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Collective excitation modes in the intermediate and superconducting states of doped and undoped indium and lead

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Abstract. Ultrasonic attenuation was studied in pure In, In + 0·003 at. % Pb, pure Pb and Pb + 0·003 at. % In in the intermediate states (for the magnetic fields 0·7 H_c and 0·9 H_c) and superconducting states, for frequencies varying from 9·9 to 29·7 MHz, in the temperature range 4·2 to 1·4 K. Collective excitation modes were observed in both the states for all the samples. There exist two distinct phases in the intermediate state but only one phase in the superconducting state in all the samples. The first phase was dependent on the magnetic field and independent of the concentration and nature of the dopant. The second phase was independent of the magnetic field and dependent essentially on the concentration of vacancies and marginally on the concentration of the dopant. The origin of the two phases has been discussed.

Keywords. Collective excitation mode; superconductivity; indium; lead; ultrasonic attenuation.

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1. Introduction

The BCS theory (Bardeen and Schriffer 1957) explain superconductivity on the basis of single-electron phonon interaction. The rapid drop in ultrasonic attenuation of superconductors below the transition temperature follows from the BCS theory by postulating single-electron phonon interaction. Besides the concept of single-electron phonon interaction, the possibility of collective excitation modes in the excitation spectrum of superconductors was postulated by Bogoliubov (1957) and Anderson (1958). Anderson and Bogoliubov showed that there are low frequency collective excitation modes for the electrons in the superconducting state which correspond to pressure waves in a neutral Fermi gas. The concept of a pressure associated with the superconducting state was first considered in regard to the electromagnetic stresses at the boundary of a superconducting region. A change in volume δV of the superconducting phase, which involves no change in free energy density f changes the total free energy F by an amount

$$\delta F = -(f_n - f_s)\delta V.$$

The normal material is converted into superconducting material in δV. There is an effective pressure $P = -\delta F/\delta V$. Collective type excitation modes for the electrons in the superconducting state can be produced under the influence of the effective pressure P. Ultrasonic attenuation studies by Claiborne and Einspruch (1960) in Nb-Zr alloys,

by Olsen *et al* (1966) on high purity single crystals of tin by Chaudhuri and Jain (1969) on impure and strained single crystals of tin have revealed oscillatory behaviour in the superconducting state which cannot be explained in terms of single electron phonon scattering process.

Similar behaviour has been reported by Mahajan and Chaudhuri (1972, 1974), Agarwal and Chaudhuri (1976) and Chaudhuri and Agarwal (1979) in the intermediate state of superconductors. The temperature-dependent oscillatory attenuation peaks have been observed by Chaudhuri and Singh (1979, 1980, 1982), both in the intermediate and superconducting states. This oscillatory attenuation observed by different workers in the superconducting and intermediate states, gives direct evidence for the existence of collective excitation modes with energies lying within the energy gap. They also give evidence for the interaction of collective excitation modes of the electron gas with the lattice phonons. Claiborne and Einspruch (1963) proposed a phenomenological model to explain the temperature-dependent oscillatory attenuation observed in the superconducting state. They assumed formation of some superconducting microstructures near localized strain sites or around impurity centers. These microstructures are separated from the bulk superconductor with distinct phase boundaries. Under the influence of a hydrodynamic pressure wave, these boundaries undergo isothermal displacements. At equilibrium condition, the displacement of the boundary would lead to a linear restoring force and, hence, to a set of temperature-dependent modes of vibration. The model interprets the presence of absorption peaks due to the exchange of energy from a compressional ultrasonic wave to the lattice phonons via the excitation modes. The maximum exchange of energy occurs from the ultrasonic wave to the electron gas when the sound frequency ν_s is equal to the frequency of one of the harmonic standing wave modes i.e. absorption peaks would occur at temperatures for which $\nu_s = \nu_n$. The condition for resonance is given by,

$$\nu_s = \nu_n = n\nu_0(1 - aT^2 + bT^4)^{1/2} \quad \text{for fixed boundaries,}$$

