

## Elastic properties of gallium substituted Bi (Pb)-2212 superconducting system at 300 K

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### Abstract

The elastic behaviour of  $\text{Bi}_{1.7-x}\text{Ga}_x\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_1\text{Cu}_2\text{O}_y$  ( $x = 0.0\text{--}0.3$ ) superconducting system has been studied using ultrasonic pulse transmission technique at 1 MHz (300 K). The values of Young's modulus, rigidity modulus, longitudinal modulus, bulk modulus, Poisson's ratio and Debye temperature of the specimens are computed from longitudinal and shear wave velocities and corrected to zero porosity. The strength of interatomic bonding is found to increase on gallium substitution, in consistency with Al-substituted Bi (Pb)-2223 system. The applicability of the heterogeneous metal mixture rule for estimating elastic constants and critical temperature has been tested, and agreement with the experimental results validates the method used.

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### 1. Introduction

Sound is strain wave propagating through a solid. The velocity of longitudinal and transverse elastic waves thus produced is a characteristic feature of a solid. The basic idea is that if there is any distortion of the solid from its equilibrium shape, the average separation of the atoms within the solid is no longer optimal. Some atoms will be too close to their neighbours, and some too far apart. In either case there will be a restoring force, which will act to return the atoms to their equilibrium separations. The dynamics of the elastic wave will be affected by the way the solid responds to the restoring force. The two factors most critical in determining this response are the restoring force per unit displacement (the natural 'springiness' of the substance), and the density of the substance. The restoring force on a small region of a solid depends on the type of distortion

(strain) that has taken place during synthesis process. The parameters that describe the restoring force per unit strain are known as the elastic moduli of a substance. In the present work we have employed ultrasonic pulse transmission technique as a tool to get idea about such stress/strain ratio.

After the discovery of high  $T_c$  superconductivity [1], several investigations were started almost simultaneously to understand the mechanism responsible for the phenomenon. Number of researchers [2–8] have reported the existence of elastic anomalies in the temperature range from room temperature to the superconducting transition temperature ( $T_c$ ), signifying the presence of lattice instabilities. As the lattice instabilities are manifest well in the elastic studies, a number of investigations were undertaken with a view to know the correlation between high value of  $T_c$  and structural instabilities.

When ceramic superconductors are subjected to high magnetic field, large stresses developed in the material. It is always desirable to have general idea of elastic moduli values that represent mechanical strength, fracture

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toughness and thermal shock resistance. From fundamental research points of view the knowledge of elastic moduli of any polycrystalline material elucidate the nature of binding forces and also help to understand thermal properties of the solids when we think about the application of any polycrystalline material, besides the knowledge of its magnetic and electric response, the elastic properties help to decide suitability of the material for specific application.

The Young's modulus of a superconductor is an important parameter in determining critical grain sizes above which microcracking will occur due to anisotropic thermal stresses that arise during processing. This phenomenon of microcracking has been determined to cause a decrease in the attainable critical current density in bulk superconductors. The bulk modulus of a solid influences the speed of sound and other mechanical waves in the material. On the other hand Debye temperature of a superconducting material may provide information about the role of phonons in superconducting mechanism.

In the present work an attempt is made to see the effect of  $\text{Ga}^{3+}$  substitutions for  $\text{Bi}^{3+}$  on elastic behaviour of  $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_1\text{Cu}_2\text{O}_y$  superconducting system at 300 K. The composition  $\text{Bi}_{1.7}\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_1\text{Cu}_2\text{O}_y$  was chosen as a starting composition because it has highest  $T_c$  ( $\sim 65$  K) and Pb plays very important role in stabilizing the phase. The substitution of Ga at Bi-site is expected to provides stability to the structure, improves conductivity value with nominal reduction in critical temperature. It is found that gallium substitution enhances the elastic properties of Bi(Pb)-2212 system in consistency with the results obtained for aluminium substituted Bi(Pb)-2223 system [9].

