CHAPTER 2

EXPERIMENTAL TECHNIQUE
2.1 BRIEF DESCRIPTION OF THE VARIOUS METHODS FOR THE DETERMINATION OF ELASTIC CONSTANTS OF CRYSTALS

A critical account of the experimental techniques available to study the elastic properties of solids is presented in this section.

Elastic constants of solids can be measured using various techniques. These techniques can be broadly divided into three categories: (1) static methods (2) X-ray methods and (3) ultrasonic methods. Static methods are the oldest of the three. The dynamic methods in general are capable of yielding higher precision of measurement. Static methods yield the isothermal moduli while the dynamic methods based on resonant techniques usually give the adiabatic moduli.

Voigt\textsuperscript{15}, in his classical experiments, has made use of the principles of bending and twisting the crystal plates and crystal bars to determine the elastic constants of the crystals. Crystal plates cut along the known directions were bent and the Young's modulus was determined. The crystal rods of known orientation were twisted and the rigidity modulus was determined. Voigt has determined the elastic constants of a large number of crystals and he was able to achieve all this by the above methods for which fairly large specimen samples are required. Refinements in these experiments were later on, introduced by Tutton\textsuperscript{16},
Bridgman\textsuperscript{17}, Mandell\textsuperscript{18}, Hansen\textsuperscript{19}, Hins\textsuperscript{20}, Swift and Tyndall\textsuperscript{21} Sunder Rau\textsuperscript{22} and others. These methods are laborious, time consuming and demand enormous skill to make accurate strain measurements. The main drawback of the static methods is that they require large samples.

In view of these disadvantages encountered in the static methods, a number of dynamic methods have come into existence for the measurement of crystal elasticity. Direct application of vibration methods to the crystals have been made by Wright\textsuperscript{23}, Davies\textsuperscript{24}, Geens\textsuperscript{25}, Mason\textsuperscript{26}, Hunter and Siegel\textsuperscript{27} and others. The main principle involved in these methods is that an alternating driving force applied to the specimen excites the appropriate vibrations in it. The response is measured over a particular range of frequencies. From a knowledge of the sample dimensions and the frequency at which the response is maximum the elastic constants could be calculated.

The electrostatic method of exciting the specimens into vibration was first used by Ide\textsuperscript{28}. He studied the elastic properties of rock specimens. This method was used by Bordeni\textsuperscript{29} to determine the velocity of extensional waves in metals such as lead, tin, aluminium and cadmium up to their melting points. By using the same method he also studied the anelastic behaviour of metals\textsuperscript{30} over a low temperature range (4.5°-300°k).
Fintel, Wagner and Walther described a simple method of setting rods into longitudinal and torsional vibrations by using a magnetic device. A resonance method was developed by Förster and Förster and Köster in which flexural vibrations are excited in rods by an electromagnetic drive. Spinner also used this method to excite the torsional vibrations in isotropic bars. This method was used by several workers to study the elastic properties of many refractory oxides. Bradfield reviewed the use of magnetostriuctive transducers to excited either longitudinal or torsional vibrations in rock samples. Van Der Burgt determined the dynamic constants of nickel and nickel-sin ferrites using a magnetostriuctive method.

Atanassoff and Hart in their method used piezo-electric crystal plates directly in the electrical circuits to study the vibrational pattern and hence to measure the elastic constants. Bhagavantam and Suryanarayana applied this method to measure the elastic constants of tourmaline and sino-blende crystals. This method has got a very limited application and the results thus obtained have to be corrected for the influence of piezo-electric effects. Further when a resonant frequency is observed it is not so easy to decide with this method to which harmonic it belongs. This method can be used in the frequency range 1 to 10 MHz and an accuracy of the order of 2 to 5 percent could be realised.
X-ray method of studying the crystal elasticity involves the measurement of the intensity of the monochromatic X-rays which interact with the lattice vibrations of the crystal. This technique has been used by Curien\textsuperscript{40}, Jacobsen\textsuperscript{41} and Johnson\textsuperscript{42} to determine the force constants of copper, iron and zinc. Gunther\textsuperscript{43} has used X-ray diffraction technique to measure the elastic constants of aluminium. Wooster and Ramachandran's\textsuperscript{44} method depends on the coherently diffracted X-ray beam which is scattered by interaction with the thermal waves of the lattice. The procedure consists of observing the variations of intensity along any specific crystallographic direction through the individual Bragg reflections. The contribution from the first order of the diffuse coherent diffraction varies linearly with temperature and is proportional to appropriate linear combinations of the elastic stiffness constants. Hence from the positions of these diffused reflections, the elastic constants of the crystal can be determined. The method is capable of yielding the ratio of the elastic constants to a high degree of accuracy though the absolute accuracy is poor compared to that achieved in dynamic methods.