$$\nu_s = \nu_n = (2_{n+1})\nu_0(1 - aT^2 + bT^4)^{1/2} \quad \text{for free boundaries,}$$

where T is the temperature, a, b and ν_0 are constants, n is a positive integer, ν_s is the sound frequency and ν_0 is the fundamental frequency for a particular excitation mode in the superconducting phase at absolute zero. Its value is given by,

$$\nu_0 = C_0(2\,Z_s)^{-1}, \tag{1}$$

Z_s being the thickness of the superconducting region and C_0 is a constant. In the intermediate state, as the bulk is divided into alternate superconducting (S) and normal (N) layers, with distinct phase boundaries, boundary effects, besides S and N layers, are also likely to contribute to the total attenuation. Several workers (1964, 1966, 1968, 1972) have essentially discussed the effect of magnetic field on the ultrasound absorption in the intermediate state with electron mean free path l larger than the thickness of normal layers and $T < T_c$. It has been shown that positions of absorption peaks remain unaffected when attenuation due to N layers and boundary effects is subtracted from the total attenuation in the intermediate state. One can, therefore, utilize the total attenuation absorption peaks to calculate a, b and ν_0 in the intermediate state using Claiborne and Einspruch theory.

The structure of the intermediate state is greatly influenced by the magnetic field and the surface energy. According to Kuper's relation (1951),

$$Z_s/d = 1/(2h)^{1/2}\,(\Delta/d_1)^{1/2} \tag{2}$$

where Z_s is the superconducting laminar thickness, $h = H/H_c$, H being the applied magnetic field and H_c the critical field, Δ is the surface energy parameter and $\Delta \approx \xi - \lambda_D$, ξ being the coherence length and λ_D the penetration depth. Any change in Δ will change Z_s; then according to equation (1) will effect ν_0, the fundamental frequency. According to Chaudhuri and Singh (1979, 1982) two distinct phases do exist in the intermediate state of the superconductor, irrespective of the presence or nature of the dopant. The first phase, identified with a small set of absorption peaks, is due to the formation of laminar structure created by the application of the magnetic field. The second phase, giving rise to large sets of absorption peaks, is due to the formation of microstructure within the superconducting laminar thickness. From the comprehensive study of the ultrasonic attenuation in the intermediate and superconducting state of In (99·9999 %), In doped with 0·003 at. % Pb, Pb doped with 0·003 at. % In and pure Pb (99·9999 %), it is possible to identify the collective excitation modes in both the states. The purpose of the present study is to correlate the role of imperfections in producing appropriate microstructures responsible for producing oscillatory attenuation in both the states.

2. Experimental

Ultrasonic attenuation measurements were taken on the single crystals of In (99·9999 %), In + 0·003 at. % Pb, Pb + 0·003 at. %, In and pure Pb (99·9999 %), using standard double ended pulse echo technique for temperatures 4·2 K to 1·4 K, in the frequency range, 9·9 to 29·7 MHz. Two X-cut quartz transducers were bonded to the single crystal using high viscosity vacuum grease. One quartz transducer was excited by 1–2 μsec radio frequency pulse to generate ultrasonic longitudinal wave. This wave, after passing through the sample, suffers partial reflection at the surface of the sample. Part of the energy is given to the receiver transducer while the remaining returns to the sample. This process goes on till the wave is completely absorbed. The receiver transducer, converts the ultrasonic energy to electrical energy. These electrical signals were amplified, detected and then displayed on the oscilloscope. The echo height was monitored with a calibrated pulse comparator. Attenuation was calculated using successive peak heights. The accuracy of measurement of attenuation was 0·02 db cm^{-1}. Single crystals of 1 cm diameter and 4 cm length were grown using Bridgman method with carbon-coated glass moulds. The ingots were cut to the length of about 1 cm using a machine described by Maddin and Asher (1950). The faces of the sample were made parallel to each other to within half a degree by a slow lapping process using 3200 mesh abrasive powder. Finally, the crystals were electropolished in $CH_3OH + HNO_3$ solution, (3:1). All the samples were then annealed for 80 hours. Samples were brought to the intermediate state by applying a transverse magnetic field of 0·7 H_c and 0·9 H_c, and readings were taken over the entire range of temperature i.e. 4·2 K to 1·4 K for 0·7 H_c, 0·9 H_c and 0 H_c. The values of the critical field H_c for different temperatures below T_c were calculated using the relation given by Lock *et al* (1951) for

