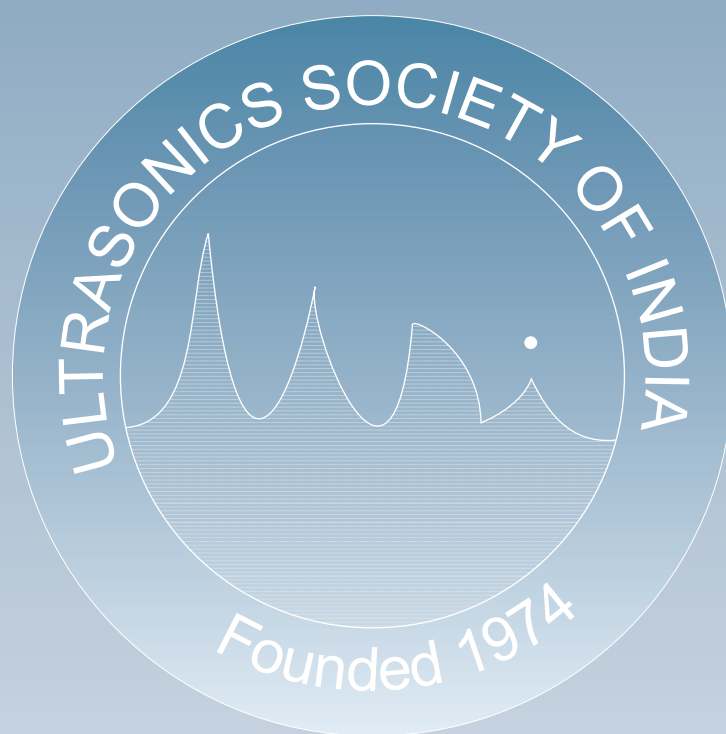
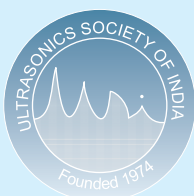


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# Journal of Pure and Applied *Ultrasonics*

No. 4	Volume 38	October-December 2016
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# Journal of Pure and Applied Ultrasonics

VOLUME 38

NUMBER 4

OCTOBER–DECEMBER 2016

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## Characterization of sodium compatible ultrasonic transducers using ultrasonic C-scan imaging

A. Ravi Gopal<sup>1</sup>, M.R. Jeyan<sup>2</sup>, C. Asokane<sup>2</sup>, Anish Kumar<sup>1\*</sup> and B. Purnachandra Rao<sup>1</sup>

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Ultrasonic technique is useful for viewing the internals of sodium cooled fast reactors. Ultrasonic transducer is the key element to carry out ultrasonic viewing and inspection. Sodium compatible high temperature ultrasonic transducers are being developed for this application. Lead zirconate titanate (PZT) crystal is used as active element. Nickel (Ni) face plate is used to facilitate sodium wetting at low temperatures. Bonding of the PZT crystal to the nickel faceplate is carried out by soldering/diffusion bonding techniques. Bond quality is important as it influences the performance of the transducer. Ascertaining the bond quality would help in fabricating high quality transducers. We have used under water ultrasonic C-scan imaging technique to assess the bond quality. In order to develop the technique, studies have been performed on a few transducers using reference defects introduced at the interface. Bond quality of the diffusion bonded high temperature transducers has been checked before and after exposure to high temperatures and the results are discussed in this paper.

**Keywords:** Ultrasonic transducer, C-scan imaging, beam profile.

### Introduction

Ultrasonic science and technology is the only modality for imaging the internal components of liquid metal cooled fast breeder reactor (LMFBR)<sup>1-3</sup>. In a typical LMFBR environment, ultrasonic transducers are introduced at the time of shut down when the reactor temperature is around 2000°C. These transducers facilitate estimation of position of fuel subassemblies, thus planning fuel handling operations. Commercial ultrasonic transducers cannot be used at these temperatures. In prototype fast breeder reactor, liquid sodium is used as the coolant and acts as the couplant for ultrasonic inspection. The bonding between the active lead zirconate titanate (PZT) and nickel (Ni) face plate is carried out by soldering/ diffusion bonding technique. Ultrasonic C- scan technique is a potential technique to evaluate the bond quality in sodium compatible ultrasonic transducers by time gating the appropriate echoes<sup>5-7</sup>. This paper gives details about the C-scan based methodology of asserting the bond quality of soldered/ diffusion bonded joint present between the active PZT

and Ni face plate. The methodology has been demonstrated with reference defect.