Schaefer and Bergmann\textsuperscript{45} were the earliest workers to make use of the phenomenon of piezo-electricity of quartz for a study of elastic constants of the crystals. In this
method a fair sized well polished transparent cube of the test material is set into vibration by a quartz transducer. Due to couplings at the boundaries and reflections of the waves, both longitudinal and shear, a three dimensional grating will be set-up in the cube. If a light beam from a circular aperture traverses such a medium, characteristic diffraction pattern results. From a knowledge of a constant, characteristic of the experimental set-up, dimensions of the diffraction pattern and the operating frequency, the elastic constants can be determined. Obviously this method is suitable for transparent solids. But in a later development\textsuperscript{46} the method is modified to suit to opaque solids also. Here the light is reflected from the surface of the vibrating specimen. The main difficulty with this method lies in that to get a fairly intense diffraction pattern, one has to apply a large amount of power to the crystal, which involves temperature fluctuations in the sample. As such the method is unsuitable for a study of the temperature dependence of elastic constants. Further more the attainable accuracy is only of the order of 5 percent. Bolef and Neves\textsuperscript{47} developed a high frequency resonance method in which a thin quartz transducer connected to the specimen is connected to a Q-meter. The resonance is indicated by a sharp dip in the Q-meter.

Hiddemann and his coworkers\textsuperscript{48}, have used the phenomenon of diffraction of light by ultrasound waves and developed a
technique, basing on it, for measurement of elastic constants of isotropic solids. Later Bes-Bardili modified this method to study the elastic constants of various metals and glasses. This method was extended to single crystals also by Bhagavantam and Ramachandra Rao. For successful application of this method, one has to employ well polished crystals at high frequencies.

Some of the defects associated with the above methods have been eliminated in the wedge method developed by Bhagavantam and Bhimasenachar. This method deals with the study of the vibrational modes of non-piezoelectric crystal plates. Hence the method has the obvious advantage of being applicable to opaque media. The specimen in the form of a plate is cemented to the quartz wedge and the system is driven by an alternating current of variable frequency, thus providing a continuous supersonic spectrum. The specimen is in contact with a transparent liquid through which a light beam is passed. As the exciting frequency of the wedge is varied, an appropriate portion of the wedge resonates and produces an ultrasonic beam of same frequency. These resonant frequencies of the specimen are detected by the maximum in the diffraction effects on the light produced by the ultrasonic grating in the liquid. The transmission frequencies thus produced have to be sorted out and assigned to their appropriate modes. A sufficient
number of such determinations permit one to evaluate the complete set of elastic constants. This method has a great advantage over the other methods in that, even small specimens can be used. This fact facilitated the determination of the elastic constants of diamond. In these experiments the frequency range employed is from 0.5 to 10 MHz and the accuracy is about five percent. The main drawback of this method is that the specimen under investigation comes into contact with the liquid. This method cannot be applied to substances such as wood, which have a tendency to absorb liquids. The same defect makes it unsuitable for the temperature work also.

In recent years there are number of methods available, especially after the advent of radar techniques, for the timing of short high frequency pulses of ultrasonic power. The method has been employed to study the elastic constants of the solids by Huntington, McSkimin, Mason, Galt, Holbrook, Arenberg, Bacon and Smith, Bradfield, Musgrave and many others. Excellent reviews have been written by McSkimin, Anderson and Libermann and Schreiber. described a method for performing pulse transmission measurements with improvement in both precision and accuracy. Essentially in the pulse method a quartz transducer is cemented to one of the plane parallel faces of the specimen. A pulse of the order of a microsecond
duration is generated and transmitted through the specimen. On reflection at the opposite surface it returns and when it arrives back at the quartz gives rise to electrical signal or echo. It is possible to observe a whole sequence of such echoes and from them to measure with increased accuracy the time interval for transit. X-cut and Y-cut quartz transducers having resonant frequencies at 10MHz are usually employed to execute longitudinal and transverse waves respectively. The pulse technique has proved valuable in the study of the influence of temperature and pressure on elastic constants of crystals. Accuracies better than one percent are easily obtained with this method.

Interferometry has been combined with pulse technique to attain higher precision in the velocity measurements with small specimens. The specimens are mounted on the end surfaces of fused quartz rods so that the multiple echoes come in a group, after an appropriate time laps of the initial pulse. The pulse length can be extended until several pulses overlap. The frequency is varied until the condition for constructive interference is indicated by clear step pattern in the overlapping echoes. In obtaining the sound velocity from differences between these resonant frequencies, the phase shifts introduced due to reflections at the boundaries of the specimen have to be taken into account. Schreiber et al have given an extensive review on the pulse methods recently.
All the ultrasonic methods, with the exception of pulse methods have one defect or other which make them unsuitable for the study of elastic constants as a function of temperature.