In and In doped with Pb and from the graph of H_c vs T for Pb and Pb doped with In. These values are correct to within 0·5 % the experimental values. Vapour pressure on the helium bath was reduced by pumping over it and the corresponding temperature was obtained from standard tables. Magnetic field was measured at the site of the specimen by means of a calibrated Hall probe, which gave the field with 2 % accuracy.

3. Results and discussion

Ultrasonic attenuation measurements on single crystals of In, In doped with Pb, Pb doped with In and pure Pb were studied; 9·9–29·7 MHz in the intermediate state (with fields 0·7 H_c and 0·9 H_c) and superconducting states from 4·2 to 1·4 K. Temperature-dependent oscillatory behaviour of attenuation was observed for all the samples in both the states. The results of measurements are indicated in figures 1–4 for In, In doped with Pb, Pb doped with In and pure Pb respectively. This is explained by the Claiborne and Einspruch model, where in each set, successive resonance peak represents the consecutive harmonic of the fundamental mode of excitation. Temperatures of successive absorption peak values were used to calculate the values of a, b and v_0 for respective samples. The values of constants are shown in tables 1 and 2.

3.1 *Intermediate states*

In the intermediate state of all the samples, for a particular magnetic field, 2 sets of absorption peaks have been found—small and large. For the first phase, characterized by a small set of absorption peaks, v_0 values increase with increase in the magnetic field i.e. from 0·7 H_c to 0·9 H_c, H_c being the critical field. According to equation (1), this

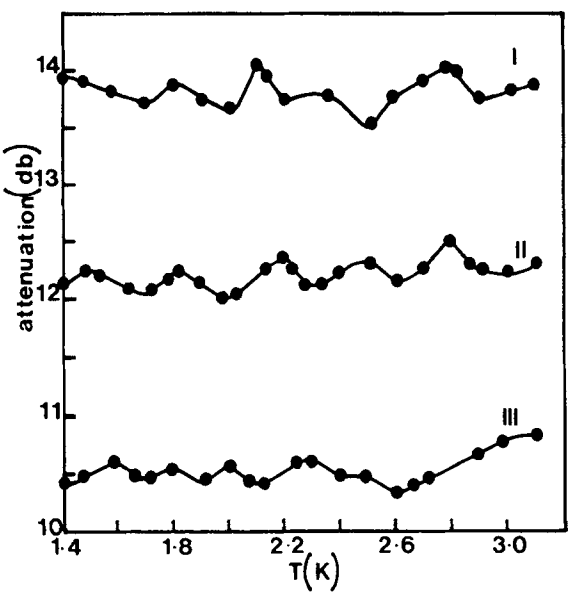

Figure 1. Plot of attenuation (db) as a function of temperature for pure In at 16·5 MHz. I. 0·7 H_c, II. 0·9 H_c, III. Superconducting state.