### Materials and Methods

The experimental set up for ultrasonic immersion testing consists of a computer (containing analog-to-digital converter card and operating system software), ultrasonic pulser-receiver, mechanical scanner, motion control subsystem, ultrasonic transducer and an immersion tank. Figure 1 shows the configuration of the transducers used in the experimental setup and a typical signal obtained during immersion ultrasonic inspection of Ni face plate bonded with active PZT element.

Ultrasonic C-scan imaging was performed using a 25 MHz immersion transducer with focal length 35.0 mm with a scan step size of 0.5 mm. The ultrasonic beam was focused at the interface between Ni face plate and the active PZT element, keeping the water path between the probe and the top surface of the casing as 24.0 mm. Ultrasonic signals were acquired at 250 MS/s (4 ns time

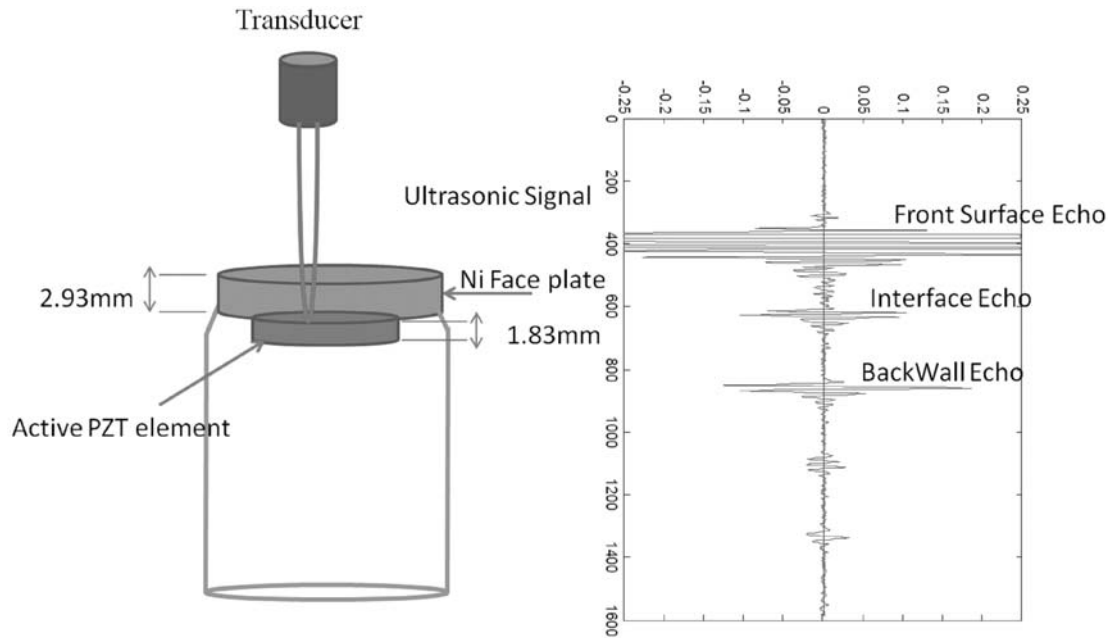


Fig. 1 Schematic of the configuration of transducers used in immersion experimental setup and expected echoes.

resolution) sampling rate. The pulse repetition frequency (PRF), energy, filter and damping settings were 1 kHz, 2μJ, 1 MHz to 35 MHz and 50Ω, respectively.

