any kind can be determined from the elastic moduli (Y: Young's modulus, G: modulus of rigidity, and  $\sigma$ : poisson's ratio) and density (d) of the material. The logngitudinal and shear wave velocities (V<sub>L</sub> and V<sub>S</sub>) can be determined with following expressions.

$$V_L = \left[\frac{Y(1-\sigma)}{d(1+\sigma)(1-2\sigma)}\right]^{1/2} \text{ and } V_S = \left[\frac{Y}{2d(1+\sigma)}\right]^{1/2} = \left[\frac{G}{d}\right]^{1/2}$$
(1)

In terms of lame's moduli ( $\lambda$  and  $\mu$ ), the ultrasonic velocities can be expressed as;

$$V_L = \left[\frac{\lambda + 2\mu}{d}\right]^{1/2} \text{ and } V_S = \left[\frac{\mu}{d}\right]^{1/2}$$
(2)

The stress strain relationships for anisotropic crystals vary with the direction. Thus velocity of ultrasonic wave varies with the direction of propagation of wave and mode of polarization. There are three type of ultrasonic velocity (one longitudinal and two shear wave) for each direction of propagation of wave in cubic (Mason, 1958; Singhal 2003) and hexagonal structured materials (Mason,1969; Alers,1958; Rosen,1970; Yadawa,2009). The expressions for the velocities are given in Table (1) and Table (2). In Tables 1-2, the V<sub>1</sub> is longitudinal and V<sub>2</sub> & V<sub>3</sub> are the shear wave velocities of ultrasonic wave. The C<sub>11</sub>, C<sub>12</sub>, C<sub>44</sub>, C<sub>33</sub> and C<sub>66</sub> are the second order elastic constants.

The Debye theory of specific heat has proven its usefulness because it is a single –parameter theory which describes the observation remarkebly well. Its one parameter, Debye temperature ( $T_D$ ) need not to be determined by any heat capacity measurements but can be calculated from the elastic moduli. Once this parameter has been determined from the elastic moduli, the Debye theory specifies the lattice contribution to the specific heat only to an accuracy of about 10 or 20% over most of temperature range. Because of this, the theoratical model assumes the solid to be an elastic continuum in which all sound waves travel at the same velocity independent of their wavelength. This model is satisfactory only in the limit of long wavelengths or low temperatures. The expression for the  $T_D$  can be given as:

$$T_D = \frac{\hbar V_D (6 \pi^2 n_a)^{1/3}}{K_B}$$
(3)

Direction of propagation	Direction of polarization	Type of wave	Velocity expression	Velocity notation
100	100	Long.	$(C_{11} / d)^{1/2}$	V <sub>1</sub> =V <sub>L</sub>
	010	Shear	$(C_{44} / d)^{1/2}$	V <sub>2</sub> =V <sub>S1</sub>
	001	Shear	$(C_{44} / d)^{1/2}$	V <sub>3</sub> =V <sub>S2</sub>
110	110	Long.	$\left(\left(C_{11}+C_{12}+2C_{44}\right)/2d\right)^{1/2}$	$V_1 = V_L$
	001	Shear	$(C_{44} / d)^{1/2}$	V <sub>2</sub> =V <sub>S1</sub>
	$1\overline{1}0$	Shear	$\left( \left( C_{11} - C_{12} \right) / 2d \right)^{1/2}$	V <sub>3</sub> =V <sub>S2</sub>
111	111	Long.	$\left(\left(C_{11}+2C_{12}+4C_{44}\right)/3d\right)^{1/2}$	$V_1 = V_L$
	Any direction in 111 plane	Shear	$\left(\left(C_{11} - C_{12} + C_{44}\right) / 3d\right)^{1/2}$	$V_2 = V_3$ $V_{S1} = V_{S2}$

Here,  $\hbar$  is quantum of action and is equal to Planck's constant divided by  $2\pi$ ; K<sub>B</sub> is Boltzmann Constant; n<sub>a</sub> is atom concentration. This Debye average velocity is important