## 2. Experimental details

A series of samples having stoichiometric compositions  $\text{Bi}_{1.7-x}\text{Ga}_x\text{Pb}_{0.3}\text{Sr}_2\text{Ca}_2\text{Cu}_3\text{O}_y$  Bi(Pb)-Ga(2212) ( $x = 0.0, 0.1, 0.2$  and  $0.3$ ) were synthesized by the solid state route using highly pure (99.99%)  $\text{Bi}_2\text{O}_3$ ,  $\text{CaCO}_3$ ,  $\text{CuO}$ ,  $\text{Sr}_2\text{CO}_3$ ,  $\text{Ga}_2\text{O}_3$  and  $\text{PbCO}_3$ . The powders after thorough mixing and grinding, the mixture was then heated about  $800^\circ\text{C}$  for 24 h followed by two more similar treatment with intermediate grinding. The calcinated powders after pressing into disc were sintered at  $810^\circ\text{C}$  for 24 h and slowly cooled to room temperature at the rate of  $1^\circ\text{C}/\text{min}$ . The structural characterization of all the samples was carried out at 300 K by X-ray diffraction technique using  $\text{Cu K}\alpha$  radiation.

In general sintered ceramic samples have large number of closed and open pores inside and on their surface hence the ordinary method of determination of a bulk density ( $\rho$ ) and porosity by hydrostatic method is found to give spurious results. In such cases immersion method gives fairly good and reliable values. In the present investigation, the bulk density ( $\rho$ ) of Bi (Pb)-Ga (2212) samples were determined

Table 1

Molecular weight ( $M$ ), X-ray density ( $\rho_x$ ), bulk density ( $\rho$ ) and pore fraction ( $f$ ) for Bi(Pb)-Ga(2212) system

Ga-content ( $x$ )	$M (\times 10^{-3})$ (kg)	$\rho_x (\times 10^{-3})$ ( $\text{kg}/\text{m}^3$ )	$\rho (\times 10^{-3})$ ( $\text{kg}/\text{m}^3$ )	$f$
0.0	887.82	6.145	5.85	0.048
0.1	912.47	6.119	5.66	0.075
0.2	943.56	6.215	5.50	0.115
0.3	971.43	6.460	5.42	0.161

by the immersion method and the values of pore fraction [ $f = 1 - (\rho/\rho_x)$  ( $\rho_x$ : X-ray density)] thus obtained are presented in Table 1.

The ultrasonic pulse transmission technique [10] was used for the measurement of longitudinal wave velocity ( $V_l$ ) and shear wave velocity ( $V_s$ ) at 1 MHz. The r.f. pulse generated by a pulse oscillator was applied to quartz transducer. The acoustic pulses were converted into electrical signals by the receiving transducer. The output signal was displayed on a digital taxtronic 2230 oscilloscope. The difference in time ( $\Delta T$ ) between two overlapping received pulse train was noted with the help of timer. The sound velocity was measured using the equation  $V = L/\Delta T$  where  $V$ , sound velocity,  $L$ , length of the superconducting specimen, and  $t$ , time. The accuracy of the sound velocity measurement was  $\pm 0.5\%$ .

A number of research reports is available in the literature describing various aspects of elastic behaviour of different superconducting systems [2–9,11]. To our knowledge no work has been reported on elastic properties of gallium substituted Pb-containing Bi-2212 superconducting series.

## 3. Results and discussion

The powder X-ray diffraction patterns for all the composition mainly reveal the Bi-2212 phase. The Bragg's reflection peaks were indexed using DEBYE & INDEX computer program. It was observed that all the samples remain tetragonal phase with negligible orthorhombic distortion [12]. The X-ray density ( $\rho_x$ ) values calculated from corresponding molecular weight (Table 1) and lattice constant values are summarized in Table 1. The detailed analysis on structural and superconducting properties of Bi(Pb)-Ga(2212) system will be presented elsewhere.