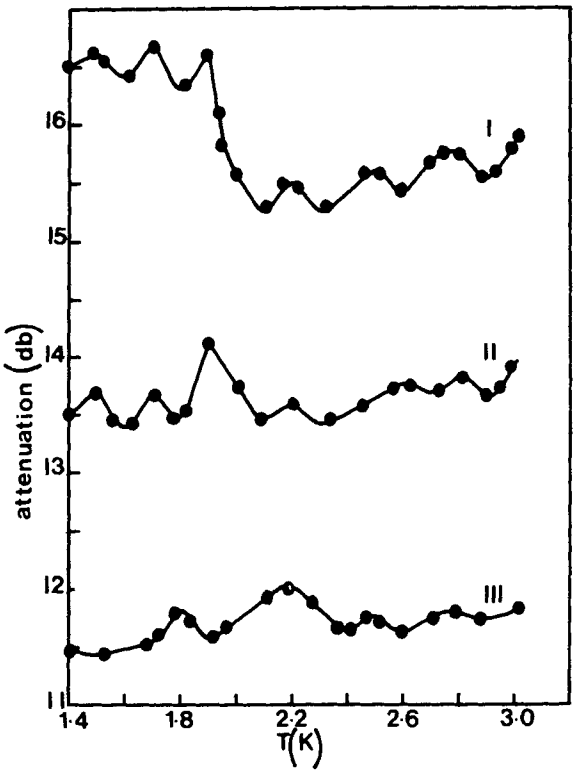

Figure 2. Plot of attenuation (db) as a function of temperature for In + 0·003 at.% Pb at 16·5 MHz. I. 0·7 H_c II. 0·9 H_c III. Superconducting state.

Table 1. Values of constants a, b and v_0 at 16·5 MHz.

	First phase			Second phase		
Fields	a K^{-2}	b K^{-4}	v_0 MHz	a K^{-2}	b K^{-4}	v_0 MHz
Pure In:						
Intermediate state						
(i) 0·7 H_c	0·0042	0·0977	0·633	0·0091	0·1597	4·98
(ii) 0·9 H_c	0·0029	0·0813	0·941	0·0091	0·1597	4·98
Superconducting state	... absent ...			0·0091	1·1597	4·98
In doped with 0·003 at.% Pb:						
Intermediate state						
(i) 0·7 H_c	0·0042	0·0977	0·633	0·0095	0·1875	7·5
(ii) 0·9 H_c	0·0029	0·0813	0·941	0·0095	0·1875	7·5
Superconducting state	... absent ...			0·0095	0·1875	7·5

indicates a decrease in the laminar thickness Z_s. This decrease in Z_s with increase in the magnetic field is in accordance with the relation given by equation (2). The origin of this phase is, therefore, due to the formation of laminar structure on account of the application of the magnetic field.