All these defects are eliminated in the composite piezoelectric oscillator originally developed by Balamuth\textsuperscript{70} and Rose\textsuperscript{71} based on the work of Quimby\textsuperscript{72}. In this technique, a small sample in the form of a rod with square cross section is cemented to a piezoelectric crystal. A determination of the resonant frequencies of the quartz and composite system makes it possible to calculate the natural frequency of the specimen and there by its elastic constant. The method has been successfully used by Durand\textsuperscript{73}, Hunter and Siegel\textsuperscript{74}, Subrahmanyan\textsuperscript{75}, Sutton\textsuperscript{76}, Jayarama Reddy\textsuperscript{77}, Rama Murthy\textsuperscript{78}, Nagi Reddy\textsuperscript{79} and Seshagiri Rao et al\textsuperscript{80} for the study of temperature variation of elastic constants of single crystals, polycrystalline materials and ferrites. The frequency range over which the observations are made is 100 to 200 kHz and the accuracy better than 0.5 percent is realised for relative measurements.

Quartz has got an $\alpha - \beta$ transformation at 577°C and hence it sets an upper limit to the temperature range that can be covered by the composite oscillator method. This difficulty was overcome by Hunter and Siegel\textsuperscript{27} by the use of three component piezoelectric oscillator method. The
additional component was a fused silica rod, inserted between the specimen and the quartz crystal. The length of the rod is adjusted to be equal to an integral number of wave lengths so that the specimen could be placed in the hot zone of a furnace while maintaining the transducer at room temperature.

Marx developed a three-part oscillator technique in which he used two quartz rods, one as a driver and the other as a gauge, cemented to the specimen on either side of it. This method was widely used to calculate the damping of the specimen by many workers.

Since the composite oscillator technique is simplest of all and versatile for the study of the temperature variation of elastic constants of ferro-alloys with desired accuracy, it is employed in the present investigation. The method is detailed in the following sections.
2.2 ELASTIC WAVES IN ISOTROPIC SOLIDS

In a solid body stress can be resolved into six components: three longitudinal and three shearing. In a similar way the strain produced can also be resolved into six components. According to Hooke's law, each component of stress can be expressed as a linear combination of the strain components and vice versa. For the most anisotropic crystal system (triclinic) there are 21 independent elastic constants. This number reduces as the symmetry of the material increases and for an isotropic body only two elastic constants are independent. The six stress-strain relations are then

\[ T_1 = (\lambda + 2\mu) S_1 + \lambda (S_2 + S_3) = \lambda M + 2\mu S_1 \]
\[ T_2 = (\lambda + 2\mu) S_2 + \lambda (S_1 + S_3) = \lambda M + 2\mu S_2 \]
\[ T_3 = (\lambda + 2\mu) S_3 + \lambda (S_1 + S_2) = \lambda M + 2\mu S_3 \]
\[ T_4 = \mu S_4 \]
\[ T_5 = \mu S_5 \]
\[ T_6 = \mu S_6 \]

... 2.2.1

where \( M = S_1 + S_2 + S_3 \). The \( T_i \)'s are the six stresses and the \( S_i \)'s are six strains. \( \lambda \) and \( \mu \) are the two Lamé constants. Still there are three other constants of interest. They are (i) Young's modulus (\( Y \)), (ii) the bulk modulus (\( B \)) and (iii) the Poisson's ratio (\( v \)). Young's modulus is defined as the
ratio of the longitudinal stress to longitudinal strain in a solid, when the extensional stress is applied along one axis. If we take this axis as z-axis, \( T_1 = T_2 = 0 \). Solving for the ratio of \( T_3 \) to \( S_3 \) when \( S_1 \) and \( S_2 \) are eliminated from 3rd equation of 2.2.1, we get

\[
\frac{T_3}{S_3} = \frac{\mu(3\lambda + 2\mu)}{\lambda + \mu} = \gamma
\]  

(2.2.2)

On the other hand if we take the ratio

\[
\frac{S_1}{S_3} = \frac{S_2}{S_3} = \sigma
\]  

(2.2.3)

the Poisson's ratio, we find

\[
\sigma = \frac{\lambda}{2\lambda + \mu}
\]  

(2.2.4)

The bulk modulus \( B \), is defined as the ratio of a hydrostatic pressure \( p \) to the relative change in volume of the material. Setting \( T_1 = T_2 = T_3 = -p \) and solving for \( N \), the change in volume, we find

\[
N = -3p/(3\lambda + 2\mu)
\]

Taking the amplitude of the ratio \( p \) to \( N 

\[
\frac{p}{N} = \frac{(3\lambda + 2\mu)}{3} = \lambda + 2/3\mu = B
\]  

(2.2.5)
From the Newton's second law one can derive the equations of motion for waves in an unbounded medium. If we consider an elementary cube of volume six \( \delta x \delta y \delta z \), the equation of motion are

\[ \rho \frac{\partial^2 u_i}{\partial t^2} \delta x \delta y \delta z = F_i \text{ where } (i=1,2,3) \]  

(2.2.6)