Table 1. Ultrasonic velocities for cubic structured materials

Direction of propagation	Direction of polarization	Type of wave	Velocity expression	Velocity notation
001 (Along unique axis or z-axis)	001	Long.	$(C_{33} / d)^{1/2}$	$V_1 = V_L$
	Any direction in 001 plane	Shear	$(C_{44} / d)^{1/2}$	V <sub>2</sub> = V <sub>3</sub> V <sub>S1</sub> =V <sub>S2</sub>
100 (or any other direction perpendicular to 001)	100	Long.	$(C_{11} / 2d)^{1/2}$	$V_1 = V_L$
	001	Shear	$(C_{44} / d)^{1/2}$	V <sub>2</sub> =V <sub>S1</sub>
	010	Shear	$\left(\left(C_{11} - C_{12}\right) / 2d\right)^{1/2}$	V <sub>3</sub> =V <sub>S2</sub>
At angle θ with the unique axis of the crystal		Long.	$\begin{split} & [\{C_{33}Cos^{2}\theta+C_{11}Sin^{2}\theta+C_{44}+\\ & \{[C_{11}Sin^{2}\theta-C_{33}Cos^{2}\theta+C_{44}\\ & (Cos^{2}\theta-Sin^{2}\theta)]^{2}+4\ Cos^{2}\theta\\ & Sin^{2}\theta\ (C_{13}+C_{44})^{2}\}^{1/2}\ \}/2d]^{1/2} \end{split}$	V <sub>1</sub> =V <sub>L</sub>
		Shear	$[\{C_{33}Cos^{2}\theta + C_{11}Sin^{2}\theta + C_{44} - \\ \{[C_{11}Sin^{2}\theta - C_{33}Cos^{2}\theta + C_{44} \\ (Cos^{2}\theta - Sin^{2}\theta)]^{2} + 4Cos^{2}\theta \\ Sin^{2}\theta (C_{13} + C_{44})^{2}\}^{1/2} \}/2d]^{1/2}$	V_=V_S1
		Snear	$[\{C_{44}Cos^2\theta + C_{66}Sin^2\theta\} / d]^{1/2}$	$v_3 = v_{S2}$

Table 2. Ultrasonic velocities for hexagonal structured materials

parameter in the low temperature physics because it is related to elastic constants through ultrasonic velocities. The Debye average velocity ( $V_D$ ) in the materials is calculated using the following equation (Oligschleger, 1996).

$$V_D = \left(\frac{1}{3}\sum_{i=1}^3 \int \frac{1}{V_i^3} \frac{d\Omega}{4\pi}\right)^{-1/3}$$
(4)

Here the integration is over all directions and summation is over the type of ultrasonic velocities. Along the [100], [111] (for cubic crystal) and [001] (for hexagonal structured crystals) direction of propagation of wave, the equation (4) reduces as:

$$V_D = \left[\frac{1}{3} \left(\frac{1}{V_1^3} + \frac{2}{V_2^3}\right)\right]^{-1/3}$$
(4a)

and along the [110] (for cubic) and any angle with the unique axis of hexagonal structured crystal, direction of propagation, the equation (4) reduces as:

$$V_D = \left[\frac{1}{3} \left(\frac{1}{V_1^3} + \frac{1}{V_2^3} + \frac{1}{V_3^3}\right)\right]^{-1/3}$$
(4b)

On the knowlewdge of elastic constants, the theoretical evaluation of ultrasonic velocity and Debye average velocity in cubic and hexagonal structured materials can be done with help of expressions written in Table (1), Table (2) and equations (4a)-(4b). There are several theories (Ghate,1965; Mori,1978; Rao,1974; Yadav AK, 2008) for the calculation of elastic constants. The elastic constants depend on the lattice parameters of structured materials. The elastic constants and elastic moduli can be calculated with the knowledge of lattice parameters.

## 5.1 B Measurement techniques of ultrasonic velocity

The study of the propagation of ultrasonic waves in materials determines the elastic constants, which provides better understanding of the behaviour of the engineering materials. The elastic constants of material are related with the fundamental solid state phenomenon such as specific heat, Debye temperature and Grüneisen parameters. The elastic constants in the materials can be determined by measuring the velocity of longitudinal and shear waves. Elastic constants are related to interatomic forces, co-ordination changes etc., and also with the impact shock, fracture, porosity, crystal growth and microstructural factors (grain shape, grain boundaries, texture and precipitates etc.). So, the study of ultrasonic velocity is useful not only for characterization of the structured materials, engineering materials, porous materials, composites, glasses, glass ceramics but also bioactive glasses, nanomaterials, nanofluids etc.