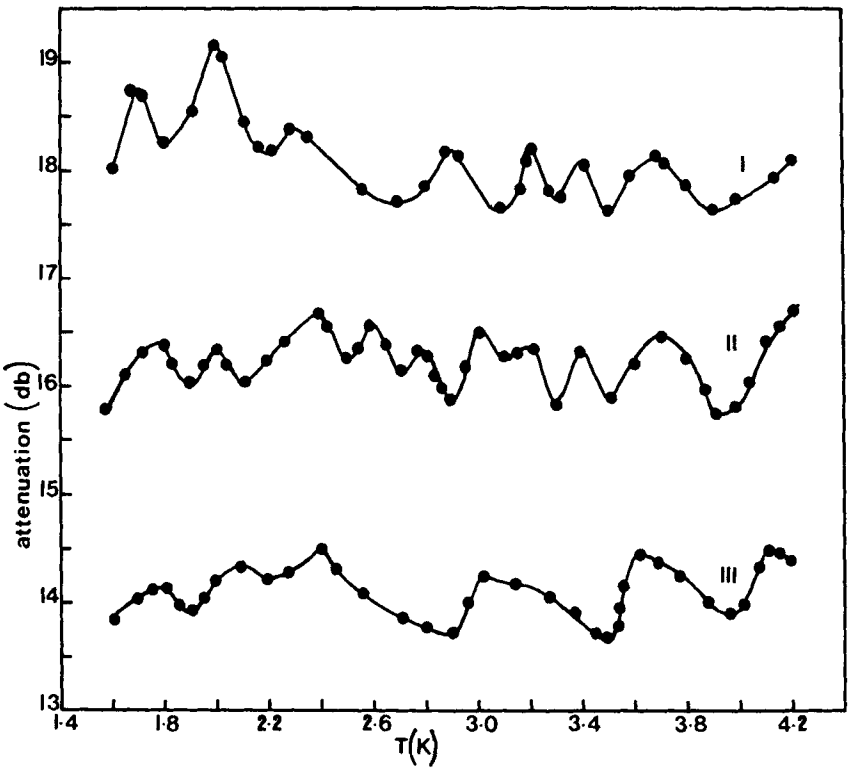

Figure 3. Plot of attenuation (db) as a function of temperature for Pb + 0·003 at.% In at 16·5 MHz. I. 0·7 H_c, II. 0·9 H_c, III. Superconducting state.

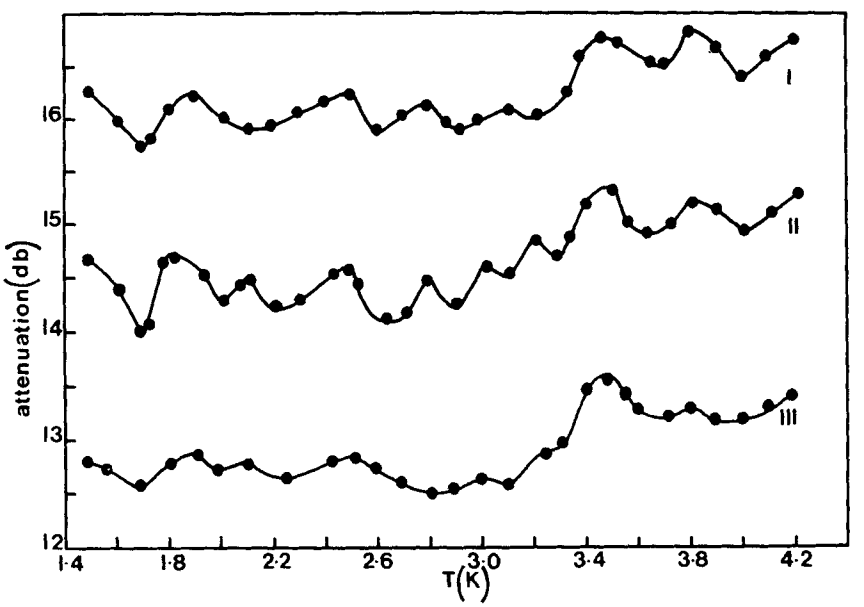

Figure 4. Plot of attenuation (db) as a function of temperature for pure Pb at 16·5 MHz. I. 0·7 H_c, II. 0·9 H_c, III. Superconducting state.

Table 2. Values of constants a, b and v_0 at 16·5 MHz.

Fields	First phase			Second phase		
	a K^{-2}	b K^{-4}	v_0 MHz	a K^{-2}	b K^{-4}	v_0 MHz
Pure Pb Intermediate state						
(i) 0·7 H_c	0·0044	0·0943	0·693	0·0067	0·1498	4·8
(ii) 0·9 H_c	0·0036	0·0863	1·087	0·0067	0·1498	4·8
Superconducting state	... absent ...			0·0067	0·1498	4·8
Pb doped with 0·003 at.% of In Intermediate state						
(i) 0·7 H_c	0·0051	0·0962	0·693	0·0041	0·1140	6·0
(ii) 0·9 H_c	0·0040	0·0870	1·086	0·0041	0·1140	6·0
Superconducting state	... absent ...			0·0041	0·1140	6·0

The v_0 values for the second phase in all the samples are independent of the magnetic field and hence the origin of the phase is entirely different. Moreover, the v_0 values of the second phase are higher than those for the first phase and hence indicate that the thickness of the second phase is much smaller than that for the first phase. The origin of this phase is, therefore, due to formation of microstructure within the superconducting laminar thickness. The formation of the microstructure is discussed in detail in § 3.4.