The elastic constants of the materials can be determined by studying propagation of ultrasonic waves through those materials. This provides better understanding of the behaviour of the engineering materials. The elastic constants of the material are related with the fundamental solid state phenomenon such as specific heat, Debye temperature and Grunesian parameter. The elastic constants of a material can be determined by measuring the velocity of the longitudinal ( $V_l$ ) and shear waves ( $V_s$ ) [13].

Table 2

Longitudinal velocity ( $V_l$ ), transverse velocity ( $V_s$ ), mean sound velocity ( $V_m$ ) and Debye temperature ( $\theta$ ) for Bi(Pb)-Ga(2212) system

Ga-content	$V_l$ (m/s)	$V_s$ (m/s)	$V_m$ (m/s)	$\theta$ (K)
0.0	3087	1909	2105	244.6
0.1	3037	1896	2089	237.9
0.2	2954	1846	2034	226.8
0.3	2831	1767	1947	214.0

The ultrasonic velocities and elastic constants are related as given by the following equations [10]

Longitudinal modulus ( $L$ ) :  $\rho(V_l)^2$

Rigidity modulus ( $G$ ) :  $\rho(V_s)^2$

Bulk modulus ( $B$ ) :  $L - \frac{4}{3}G$

Poisson's ratio ( $\sigma$ ) :  $\frac{3B - 2G}{6B + 2G}$

Young's modulus ( $E$ ) :  $(1 + \sigma)2G$

The acoustic Debye temperature of materials used to explain the well known solid state problem like lattice vibrations is determined using ultrasonic velocity. The relation is given as,

$$\theta = \frac{h}{k_B} \left[ \frac{3N_A P}{4\pi V} \right]^{1/3} V_m$$

where  $h$  is the Plank constant,  $k_B$  the Boltzmann constant,  $N_A$  the Avogadro number,  $V$  the volume calculated from the effective molecular weight and the density (i.e.  $M/\rho$ ),  $P$  the number of atoms in the molecular formula (i.e. 15 in the present case) and  $V_m$  the mean sound velocity defined by the relation

$$V_m = \left[ \frac{2/V_s^3 + 1/V_l^3}{3} \right]^{-1/3}$$

The values of ultrasonic wave velocity, elastic moduli and Debye characteristic temperature are summarized in Tables 2 and 3. The elastic constants are related to interatomic forces, co-ordination changes, etc; and also the impact shock, fracture and crack growth [14]. For porous materials like cast metal, ceramics and most composites, the relation between elastic moduli and velocity are complex. Elastic moduli in these materials are function of pore size, shape and orientation. The other microstructural factors such as grain shape, grain boundaries, texture and precipitates

Table 3

Longitudinal modulus ( $L$ ), bulk modulus ( $B$ ), rigidity modulus ( $G$ ), Young's modulus ( $E$ ) and Poisson's ration ( $\sigma$ ) (porous) for Bi(Pb)-Ga(2212) system

Ga-content	$L$ (GPa)	$B$ (GPa)	$G$ (GPa)	$E$ (GPa)	$\sigma$
0.0	55.75	27.32	21.32	50.74	0.19
0.1	52.20	25.07	20.35	48.03	0.18
0.2	47.99	23.00	18.74	44.23	0.18
0.3	43.44	20.88	16.92	39.93	0.18

have pronounced effect on the relation between elastic moduli and velocity [15–16].

In general, the samples prepared by solid state reaction method are found to be porous. The interconnected pores of a sintered material provide an opportunity for free movement of oxygen into the bulk of a sample, so oxygen is distributed throughout the volume of the sample. Furthermore, it also relieves the internal stresses resulting in obtaining less constrained polycrystalline materials. Therefore, porosity plays an important role in governing certain important physical properties. In order to improve the mechanical properties of ceramic materials it is essential to understand the relationship between porosity and its elastic behaviour. The measured elastic moduli do not have much significance unless they are corrected to zero porosity. In engineering practice, the elastic constants often used are the Young's modulus, rigidity modulus and Poisson's ratio. As the superconducting specimens under study are porous ( $f \approx 0.05$ – $0.16$ ), the values of wave velocity and elastic constants are corrected to zero porosity using the formula [17–19]:

$$V_l = V_{l0}(1 - C_l f)$$

$$V_s = V_{s0}(1 - C_s f)$$

$$\frac{1}{E_o} = \frac{1}{E} \left[ 1 - \frac{3f(1 - \sigma)(9 + 5\sigma)}{2(7 - 5\sigma)} \right]$$

$$\frac{1}{G_o} = \frac{1}{G} \left[ 1 - \frac{15f(1 - \sigma)}{(7 - 5\sigma)} \right]$$

$$\sigma_o = \left( \frac{E_o}{2G_o} \right) - 1$$

$$L_o = \rho x V_{l0}^2$$

$$B_o = L_o - \frac{4}{3}G_o$$

where  $C_l$  and  $C_s$  are constants of the material. The subscript 'o' denotes non-porous materials. The exact expressions for the constants are as follows:

$$C_l = \left( \frac{1}{2} \right) \left[ \frac{C_E + 2C_\sigma \sigma_o^2 (2 - \sigma_o)}{(1 - \sigma_o)(1 + \sigma_o)(1 - 2\sigma_o) - 1} \right]$$

$$C_s = \frac{1}{3}$$

$$C_E = \frac{1}{18}(29 + 11\sigma_o) \text{ and}$$

$$C_\sigma = \frac{5}{9} + \frac{11}{18}\sigma_o - \frac{1}{18\sigma_o}$$

The corrected values of elastic parameters are given in Table 4.

It can be seen from the Table 4 that, longitudinal modulus, bulk modulus (up to 0.2), Young's modulus and rigidity modulus increase with increasing Ga-content ( $x$ ). Following Wooster's work [20], the variation of elastic constants with increasing Ga-concentration for Bi in Bi(Pb)-Ga(2212) superconducting system may be

Table 4

Wave velocity and elastic moduli (corrected to zero porosity) for Bi(Pb)-Ga(2212) system

Ga-content	$V_{lo}$ (m/s)	$V_{so}$ (m/s)	$L_o$ (GPa)	$B_o$ (GPa)	$G_o$ (GPa)	$E_o$ (GPa)	$\sigma_o$
0.0	3606	1947	79.90	48.45	23.59	56.12	0.19
0.1	3719	1941	84.63	52.66	23.98	56.43	0.18
0.2	3750	1877	87.40	54.84	24.42	57.41	0.18
0.3	3500	1811	79.13	45.40	25.03	58.84	0.18

interpreted in terms of interatomic bonding. Thus, it can be inferred from the increase in elastic moduli with concentration ( $x$ ) that the interatomic bonding between various atoms is being strengthened continuously. The strength of interatomic bonding and thus elastic constants value basically depends up on the length of inter atomic bonding and type of cations involved in bond formation. Earlier it has been observed that, substitution of small amount of Pb for Bi in Bi-2212 and Bi-2223 systems enhance the superconducting properties but deteriorate the elastic properties [11]. On the other hand when Bi- is replaced by  $Al^{+3}$  ions in the Bi(Pb)-2223 system [9] it improves the elastic behaviour of the system. This finding can be explained on the basis of change in length of interatomic bonding. When  $Bi^{3+}$  ions with ionic radius of 0.96 Å are replaced by  $Pb^{2+}$  ions with larger ionic radius (1.20 Å) it increases the bond length and as a result strength of interatomic bonding is expected to decrease. On the other hand replacement of  $Bi^{3+}$  ions by smaller  $Al^{+3}$  ions (0.51 Å), reduces the length of interatomic bonding, as a result one can expect increase in strength of interatomic bonding and in turn elastic moduli values. In the present case  $Ga^{+3}$ -ions with smaller ionic radius (0.62 Å) replace the  $Bi^{+3}$  ions, as expected, strength of interatomic bonding and magnitude of elastic constants increase with increasing Ga-concentration in the system (Table 4).