Figure 2 shows the drawing showing the dimensions of reference transducers without and with a circular

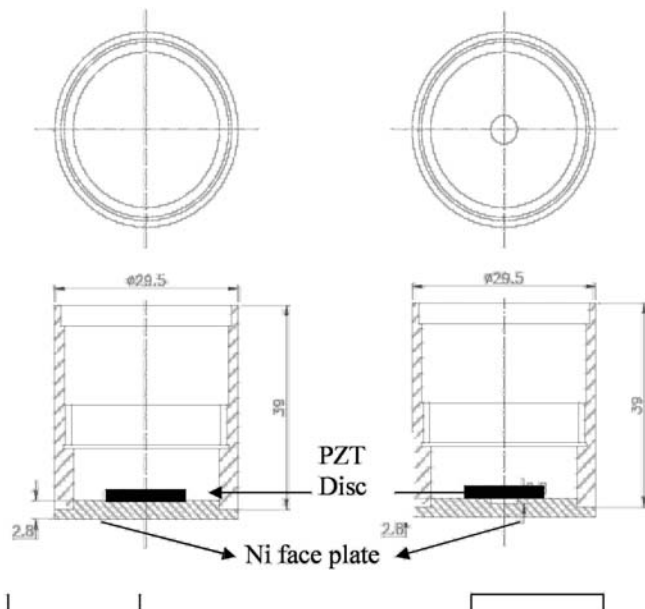


Fig. 2 Drawing showing inner side of the face plate of the reference transducers and cross section of reference transducers with and without the circular defect at the center of the inner side of the face plate of ultrasonic transducer.

reference defect at the center of the inner side of the face plate of ultrasonic transducer. In case of sodium compatible ultrasonic transducer, the Ni face plate and the active PZT was diffusion bonded and in case of reference transducer the Ni face plate and active PZT was bonded using a standard couplant. The beam profile of the reference transducers were obtained by using a pinducer as receiver and the transducers as transmitter in through-transmission mode for the same pulser settings.

## Results and Discussion

Figures 3(a) and (b) show typical ultrasonic A-scan signals acquired for Ni face plate and active PZT, respectively. Using a screw gauge, thickness of Ni face plate and active PZT material were measured as 2.93 mm and 1.83 mm, respectively. The corresponding ultrasonic velocity in Ni face plate and active PZT material were calculated using the A-scan signals as 5550 m/sec and 4377 m/s respectively. Figures 4(a) and 4(c) show the C-scan image of sodium compatible ultrasonic transducer obtained by time gating the interface echo, prior and after high temperature exposure respectively. Figure 4(b) shows the C-scan image of the reference transducer with 3 mm central defect at the inner side of the face plate obtained by time gating the echo corresponding to the 3 mm central defect rather than