Where \( u_i \) are the displacements, \( u, v \) and \( w \) along the \( x, y, z \) directions respectively and \( \rho \) is the density. The force components, \( F_i \) in the three directions, are determined by rates of change of stresses along the edges of the unit volume and can be written as

\[ F_i = \frac{\partial F_{ij}}{\partial x_j} \delta x \delta y \delta z \]  

(2.2.7)

Equation (2.2.6) can be written as

\[ \rho \frac{\partial^2 u_i}{\partial t^2} \delta x \delta y \delta z = \frac{\partial T_{ij}}{\partial x_j} \delta x \delta y \delta z \]  

(2.2.8)

Stresses are related to strains through elastic constants and so

\[ \frac{\partial^2 u_i}{\partial t^2} = \frac{\partial \sigma_{ijkl}}{\partial x_j} \]  

(2.2.9)
But for an isotropic solid, this equation reduces to three well known equations

\[ \rho \frac{\partial^2 u}{\partial t^2} = \frac{\partial}{\partial x} \left( \lambda M + 2 \mu S_1 \right) + \frac{\partial}{\partial y} (\mu S_6) + \frac{\partial}{\partial z} (\mu S_2) \]

\[ \rho \frac{\partial^2 v}{\partial t^2} = \frac{\partial}{\partial x} (\mu S_6) + \frac{\partial}{\partial y} (\lambda M + 2 \mu S_2) + \frac{\partial}{\partial z} (\mu S_4) \]

\[ \rho \frac{\partial^2 w}{\partial t^2} = \frac{\partial}{\partial x} (\mu S_5) + \frac{\partial}{\partial y} (\mu S_4) + \frac{\partial}{\partial z} (\lambda M + 2 \mu S_3) \]

where \( M = S_1 + S_2 + S_3 \) \hspace{1cm} (2.2.10)

The most generalised solution was obtained by Christoffel.

The above equation of motion could be written in terms of \( u, v, w \) and \( n, (n = kx + ly + mz \text{ and } k, l, m \text{ are the direction cosines of the normal to the plane}) \) by introducing a series of moduli \( \lambda_{11} \) to \( \lambda_{33} \) which are functions of Lamé constants and the direction cosines \( k, l, m \). Equations 2.2.10 in this system take the form

\[ \rho \frac{\partial^2 u}{\partial t^2} = \lambda_{11} \frac{\partial^2 u}{\partial n^2} + \lambda_{12} \frac{\partial^2 v}{\partial n^2} + \lambda_{13} \frac{\partial^2 w}{\partial n^2} \]

\[ \rho \frac{\partial^2 v}{\partial t^2} = \lambda_{12} \frac{\partial^2 u}{\partial n^2} + \lambda_{22} \frac{\partial^2 v}{\partial n^2} + \lambda_{23} \frac{\partial^2 w}{\partial n^2} \hspace{1cm} (2.2.11) \]

\[ \rho \frac{\partial^2 w}{\partial t^2} = \lambda_{13} \frac{\partial^2 u}{\partial n^2} + \lambda_{23} \frac{\partial^2 v}{\partial n^2} + \lambda_{33} \frac{\partial^2 w}{\partial n^2} \]
where
\[ \lambda_{11} = k^2 (\lambda + \mu) + \mu \]
\[ \lambda_{12} = kl (\lambda + \mu) \]
\[ \lambda_{13} = mk (\lambda + \mu) \]
\[ \lambda_{23} = lm (\lambda + \mu) \]
\[ \lambda_{22} = l^2 (\lambda + \mu) + \mu \]
\[ \lambda_{33} = m^2 (\lambda + \mu) + \mu \]

(2.2.12)

The solution of the above equation (2.2.11) for the isotropic medium indicates three waves propagated, but it is in special cases only that the particle motion will be perpendicular to the direction of propagation of the wave. The three velocities satisfy the relation

\[
\begin{vmatrix}
\lambda_{11} - \nu^2 & \lambda_{12} & \lambda_{13} \\
\lambda_{12} & \lambda_{22} - \nu^2 & \lambda_{23} \\
\lambda_{13} & \lambda_{23} & \lambda_{33} - \nu^2 \\
\end{vmatrix}
= 0
(2.2.13)
\]

Solving the determinant and using the relation for the direction cosines

\[ k^2 + l^2 + m^2 = 1 \]

(2.2.14)

it becomes

\[
(\lambda + 2\mu - \nu^2) (\mu - \nu^2)^2 = 0
(2.2.15)
\]
Thus, irrespective of the orientation employed, there is one wave with velocity $v_1$ and two with velocity $v_t$. 

$$v_1 = \left[ \frac{\lambda + 2\mu}{\rho} \right]^{1/2}$$  \hspace{1cm} (2.2.16) 

and

$$v_t = \left[ \frac{\mu}{\rho} \right]^{1/2} = \left[ \frac{G}{\rho} \right]^{1/2}$$ \hspace{1cm} (2.2.17) 

$v_1$ corresponds to a wave in which the particle vibration is along the direction of propagation (longitudinal wave), whereas $v_t$ corresponds to a wave for which the direction of particle vibration is perpendicular to the direction of propagation, i.e. a transverse wave.