It is interesting to note that elastic moduli corrected to zero porosity ( $B_o$ ,  $E_o$  and  $G_o$ ) (Table 4) show inverse trend as compared to porous values (Table 3) as function of Ga-content ( $x$ ), i.e. they are found to increase with Ga-content. This also leads to conclusion that porosity plays a very important role in governing elastic properties of polycrystalline oxide compositions. The control over pore fraction may results in tailoring the elastic behaviour of the materials. The observed decrease in Debye temperature ( $\theta$ ) (Table 2) with gallium concentration suggested that lattice vibrations are enhanced due to Ga-substitution. The magnitude of elastic constants and Debye temperature (Table 2) are consistent with other Bi-2212 based superconducting systems [11]. The value of Poisson's ratio is found around 0.18, for all the compositions. This value lies in the range from  $-1$  to  $0.5$  which is in conformity with the theory of isotropic elasticity.

It is always desirable to have a general idea of elastic moduli values before synthesis and characterization of the

material in order to tailor the properties. Recently, we have developed and successfully implemented an heterogeneous-metal-mixture rule (MMMR) to estimate elastic constants of various spinel ferrites [21–23], garnets [24], La-based perovskites [25] and superconductors [9]. The aim of the present work is to test the validity of this model for estimating elastic moduli of various Bi(Pb)-Ga(2212) superconducting compositions and to compare the results with experimentally obtained values. The basic idea behind this model is that: the density, longitudinal and transverse wave velocities and thus elastic moduli and Debye characteristic temperature of such polycrystalline oxide compositions, depend upon the density and elastic wave velocity of individual metallic cations present in system.

According to this model “The elastic constant and Debye temperature value of polycrystalline oxide material ( $K_{pm}^*$ ) is equal to the average stoichiometric compositional addition of elastic constant values of metallic elements present in the material.” The elastic moduli such as Young's modulus, bulk modulus, rigidity modulus and Debye temperature values of various metallic elements are taken from the literature [26–28] and are used to estimate  $K_{pm}^*$ . The elastic constant value, to be estimated, for a given superconducting system can be given as:

$$K_{pm}^* = \frac{1}{n} \sum_{i=1}^{\infty} C_{in} K_n$$

where  $K_{pm}^*$  is the either bulk modulus, Young's modulus, rigidity modulus or Debye temperature of the composition to be estimated,  $n$  the total concentration of metallic cations involved in the chemical formula of the polycrystalline material ( $n = 7$  in the present case),  $C_{in}$  the concentration of the  $n$ th cation in the formula unit while  $K_n$  is the corresponding modulus of the metallic element.

The values of elastic moduli ( $B^*$ ,  $E^*$ ,  $G^*$ ) and Debye temperature ( $\theta^*$ ) obtained from MMM rule are summarized in Table 4. The results of our calculations are in conformity with the elastic constant and Debye temperature values obtained from ultrasonic pulse transmission technique with reasonable accuracy (Table 4). This finding is interesting in the sense that the elastic moduli of such oxide compositions can be estimated from the elastic constants of metallic elements present in the system and surprisingly the oxygen does not seem to play significant role for assigning elastic constants of these oxide compositions. Further, investigation in this direction is in progress.

We have extended the use of Debye temperature values calculated from MMM rule for estimating superconducting transition temperature ( $T_c$ ) for Bi(Pb)-Ga(2212) system and compared the values with those deduced experimentally from thermal variation of d.c. resistivity measurements [12]. The formulation is based on Debye temperature values for pristine,  $\theta(0.0)$ , substituted compositions,  $\theta(x)$ , obtained from MMM rule (Table 5) and transition temperature found

Table 5  
Elastic constants and Debye temperature (MMMR) for Bi(Pb)-Ga(2212) system