3.2 *Superconducting state*

Attenuation peaks show the presence of only one phase. Absence of the first phase is expected as the laminar structure is absent in the superconducting state. According to table 1 the values of a, b and v_0 for pure In agree exactly with the corresponding values for the second phase in the intermediate state, showing that the same microstructure is also present in the superconducting state. A similar result has been obtained by Chaudhuri and Singh (1982). This shows that the microstructure is related to the basic physical state of the sample.

3.3 *First phase*

From table 1, it can be seen that v_0 values for the first phase at 0·7 H_c for pure In and In doped with Pb are the same i.e. 0·633 MHz. Similarly v_0 values for In and In doped with Pb at 0·9 H_c for the first phase are the same i.e. 0·941 MHz. From table 2, it is seen that the same result is repeated for Pb and Pb doped In samples. Using equation (2), this means that superconducting laminar thickness at a particular magnetic field (either at 0·7 H_c or at 0·9 H_c) for pure and doped samples is the same. This shows that superconducting laminar thickness, and hence the origin of the first phase is independent of the nature or presence of the dopant and is entirely dependent upon the magnetic field.

From tables 1 and 2, it can be seen that v_0 values for the first phase in In and In doped with Pb at 0·7 H_c are different from those in Pb and Pb doped with In for the respective magnetic fields. This shows that the superconducting laminar thickness Z_s is different in both the cases. In and Pb, being two different materials, have different values of

coherence length ξ and penetration depth λ_D. As the surface energy parameter is $\Delta \approx \xi - \lambda_D$, Δ also is different in both the cases. This according to equation (2), means that the values of Z_s will be different. Now, using the relation $\nu_0 = C_0/2Z_s$, we obtain

$$\frac{\nu_{00{\cdot}7H_c}}{\nu_{00{\cdot}9H_c}} = \frac{Z_{s0{\cdot}9H_c}}{Z_{s0{\cdot}7H_c}}$$

From table 1, for pure In, we get

$$\frac{Z_{s0{\cdot}9H_c}}{Z_{s0{\cdot}7H_c}} = \frac{0{\cdot}633}{0{\cdot}941} = 0{\cdot}67. \tag{a}$$

From relation (2), we get:

$$\frac{Z_{s0{\cdot}7H_c}}{d_1} = \frac{1}{(2\times 0{\cdot}7)^{1/2}}(\Delta/d_1)^{1/2}, \quad \frac{Z_{s0{\cdot}9H_c}}{d_1} = \frac{1}{(2\times 0{\cdot}9)^{1/2}}(\Delta/d_1)^{1/2},$$

$$\frac{Z_{s0{\cdot}9H_c}}{Z_{s0{\cdot}7H_c}} = (0{\cdot}7/0{\cdot}9)^{1/2} = 0{\cdot}88 \tag{b}$$

This value is higher than the value given by the relation (a). Similarly, for pure lead, we get,

$$\frac{Z_{s0{\cdot}9H_c}}{Z_{s0{\cdot}7H_c}} = \frac{0{\cdot}693}{1{\cdot}086} = 0{\cdot}63.$$

We see that this is in good agreement with that obtained for pure In according to relation (a). Thus, the ratio of the laminar thickness at 0·9 H_c to that at 0·7 H_c, as obtained from the concept of collective excitation mode, is in good agreement for pure In and pure Pb but is lower than the ratio obtained from the consideration of the role of surface energy parameter and the breaking up of type I superconductors into normal and superconducting layers on the application of suitable magnetic field. This difference may be attributed to the approximations involved in computing Z_s according to Kuper's relation. We also observe from table 1 that for pure In and In doped with Pb samples ν_0 value is 0·633 MHz for 0·7 H_c and is 0·941 MHz for 0·9 H_c. Thus ν_0 value for particular magnetic field is the same for doped and undoped samples. Similar results are obtained for pure lead and lead doped with indium samples. This means that superconducting laminar thickness Z_s for a particular magnetic field is same for the doped and undoped samples. This according to equation (2), implies that the surface energy parameter is the same for doped and undoped samples. This also means that surface energy parameter is unaffected by doping. As $\Delta \approx \xi - \lambda_D$, ξ and λ_D being coherence length and penetration depth respectively, either the difference of the two or ξ and λ_D independently should remain unchanged. Now,