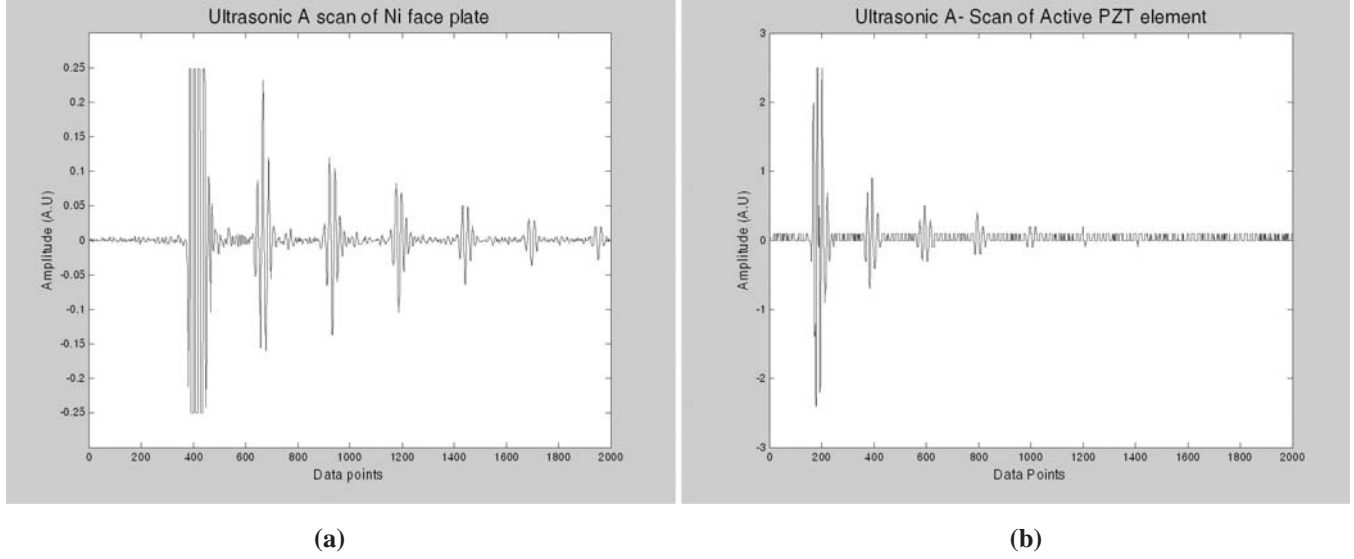


Fig. 3 Ultrasonic A-scan signal obtained in (a) 2.93 mm thick Ni Faceplate and (b) 1.83 mm thick PZT disc.

the interface echo. Three horizontal cursors X-X', Y-Y' and Z-Z' are also shown in the Figs. 4(a), 4(b) and 4(c), respectively. These cursors mark the diameter of the transducers.

A C-scan image is generated using several B-scans. Figures 4(d), 4(e) and 4(f) correspond to the B-scan images obtained while scanning across the X-X', Y-Y' and Z-Z', respectively. Three vertical cursors U-U', V-V' and W-W' are shown in Figs. 4(b), 4(e) and 4(f), respectively. A typical B-scan image is composed of several A-scan signals. Figures 4(g), 4(h) and 4(i) correspond to the A-scan signals obtained while placing the transducer at the points U-U', V-V' and W-W', respectively.

From the A-scan signals it can be observed that the interface echo always appears approximately at about 250 data points from the front surface echo. These 250 data points correspond to the 2.93 mm thick Ni face plate. Figure 4(h) corresponds to the A-scan signal obtained at the center of the reference transducer. Here, an additional echo appears prior to the interface echo. Since a 0.8 mm deep defect is made at the center of the inner face of the face plate of the reference transducer, the effective distance seen by the impinging ultrasonic pulse is 0.8 mm less as compared to the sodium compatible ultrasonic transducer. This results in an echo prior to the interface echo whose amplitude is greater than the interface echo amplitude.

The amplitude of the interface echo is dependent upon

the acoustic impedance  $Z$  given by

$$Z = \rho v \quad (1)$$

where  $\rho$  is density and  $v$  is velocity of ultrasound in media. At an interface when two media are encountered, the reflection coefficient is given as :

$$R = \frac{(Z_2 - Z_1)}{(Z_2 + Z_1)} \quad (2)$$

where  $Z_1$  and  $Z_2$  are the acoustic impedance of the first and second media respectively.

Ultrasonic inspection is carried out from the Ni face plate side and the first reflection is obtained from the water-substrate interface as 'front surface echo'. Depending upon the acoustic impedance mismatch, and if the quality of the bond is good, a part of the ultrasonic energy penetrates in the active PZT element and the rest is reflected at the interface as an 'interface echo'. Greater the difference in acoustic impedance, greater will be the amplitude percentage of reflection. The ultrasonic energy that propagates into the active PZT element is reflected from the back surface of the element as a 'back-wall echo'.

Figures 4(g) and 4(i) correspond to the A-scan signals obtained on the sodium compatible ultrasonic transducer prior and after the exposure to high temperature respectively. Prior to exposure to high temperature, when the bonding is good, greater amount of ultrasonic energy is transmitted and lesser amount is reflected at the interface. As a result, the amplitude of the interface echo is less as compared to the back-wall echo which can be