Ga-content	$B^*$ (GPa)	$G^*$ (GPa)	$E^*$ (GPa)	$\theta^*$ (K)
0.0	54.55	19.67	47.20	206.20
0.1	54.92	20.04	48.18	209.10
0.2	55.28	20.41	49.15	212.00
0.3	55.56	20.78	50.12	214.86

Table 6  
Superconducting transition temperature ( $T_c$ ) for Bi(Pb)-Ga(2212) system

Ga-content	$T_c(0.0)$ (K) resistivity [12]	$T_c$ (K) MMMR	Error (%)
0.0	65.0	–	–
0.1	62.5	64.09	2.48
0.2	61.8	63.19	2.20
0.3	60.3	62.29	3.19

experimentally for un-substituted composition  $T_c(0.0)$  [12]. It is given by:

$$\left| \frac{T_c(0.0) - T_c(x)}{\theta(x) - \theta(0.0)} \right| = K = \text{constant.}$$

where  $T_c(x)$  is the transition temperature to be estimated for substituted composition. The value of  $K$  is determined from the ratio of  $T_c(0.0)$  to the  $\theta(0.0)$ . In the present case it is found to be 0.31523. The value of  $T_c(x)$  for  $x = 0.1, 0.2$  and  $0.3$  estimated from above equation is listed in Table 5. This formula is analogous to the known relation for superconducting materials:  $(T_c/\theta) = \text{constant}$  [28]. For the sake of comparison and to facilitate the discussion the values of  $T_c(x)$  ( $x = 0.1, 0.2$  and  $0.3$ ) deduced from resistivity measurements, are also given in the Table 5. There it is quite reasonable agreement between the two. When no such simple formulation is available in the literature for theoretical estimation of superconducting transition temperature ( $T_c$ ), this will provide a great tool for the same (Table 6).

#### 4. Conclusions

Based on results of elastic moduli determination through the ultrasonic pulse transmission technique and the heterogeneous metal mixture rule for Bi(Pb)-Ga(2212) superconducting system at 300 K, it is concluded that:

- The observed increase of elastic constants with gallium substitution suggests strengthening of interatomic bonding.
- The elastic moduli corrected to zero porosity are in good agreement to those determined through the metal-mixture rule, confirming the validity of the method.
- In polycrystalline oxide compositions oxygen anions play an important role for structure formation but less decisive to the elastic constants values; further strength

of a given material is mainly governed by the strength of cation–cation bonding rather than strength of cation–anion bonding in the system

- The superconducting transition temperature for substituted compositions, can be estimated theoretically from the corresponding Debye temperature values and  $T_c$  of pristine composition.

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#### Appendix A. Illustrative calculations for elastic constants determination through MMM rule

- Bi<sub>1.5</sub>Ga<sub>0.2</sub>Pb<sub>0.3</sub>Sr<sub>2</sub>Ca<sub>1</sub>Cu<sub>2</sub>O<sub>y</sub> ( $x = 0.2$ )

$$B_{\text{pm}}^* = \frac{1}{7}[(1.5)31 \text{ GPa} + (0.2)56.9 \text{ GPa} + (0.3)46 +$$

$$2(11.6) \text{ GPa} + (1)17 \text{ GPa} + (2)140 \text{ GPa}] = 55.28 \text{ GPa}$$

MMMR (ref. [27])

$$B_o = 54.84 \text{ GPa} \quad \text{UPT}$$

- Bi<sub>1.7</sub>Pb<sub>0.3</sub>Sr<sub>2</sub>Ca<sub>1</sub>Cu<sub>2</sub>O<sub>y</sub> ( $x = 0.0$ )

$$G_{\text{pm}}^* = \frac{1}{7}[(1.7)12 \text{ GPa} + (0.3)5.6 \text{ GPa}$$

$$+ (2)6.1 \text{ GPa} + (1)7.4 \text{ GPa} + (2)48 \text{ GPa}] = 19.67 \text{ GPa}$$

MMMR (ref. [26])

$$G_o = 23.59 \text{ GPa} \quad \text{UPT}$$

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