One can obtain the Lamé constants $\lambda$ and $\mu$, the bulk modulus $\mathcal{B}$ and the Young's modulus $Y$ from equations (2.2.16), (2.2.17), (2.2.5) and (2.2.2) respectively.
2.3 COMPOSITE OSCILLATOR TECHNIQUE

In the present study the composite piezo-electric oscillator method originally developed by Balmuth\textsuperscript{70} and Rose\textsuperscript{71} has been set-up and the same has been employed for the measurement of elastic constants.

A. Theory of the method

The principle of the composite oscillator technique is based on the determination of natural frequency of a loaded bar executing either longitudinal or torsional oscillations. An X-cut piezo-electric quartz bar of length 2cm and 3mm square cross-section is taken and the faces normal to the electric axis are silvered. This quartz bar is excited by precision oscillator and resonance is detected by observing the resulting current. As a result of the harmonically varying piezo-electric stress in the quartz, which accompanies the electric field between the electrodes, the system attains a state of forced vibration, the frequency of which is the same as that of the applied potential. Hence from the oscillator frequency the natural frequency of the quartz bar is known. A bar of the sample material of same cross-section but of natural frequency slightly different from that of the quartz bar is cemented to one end of the quartz bar using a suitable adhesive. The frequency of the composite
system is determined. The frequency of the specimen bar can be deduced from a knowledge of the frequencies of the quartz bar and composite system and hence the appropriate elastic constant is calculated using its length and density. The full details of the experiment are described in the following sections.

The theoretical treatment given by Balsamuth is briefly outlined here. The charge that has to be applied between the electrodes of the quartz in order to establish a potential difference \( E \) can be separated into two parts: a part of the ordinary capacitive sort, equal to \( C^1 E \), where as \( C^1 \) is the interelectrode capacitance and the second part required to neutralise the piezo-electric charge produced by the vibrational strain in the quartz, and proportional to the space average value of the strain

\[
Q = C^1 E - k^1 d \frac{\partial S_{av}}{\partial t}
\]  

(2.3.1)

where \( k^1 \) is a geometric constant, \( d \) is the appropriate piezo-electric coefficient and \( S_{av} \) is the average strain.

The current \( I \), which flows through the oscillator is given by

\[
I = C^1 \frac{dE}{dt} - k^1 d \frac{dS_{av}}{dt}
\]  

(2.3.2)

Now \( S_{av} \) is proportional to \( E \), so that the ratio \( z = \frac{E}{I} \) is
independent of $E_0$. $Z$ is the electrical impedance of the oscillator.

The piezo-electric stress in the quartz cylinder is represented by harmonically varying surface tractions over its end faces. The equation of motion of each medium is assumed to be of the form

$$\rho \frac{\partial^2 u}{\partial t^2} = p \left[ 1 + T \frac{\partial}{\partial t} \left( \frac{\partial^2 u}{\partial x^2} \right) \right]$$

(2.3.3)

where $u$ is particle displacement, $\rho$ the density, $p$ the appropriate elastic modulus and $T$ a dissipative coefficient.

The particle displacement in each medium which is a solution of the equation 2.3.3 is given by an expression of the form

$$u = \left[ A \exp \left( \alpha + j \beta \right) x + B \exp \left( \alpha - j \beta \right) x \right] e^{i \omega t}$$

(2.3.4)

where $\omega = 2 \pi f$, $\beta = \omega \left( \rho / \rho \right)^{1/2}$ and $\alpha = 1/2 \omega \beta T$. It is assumed that $T$ is sufficiently small, that $\omega^2 T^2 \ll 1$. The four amplitude coefficients ($A$ and $B$ of each medium) are evaluated by means of the four simultaneous equations which express, respectively, the continuity of stress and displacement at the interface and of stress at the end faces of the oscillator. The strain average $S_{av}$ is calculated from the expression for $u$ in the quartz and the electrical impedance of the oscillator follows from 2.3.2.
The expression for $z$ can be written in the form

$$\frac{1}{z} = j\omega \frac{1}{Z_m}$$

(2.3.5)

In the neighbourhood of resonance, the expression for $Z_m$ is same as that for the electrical impedance of a series resonant circuit. It follows that near resonance a composite oscillator is electrically equivalent to a series electrical network of impedance $Z_m$ shunted by a capacitance $C^1$. If $Z_m$ be written in the form

$$Z_m = R + jX$$

(2.3.6)

then for a two part oscillator

$$R = \left(\frac{K_o}{D}\right) \left[ (M_1 z_1/y_1) (\sec^2 y_1 - P_1) + (M_2 z_2/y_2) (\sec^2 y_2 - P_2) \right]$$

(2.3.7)

$$X = \left(\frac{K_o}{D}\right) (M_1 P_1 + M_2 P_2)$$

(2.3.8)

where $D = (1 - \sec y_2) \left[ 1 - (P_2 y_2^2/M_2) (M_1 P_1) \right]$  

(2.3.9)

$M_i$ = Mass of the cylinder (i=1,2)

$L_i$ = Length of the cylinder

$z_i = L_i \varepsilon_i / y_i = \varepsilon_i / f_i$

$$f_i = (1/2L_i) \left( P_i/\ell_i \right)^{1/2} / P_i = \frac{(\tan y_i)}{y_i}$$
f = frequency of the composite system and subscripts 1 and 2 refer to the specimen and quarts respectively.