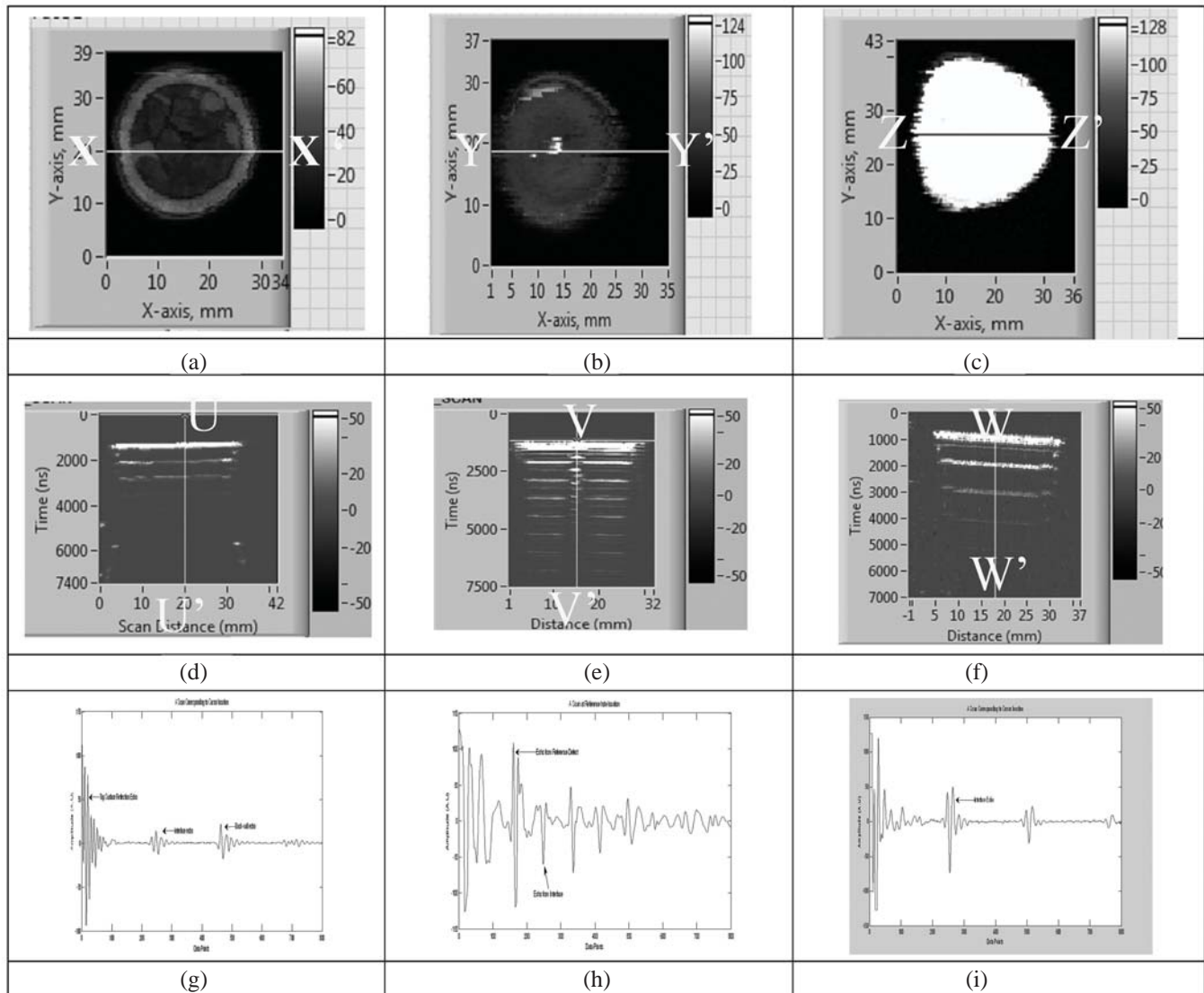


Fig. 4 Ultrasonic C-scan image of (a) sodium compatible ultrasonic transducer prior to high temperature exposure (b) reference transducer and (c) sodium compatible ultrasonic transducer after high temperature exposure, obtained by time gating the echoes of interest. (d), (e) and (f) show B-scan images corresponding to  $XX'$ ,  $YY'$  and  $ZZ'$  as shown in (a), (b) and (c), respectively. (g), (h) and (i) show A-scan signals corresponding to  $UU'$ ,  $VV'$  and  $WW'$  as shown in (d), (e) and (f), respectively.

seen in Fig. 4(g). The exposure to high temperature results in the failure of bond between the Ni face plate and active PZT. Due to the failed bond, greater amount of ultrasonic energy is reflected and lesser amount is transmitted to the back-wall. From the Fig. 4(i) the amplitude difference between the interface echo and the back-wall echo can be noticed.