Interferometer or continuous wave method and pulse technique are the general electrical method for the measurement of ultrasonic velocity. In CW method, the wavelength of wave in the test material is measured, which in turn provides the ultrasonic velocity with relation  $V = v \lambda$ . While in the Pulse technique, transit time (t: the time needed for a signal to travel between the front and back surface of the specimen or concerned medium) is measured with the help of echo pattern. If x is thickness of the material then ultrasonic velocity becomes equal to 2x/t.

For precise measurement, the Pulse technique has been improved in the form of following techniques (Papadakis, 1976, Raj, 2004).

- a. Sing around
- b. Pulse superposition method
- c. Pulse echo overlap method
- d. Cross-correlation method and
- e. Phase slop method
- f. Pulse transmission method

The pulse echo-overlap, pulse transmission and pulse superposition techniques are widely used techniques due to their absolute accuracy and precision respectively. Now a day, computer controlled devices of pulse echo overlap and pulse superposition techniques are being used. Resonance ultrasound spectroscopy and Laser interferometry are the recent techniques for the measurement of ultrasonic velocity in thin film, crystal, textured alloy etc.

## 5.1 C Application of ultrasonic velocity

Ultrasonic velocity has a wide range of application in the field of material characterization. Yet it is useful for the characterization or study of all the three phase of matter but here we concentrate only its application to solid materials. It is used in the study of following properties of materials.

1. Elastic constants: The elastic moduli of a material are important for the understanding of mechanical behaviour. If  $V_L$  and  $V_S$  are the measured ultrasonic velocities of longitudinal and shear wave then longitudinal modulus (L), Shear modulus (G), Bulk modulus (B), Poisson's ratio ( $\sigma$ ),Young modulus (Y) and lame's modulus ( $\lambda$  and  $\mu$ ) can be obtained with the following expression.

$$L = V_L^2 d$$

$$\mu = G = V_S^2 d$$

$$B = L - (4/3)G$$

$$\sigma = \frac{L - 2G}{2(L - G)}$$

$$Y = (1 + \sigma)2G$$

$$\lambda = (V_L^2 - 2V_S^2)d$$
(5)

We can also find the stiffness constants or second and forth order elastic constants with the velocity. Using Table (1)-(2), one can find the second order elastic constants along different crystallographic direction for cubic and hexagonal structured materials. If we have ultrasonic velocity under different physical condition like temperature, pressure, composition of materials etc. then we can predict the mechanical behaviour of material in different physical condition. The anisotropy of material can be explained with the knowledge of anisotropy factor A=[2C<sub>44</sub>/(C<sub>11</sub>-C<sub>12</sub>)]. Knowledge of pressure derivatives of the elastic constants of a structured material can be used for the evaluation of Grüneisen parameter ( $\gamma$ ). The Grüneisen parameter is used to describe anharmonic properties of solids. The quasi harmonic model is usually the starting point for the evaluation of mode frequency of crystal lattice and V is the volume of the crystal. The values of  $\gamma_i$  for low frequency acoustic modes in a given material can be obtained with the pressure derivates of elastic constants of that material. Finally the Grüneisen parameter is obtained with the average of  $\gamma_i$  as shown in the following expression.

$$\gamma = \left[\sum_{i=1}^{3N} C_i \ \gamma_i / \sum_{i=1}^{3N} C_i \right]$$
(6)

Different workers (Mason, 1965; Brugger, 1964; Anil, 2005; Yadav, 2007; Yadawa, 2009; Yadav AK,2008) have studied this property of the different structured materials like isotropic, cubic, rhombohedral and hexagonal structured materials.

2. Debye temperature and Debye average velocity: These parameters are essential for the understanding of lattice vibration and low temperature properties of the material. These parameters can be found directly with the velocity values using equation (4) for the cubic and hexagonal structured materials. A detail study of Debye temperature, velocity and related theories of different structured materials can be seen elsewhere (Alers, 1965).