The resonant frequencies are those at which the reactive part vanishes.

1.6 \( M_1 P_1 + M_2 P_2 = 0 \)

\[
M_1 \frac{\tan (xf/f_1)}{(xf/f_1)} + M_2 \frac{\tan (xf/f_2)}{(xf/f_2)} = 0 \quad (2.3.10)
\]

Equation 2.3.10 describes the behaviour of quarts alone if \( M_1 = 0 \). Then \( f_1 \), the frequency of the specimen, can be evaluated from the above equation when \( f \) and \( f_2 \) are known.

When the vibrations are longitudinal

\[
f_1 = \left( \frac{Y^{1/2}}{2} \frac{b^{1/2} L}{2} \right) \left[ 1 + x^2 \left( \frac{b^2 + c^2}{2} \right) \right]^{-1} \quad (2.3.11)
\]

and for the shear vibrations

\[
f_2 = \left( \frac{G^{1/2}}{2} \frac{b^{1/2} L}{2} \right) \quad (2.3.12)
\]

where \( Y \) - Young's modulus, \( G \) - rigid modulus

\( L \) - length of the specimen, \( b \) - breadth of the specimen,

\( c \) - thickness of the specimen and \( \sigma \) the Poisson's ratio.

It should be noted that the value of the term containing \( \sigma \) is of the order of \( 10^{-3} \) and so in most cases it is
neglected. However, $\sigma$ may be computed with sufficient accuracy with approximate values of elastic constants obtained when the term is set equal to zero, and this value of $\sigma$ is used to obtain the correction.

The evaluation of $f_1$ demands a precise solution of equation 2.3.10 and this is a laborious procedure as it involves successive approximations. For most purposes the approximate solution

$$f_1 = f + M_2 \frac{(f - f_2)}{M_1} \quad (2.3.13)$$

can be used if $f$, $f_1$, and $f_2$ differ by 10% only. This condition is always satisfied experimentally in the present work.

B. Description of the experimental set-up

The electronic set-up employed to determine the resonant vibration of the piezoelectric oscillator is represented diagramatically in Fig. 2.3.1. Radio frequency output from a heterodyne beat oscillator is fed to a two stage amplifier. The heterodyne-beat oscillator used in the present study is the one designed by Subrahmanyan and has got a high frequency stability. The crystal is connected across a tuned circuit, which is loosely coupled to the output stage of the amplifier. The frequency of the
Fig. 2.3.1 Block diagram of the electrical circuit
Fig. 2.3.2 Amplifier circuitry
The oscillator can be varied from 85 to 180 kHz and can be scanned the entire range of frequency in four settings of the band switch.

To keep the gain of the amplifier constant, regulated voltage is supplied to the amplifier stage. The amplifier circuit is shown in Fig. 2.3.2.

The composite bar and the loading circuit form a parallel resonant circuit and so its impedance is maximum at resonance. A microammeter in series with a crystal diode IN 34 is used as a detector to trace the response of the coupled circuit. The resonant frequency of the oscillator is indicated by a sharp minimum in the microammeter.

C. Transducers

Quartz crystals of two different types are used for the excitation of the specimen under investigation. For longitudinal vibrations, X-cut quartz bars of square cross-section as represented in Fig. 2.3.3 are convenient. The faces perpendicular to the electrical axis are silvered and supported by slips at nodal points as shown in Fig. 2.3.4. The quartz bars used are 3mm square in cross-section.
Fig. 2.3.4 Crystal holders — longitudinal and transverse arrangement
Torsional vibrations are excited by a Y-cut quartz rod of circular cross-section with four electrodes arranged as recommended by Glebe and Scheibe. This arrangement and the necessary crystal holder assembly are shown in Figs. 2.3.3. and 2.3.4 respectively. The length of the crystal is along X-axis and the central lines of the four electrodes make an angle of 45° with respect to Y-axis. When opposite pairs are connected together, a torsional mode can be generated. The field in one direction along Y-axis generates an Xy shear, which moves the top plane with respect to bottom plane. The other pair of electrodes has reverse field and hence the reverse shear is generated in the other half of the crystal. The arrangement rotates the top plane with respect to bottom plane of the crystal and produces a torsional mode.