$$\xi = \frac{0{\cdot}6(l\xi_0)^{1/2}}{(1-l)^{1/2}} \quad \text{where} \quad \xi_0 = \frac{2\hbar V_F}{\pi E_g} \text{ and } l = \text{mean free path}$$

$$\lambda_D = \lambda_L(\xi_0/l)^{1/2} \quad \text{where} \quad \lambda_L = (mc^2/4\pi ne^2)^{1/2}.$$

From these equations, it is seen that the nature of dependence of ξ and λ_D on l is different and hence Δ cannot be maintained constant if l changes. Hence values of ξ and λ_D

involved here, cannot be related to the mean free path l. It appears, therefore, that one has to consider the intrinsic values of coherence length and penetration depth i.e. ξ_0 and λ_L.

3.4 *Second phase*

It can be seen from tables 1 and 2 that the v_0 value of 4·97 MHz for pure In is very nearly equal to 4·8 MHz for pure Pb. Since $v_0 = C_0(2Z_s)^{-1}$, v_0 is a measure of Z_s i.e. the extent of the second phase. It is evident from the results that the extent of microstructure in both the samples is the same. This indicates that the nature and type of pinning responsible for the formation of microstructure is the same.

The value of v_0 for In doped with Pb is 7·5 MHz whereas that for Pb doped with In is 6·0 MHz. These results show that v_0 values have been slightly affected on account of doping.

The ultrasonic attenuation measurements by Chaudhuri and Verma (1978) in single crystals of pure tin at 300 K for frequencies varying from 3–50 MHz exhibit resonance type of behaviour in the attenuation which arises due to dislocations only, excluding the contribution due to electrons which is negligible at room temperature. Granato and Lücke (1956) have drawn the analogy between the vibration under an alternating stress of a dislocation line segment pinned through Cottrell (1948) mechanism by point defects i.e. vacancies or interstitial atoms, substitutional or interstitial impurity atoms and the problems of forced vibration of a damped vibration string. The resonance frequency is given by

$$\omega_R = 2\pi v_R = \frac{1}{L_c}(2G/\rho(1-\sigma)^{1/2}, \tag{3}$$

where G is the shear modulus of the material, ρ the density of the material and σ the Poisson's ratio. The length L_c is determined by minor pins due to point defects. In our experimental crystal of high purity In (99·9999 %) and high purity Pb (99·9999 %), the point defects are mainly vacancies and act as minor pins. It is assumed that for zero applied stress, the dislocations are straight and pinned down by the vacancies already present in the crystal. The interaction between the vacancy and the dislocation causes the vacancy to segregate round dislocations in equilibrium distribution. Each dislocation gathers round itself an atmosphere of vacancies. The concentration C of vacancies according to Granato and Lücke (1966) is given by a/L_c where a is the atomic distance along the dislocation line and consequently it increases with dislocation density, since it has been found (Chaudhuri and Verma 1979; Verma and Chaudhuri 1981) that L_c decreases with increase in dislocation density. It is, therefore, clear that localized inhomogeneity which acts as a microstructure, can be created between two pinning points of the dislocation.

Ultrasonic attenuation measurements by Chaudhuri and Das (1976) at 1·3 K, in the superconducting state of tin and by Chaudhuri and Verma (1978) at helium temperatures, 77 K and 300 K, in tin, show that ultrasonic attenuation versus frequency has a resonance type of behaviour and the resonance frequency goes on decreasing as the dislocation density is reduced. According to equation (3), the dislocation resonance frequency is inversely proportional to the average loop length. This means that the average loop length increases with decrease in dislocation density. If the microstructure is formed between the pinning points, then its thickness is likely to increase with

decrease in dislocation density. A study of ultrasonic attenuation in the intermediate state of In doped with 0·002 at.% of Bi for various physical states by Chaudhuri and Singh (1982) shows that the extent of the second phase is very much dependent on the physical state of sample. Table 3 giving the results of their measurement, show that the direct correspondence between ν_R and ν_0 value of the second phase and the size of the microstructure in the second phase can be correlated with the average distance between the pinning points. The table also shows that ν_R and ν_0 change appreciably with change in the physical state of the sample.