3. Porosity: The porosity of the porous material can be examined with the knowledge of elastic moduli and Poisson's ratio as a function of pore volume fraction. These parameters can be evaluated with help of measured velocity and density. A simple expression of Young modulus and shear modulus for a porous material can be written as,

$$Y = Y_0 \exp(-ap-bp^2) |$$

$$G = G_0 \exp(-ap-bp^2) |$$
(7)

Here  $Y_0$  and  $G_0$  are the modulus of material without pore; a,b and c are the constants; p is pore volume fraction which is equal to {1-(d/d\_0)} and; d is the bulk density determined experimentally from mass and volume while  $d_0$  is the theoretical density determined from XRD.

The elastic moduli and Poisson ratio measured ultrasonically are compared with the theoretical treatment for the characterization. The elastic moduli of porous material are not only the function of porosity but also the pore structure and its orientation. The pore structure depends on the fabrication parameters like compaction pressure, sintering temperature and time. If the pores are similar in shape and distributed in homogeneous pattern then a good justification of mechanical property can be obtained with this study.

5. Grain size: There is no unique relation of average grain size with the ultrasonic velocity. The following typical graph (Fig. 5) shows a functional relation among velocity (V), grain size (D) and wave number (k). This has three distinct regions viz. decreasing, increasing and oscillating regions. Both the I and II region are useful for the determination of grain size determination, whereas region III is not suitable.



Fig. 5. Ultrasonic velocity as a function of kD

The obtained grain size with this study has good justification with grain size measured with metallography. The important advantage of using ultrasonic velocity measurements for the grain size determination is the accuracy in which ultrasonic transit time could be determined through electronic instrumentation. The different workers (Palanichamy, 1995) have studied this property for polycrystalline material with the study of ultrasonic velocity. 6. Anisotropic behaviour of compositional material: The intermetallic compound and alloys

are formed by the mixing of two or more materials. These compounds have different mechanical properties depending on their composition. The different mechanical properties like tensile strength, yield strength, hardness (Fig.6) and fracture toughness at different

composition (Fig. 7), direction/orientation (Fig.8) and temperature can be determined by the measurement of ultrasonic velocity which is useful for quality control and assurance in material producing industries (Krautkramer, 1993; Raj, 2004; Yadav & Singh 2001; Singh & Pandey, 2009, Yadav AK, 2008).



Fig. 6. Variation of velocity or hardness with temperature for some mixed materials



Concentration

Fig. 7. Variation of velocity with concentration in some glasses



Fig. 8. Variation of V<sub>D</sub> with the angle from the unique axis of hexagonal structured crystal

7. Recrystallisation: The three annealing process that amend the cold work microstructure are recovery, recrystallisation and grain growth. Among these processes, recrystallisation is the microstructural process by which new strain free grains form from the deformed microstructure. Depending on the material, recrystallisation is often accompanied by the other microstructural changes like decomposition of solid solution, precipitation of second phases, phase transformation etc. The hardness testing and optical metallography are the common techniques to the study the annealing behaviour of metals and alloys. A graph of longitudinal and shear wave velocity with annealing time (Fig.9) provides a more genuine understanding of recrystallisation process.



Annealing Time

### Fig. 9. Variation of $V_L$ or $V_S$ with annealing time

The variation of shear wave velocity represents a slight increase in recovery region followed by a rapid increase in the recrystallisation region and saturation in the completion of recrystallisation region. The slight increase in the velocity in the process of recovery is attributed to the reduction in distortion of lattice caused by the reduction in point defect due to their annihilation. The increase in velocity during recrystallisation is credited to the change in the intensity of lattice planes. The variation in longitudinal velocity have the just opposite trend to that of shear wave velocity which is credited to the change in texture and the dependence of velocity directions of polarisation and propagation of wave. The variation of velocity ratio  $(V_L/V_S)$  with annealing time shows a clear picture of recrystallisation regime (Fig. 10).



Annealing Time

Fig. 10. Variation of  $V_L/V_S$  with annealing time

The selection of ratio avoids the specimen thickness measurement and enhances the accuracy. In short we can say that the velocity measurement provides the accurate prediction of on set and completion times of recrystallisation.