A set of X-cut-quartz rods, free from defects, ranging in frequency 85 to 180 kHz is prepared from raw quartz. Y-cut quartz rods supplied by Bharat Electronics Limited, Bangalore are employed in the present work. Each specimen under test is to be tried, with different quartz transducers until the composite system has a frequency with in 5 percent of the quartz and the specimen is chosen.

D. Preparation of the specimen bars

The method of cutting and preparing the specimens
depend on the hardness of the material. Since the ferro-alloys employed in the present work are too hard, they are cut with a diamond wheel. Finally they are ground to smooth finish on mechanical grinders.

Cylindrical rods are prepared using the following method. A specimen bar with square cross-section is first cut and then the edges are ground off on a charged flat plate. The final cylindrical form is obtained by grinding the specimen in a split cylindrical die. The length of the specimens are measured to an accuracy of 0.001 cm using a vernier microscope. The densities are determined by hydrostatic method, using an analytical balance.

E. Ferro-alloy samples

Ferrochrome (Fe-63%, Cr-30%, Mn-0.6%, P-6%) and Ferrosilicon (Fe-85%, Ni-9.9%, Si-4.27%, C-0.58%) used in the present study have been obtained from the Facor Engineering Corporation, Bombay.

F. Cementing

The composite oscillator is formed by cementing the quartz rod to the specimen of identical cross-section. The adhesive used for cementing, consists of a paste containing one part by weight of calcium carbonate and
five parts by weight of sodium silicate. The composite system works satisfactorily after it is kept under pressure for about twenty four hours at room temperature. The following precautions are followed in cementing the specimens.

(i) A thin layer of adhesive is to be used. It was pointed out by Balsamuth that the effect of cement is negligible provided the frequency of the specimen and the composite system are within 5 percent. This condition is of course, satisfied throughout the present work.

(ii) It is very important to see that a minimum amount of cement is left off adhering to the sides of the oscillator. The effect of such excess material is to load the oscillator and hence the velocity versus temperature curve is depressed parallel to itself though to a small extent.

G. Temperature study

The composite bar with the holder is placed at the centre of an electric furnace. It consists of a thick fused silica tube on which is wound a nichrome wire of sufficient length at regular intervals. The nichrome wire is completely hidden under a thick coating of asbestos paste and ever which an asbestos rope of 2 mm thick is wound so that the furnace is not directly exposed to the atmosphere.
resulting in the radiation of heat outwards. The temperature of the furnace is raised by passing an electric current through the nichrome wire and is controlled by an autotransformer. By passing a fixed amount of current for a sufficiently long interval of time (≈ 30 minutes), a steady state will be reached. The temperature is measured by a calibrated iron constantan thermo-couple to an accuracy better than ± 0.5°C and the couple is placed very close to the specimen. The temperature dependence of elastic constants of the ferro-alloys are studied in the temperature range 30° - 360°C.

H. Accuracy of the method

The chief source of error is non-uniformity of the cross-section in the specimens. Such non-uniformity produces a small shift parallel to itself of the entire velocity versus temperature curve. The estimated uncertainty in the accurate values of the directly measured velocities is one percent. However, greater accuracy can be achieved in making relative measurements of the moduli rather than their absolute values. These factors have been taken into account while fitting the curves to observed points.

I. Determination of Curie temperature (Tc)

All ferromagnetic substances exhibit the phenomena of
hysteresis. At the Curie temperature the ferromagnetic material transforms into paramagnetic state and looses the property of hysteresis. This behaviour has been utilised to determine the Curie temperature of the specimens under study.

The sample material is cut in the form of a toroid and it has been used as the core to display the hysteresis of the sample on the cathode ray oscilloscope. Two windings are arranged on the specimen core; one the primary and the other secondary. A resistor \( R_s \) is connected in series with the primary winding and the voltage drop across it is used as the deflection for the X-plates of CRO as indicated in Fig. 2.3.5.

The vertical deflection is proportional to magnetic induction \( B \). The secondary windings on the specimen core serves for this, the voltage being

\[ V_s \propto \frac{dB}{dt} \]

This voltage is electrically integrated by feeding it via a large resistor \( R \) (here 1 M\( \Omega \)) to a capacitor \( C \) (here 4\( \mu \)F) in such a way that \( R \gg \frac{1}{\omega C} \).

The secondary windings is divided into two parts so that both ends lie outside and have approximately the same
Fig. 2.3.5 Experimental setup for the determination of $T_c$. 

- Furnace
- Specimen cone
- Cathode ray oscilloscope
- N
- $N_p$
- $N_d$
- $R$
- $C$
- $R_s$
- $S$
capacitance to the primary winding and to the core. Screening is provided between primary and secondary so that the secondary voltage is entirely due to magnetic induction.