If we observe the ν_0 value of the second phase for the doped and undoped highly annealed specimens, it will be noticed that the ν_0 value does not change appreciably with doping.

The value of ν_0 for annealed 99·9999% In sample studied by us is 4·8 MHz, and that for annealed In doped with 0·002 at. % Bi, as stated before, is 6·5 MHz, while that for In doped with 0·003 at. % Pb studied by us is 7·5 MHz. These results clearly shows that the ν_0 value for the second phase is governed more by the physical state of the specimen than by the concentration of the dopant. It should be noted here that one can compute n, the number of point defects per cc, and the concentration C of the point defects along the dislocation string, if the dislocation density Λ is known. Table 3 also incorporates the result of computation for In doped with 0·002 at. % Bi.

Table 4 gives the values of ν_0, ν_R, Z_s, L_c, Λ, n and C for pure In and pure Pb samples studied by us which were annealed for 80 hours. It is observed from table 3 that as ν_R value i.e. ν_0 value increases with increase in dislocation density Λ, the concentration of the point defects C along the dislocation line also goes on increasing and the number of point defects available for pinning also increases. Comparison of the number of point

Table 3. Values of ν_0, ν_R, Z_s, L_c, Λ, n and C for In doped with 0·002 at.% of Bi.

Annealing time Hours	Λ cm^{-2} ($\times 10^5$)	ν_0 MHz	ν_R MHz	Extent of micro-structure Z_s(cm)($\times 10^{-3}$)	Average loop length L_c cm($\times 10^{-3}$)	$n = \Lambda/L_c$ cm^{-3}	$C = a/L_c$ ($\times 10^{-8}$)
24	8	21·5	18	2·33	1·6	50×10^7	625
29	2·5	15·8	12	3·17	2·39	$10·5 \times 10^7$	417
80	0·01	6·5	5	7·69	5·73	$17·5 \times 10^4$	175

Table 4. Values of ν_0, ν_R, Z_s, L_c, Λ, n and C for pure In and pure Pb.

Samples	ν_0 (MHz)	ν_R (MHz)	Z_s (cm) ($\times 10^{-3}$)	L_c (cm) ($\times 10^{-3}$)	Λ (cm^{-2}) ($\times 10^5$)	n (cm^{-3}) ($\times 10^4$)	$C = a/L_c$ ($\times 10^{-8}$)
Pure In 99·9999% annealed for 80 hr	5	3·85	10	7·44	0·006	8	134
Pure Pb 99·9999% annealed for 80 hr	4·8	3·7	10·4	7·74	0·006	7·75	129

defects in the highly annealed doped In sample with that of vacancies in the highly annealed undoped In sample shows that the number of point defects in the doped sample is of the same order of magnitude as that of vacancies for the undoped one. Thus, the point defects are mainly vacancies. Hence vacancies, which characterize the physical state of the specimen, are the main source of pinning the dislocation string and hence are primarily responsible for the formation of microstructure.

The marginal increase in v_0 value of the doped samples can be attributed to the addition of impurity atoms which offer more pinning points.

4. Conclusions

Ultrasonic attenuation measurements in the intermediate and superconducting states show the importance of the results in identifying the mechanism of absorption which is different from the one postulated by the BCS theory. Further, the microstructures or laminar structures which can be produced either by inhomogeneity in the system due mainly to the localized strain sites or by suitable application of a magnetic field which give rise to a temperature-dependent oscillatory attenuation. These results have been utilized to identify the origin of the structures and throw considerable light on the role of vacancies for the creation of microstructures.

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