8. Precipitation: For the desired strength of material or component, the precipitation is a process like recrystallisation. It is a metallurgical process for the improvement of strength of material. The strength of improvement depends on spacing, size, shape and distribution of precipitated particles. A measurement of longitudinal ultrasonic wave velocity with ageing time provides precise value of Young modulus at different ageing temperature (Bhattacharya, 1994; Raj, 2004). With the knowledge Young modulus, the strength of material at different time of ageing can be predicted. Thus ultrasonic evaluation may be handy tool to study the precipitation reaction involving interstitial elements because this mechanism is associated with large change in the lattice strain.

9. Age of concrete: There are several attempts that have been made to find the elastic moduli, tensile strength, yield strength, hardness, fracture toughness and brittleness of different materials (Lynnworth, 1977; Krautkramer,1977). Similarly the age of concrete material can be determined with knowledge of crush strength that can be found with the ultrasonic velocity. A graph of pulse velocity of ultrasonic wave and crush with age of concrete is shown in Fig 11.



Fig. 11. Variation of velocity and crush strength with age of concrete

10. Cold work and texture: The texture of compounds can be understood with the knowledge of ultrasonic velocity. The expression of texture designates an elastic anisotropy due to the non-random distribution of crystalline directions of the single crystals in the polycrystalline aggregates. On the contrary, the isotropic, untextured solid is characterized by a totally random distribution of the grains. A study on texture gives insight into the materials plastic properties. Ultrasonic velocity measurements provide the state of texture in the bulk. For this purpose, ultrasonic velocity with cross correlation method {V<sub>IJ</sub>; where I (direction of propagation) or J (direction of polarization) =1,2,3; 1:rolling, 2: transverse, 3:normal) }or Rayleigh wave velocity in transverse direction is measured as function of cold work (Raj,2004). Accordingly, three longitudinal (V<sub>11</sub>, V<sub>22</sub>, V<sub>33</sub>) and six shears (V<sub>12</sub>, V<sub>21</sub>, V<sub>23</sub>, V<sub>32</sub>, V<sub>31</sub> and V<sub>13</sub>) wave velocities are measured. The velocities are found to be identical when the direction of propagation and direction of polarization are interchanged. Yet the measured velocities of longitudinal and shear wave propagating perpendicular to rolling

direction are important for estimation of cold work with good precision but  $V_{33}$  and  $V_{32}$  are found to be more suitable due to being easier in measurement. With the following relation, we can estimate the degree of cold work with help of velocity ratio ( $V_{33}$  / $V_{32}$ ).

 $V_{33} / V_{32} = 0.00527$  (% cold work) -1.83; { Correlation coefficient = 0.9941} (8)

The following graph (Fig. 12) represents the variation of velocity ratio with cold work.



Cold work (%)

Fig. 12. Variation of velocity ratio with cold work

The Rayleigh wave velocity in transverse direction decreases with cold work and is linear in nature. A scatter in measurement is mainly attributed to the local variation in the degree of deformation, particularly close to surface caused by scattering. Both the methods are appropriate for the evaluation of cold work percentage in stainless steel. Thus measurement of bulk and surface Rayleigh wave velocities on cold rolled plates provide a tool to monitor the percentage of cold work during rolling operation.

## 5.2 Ultrasonic attenuation

The intensity of ultrasonic wave decreases with the distance from source during the propagation through the medium due to loss of energy. These losses are due to diffraction, scattering and absorption mechanisms, which take place in the medium. The change in the physical properties and microstructure of the medium is attributed to absorption while shape and macroscopic structure is concerned to the diffraction and scattering. The absorption of ultrasonic energy by the medium may be due to dislocation damping (loss due to imperfection), electron-phonon interaction, phonon-phonon interaction, magnon-phonon interaction, thermoelastic losses, and bardoni relaxation. Scattering loss of energy is countable in case of polycrystalline solids which have grain boundaries, cracks, precipitates, inclusions etc. The diffraction losses are concerned with the geometrical and coupling losses, that are little or not concerned with the material properties. Thus in single crystalline material, the phenomenon responsible to absorption of wave is mainly concerned with attenuation. An addition of scattering loss to the absorption is required for knowledge of attenuation in polycrystalline materials. So, the rate of ultrasonic energy decay by the medium is called as ultrasonic attenuation.

The ultrasonic intensity/energy/amplitude decreases exponentially with the source. If  $I_X$  is the intensity at particular distance x from source to the medium inside then:

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