The toroid is placed in the furnace (details discussed in section 2.3 C) and the temperature is raised. As the temperature increases the sample material goes from ferromagnetic state to paramagnetic state and hysteresis loop becomes a straight line. The temperature at which the hysteresis disappears is the Curie temperature.
2.4 ELASTIC BEHAVIOUR OF ALUMINIUM AND BISMUTH - COMPARISON WITH LITERATURE DATA

The classical theory of elasticity states that stress and strain are uniquely related to each other. A solid is said to be perfectly elastic only when the above condition is satisfied. But in the case of metals there are many deviations from the perfect elastic behaviour even for small stresses. This has been pointed out by Weber as early as 1825 in his investigation on galvanometer suspensions. The suspension, on the release of couple, did not come to zero at once but approaches asymptotically. Such a behaviour was named as elastic after-effect. Other effects which are shown by real solids are, internal friction, stress relaxation and variation of elastic moduli with frequency. Many of these anelastic effects are attributed to the nature of grain boundaries in metals.

To test the reliability of the composite oscillator technique developed in the present work, measurements on two standard polycrystalline specimens (Aluminium and bismuth) have been carried out as a function of temperature. The results on these two samples are presented in this section.

Aluminium

It is a bluish white metal with bright lustre. This
belongs to HI group of the periodic table. It is praised for its lightness, high electrical and thermal conductivities (next to copper), high ductility, resistance to corrosion and for its high reflectivity of heat and light. Its dust is used in paint industry and the metal can yield extremely thin foils which are used as wrappers in industry. Its alloys (Duraluminum) have got wide application in air-craft industry.

The natural frequencies of the metal, both longitudinal and torsional, are measured as a function of temperature in the range 30-300°C using the composite piezo-electric oscillator. The thermal expansion data is taken from the literature. This data have been used to obtain the longitudinal and shear velocities in the specimen. The elastic constants, Young's modulus and rigidity modulus, are presented in Table 2.4.1 along with the literature data. The agreement between the two sets of values is quite good. The variation of longitudinal velocity and torsional velocity with temperature are shown in figures 2.4.1 and 2.4.2 respectively along with the results of Sutton. The examination of the Fig. 2.4.1 reveals that increase in temperature results in a decrease in velocity and there is a sudden drop in the longitudinal velocity versus temperature graph at \( \approx 240°C \). A similar behaviour has been observed in the torsional velocity versus temperature (Fig.2.4.2)
Table 2.4.1 Variation of elastic moduli with temperature in aluminium

<table>
<thead>
<tr>
<th>S.No.</th>
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<th>G x 10^{-11}</th>
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(Unit: dynes cm\(^{-2}\))
Fig. 24.1 Variation of longitudinal velocity with temperature in aluminium

- ○ Present study
- + Sutton
Fig. 2.4.2 Variation of torsional velocity with temperature in aluminium
Graph at \(260^\circ C\). Ke\(^{35}\) has determined the internal friction and rigidity modulus of fine grained polycrystalline aluminium, using dynamic method from room temperature to \(450^\circ C\). A sudden drop in temperature versus rigidity graph was observed at \(200^\circ C\). This behaviour of the polycrystalline aluminium has been explained by Ke as due to the viscous slip along the grain boundaries. The temperature at which the curve changes slope corresponds to the temperature at which the relaxation time associated with the slip is comparable to the period of vibration.

Similar observations have been reported by Ke\(^{35}\) in \(\alpha\)-brass and \(\alpha\)-iron\(^{36}\). Yousef and Kamel\(^{37}\) and Subrahmanyan\(^{75}\) have reported similar phenomena in cadmium. In the single crystals of cadmium no such phenomena has been noticed.

Recent measurements of internal friction\(^{38}\) for aluminium at a frequency 120 kHs show a peak at \(290^\circ C\). This value is in reasonable agreement with the present values 240\(^\circ C\) at 130 kHs and 260\(^\circ C\) at 144 kHs. The dependence of transition phenomena on frequency suggests that the mechanism involved is, stress relaxation at grain boundaries.

Bismuth:

Bismuth belongs to V group of the periodic classification. The single crystal of bismuth belongs to rhombohedral system.
Fig. 2.4.3 Temperature variation of longitudinal velocity in bismuth
Fig 2.4.4  Temperature variation of torsional velocity in bismuth
The longitudinal and torsional frequencies are determined as a function of temperature in the range $30^\circ-180^\circ$C for this metal using the composite oscillator technique detailed in section 2.3. The longitudinal and torsional velocities are evaluated from the natural frequencies. The thermal expansion data is taken from literature. The elastic constants - Young's modulus and rigidity modulus are presented in Table 2.4.2 along with results of Reddy. There is a good agreement between the two sets of values. The longitudinal and torsional velocities are diagrammatically represented in Figs. 2.4.3 and 2.4.4 respectively along with the data of Reddy for comparison. Both the longitudinal and shear velocities decrease with increase in temperature as can be seen from the Figs. 2.4.3 and 2.4.4.
Table 2.4.2. Temperature variation of Young's modulus and rigidity modulus of bismuth.

<table>
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(Units: dynes, cm\(^{-2}\))