Figure 5(a) shows the beam profile obtained through C-scan imaging for the reference transducer without the central defect. Figure 5(b) shows the beam profile

obtained through C-scan imaging of the reference transducer with the central defect at the inner side of the face plate. Both the transducers were excited with same input pulser settings. C-scan data was acquired in through-transmission mode with reference transducer as emitter and pinducer as receiver. It can be noticed from the two figures that the maximum limit of gray scale in Fig. 5(b) is lower than that in Fig. 5(a) indicating lesser amount of radiated field. The marked rectangle region in Fig. 5 (b) shows lesser amplitude of the radiated field.

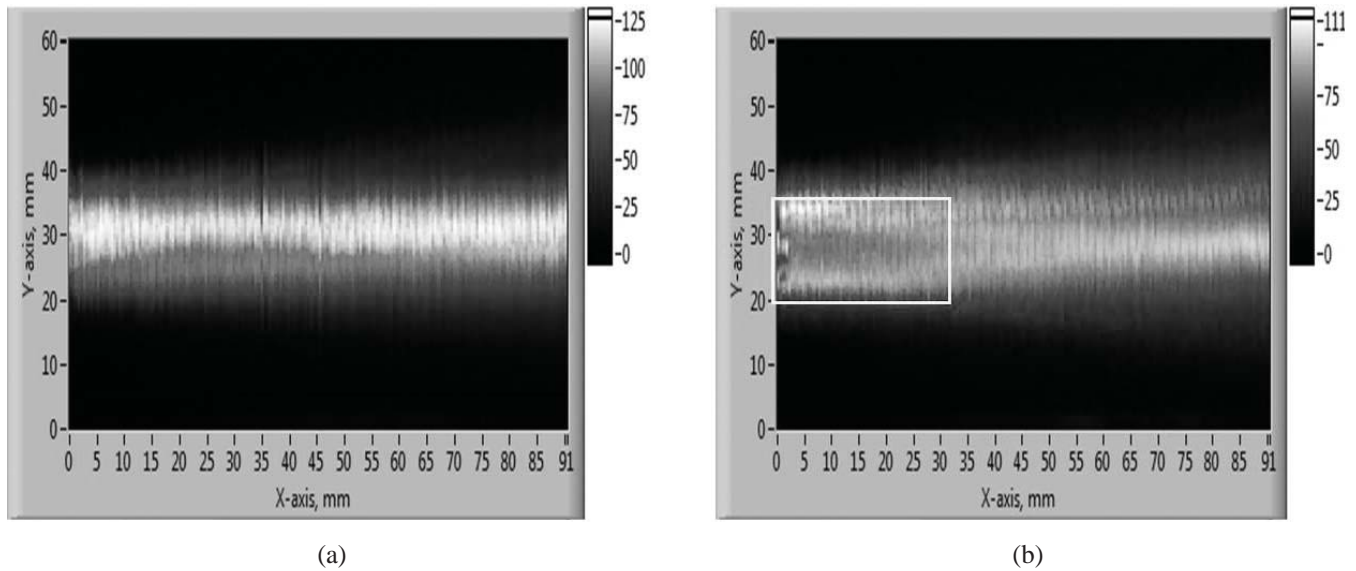


Fig. 5 C-scan image generated by scanning with a pinducer in the plane perpendicular to front face of the reference transducer (a) without central defect and (b) with central defect.

## Conclusion

In the present study, a methodology for evaluation of bond quality was developed and was implemented on a few piezoelectric ultrasonic transducers using reference defects introduced at the interface. The study indicates that ultrasonic C-scan imaging can be used for assessing interface quality in the sodium compatible ultrasonic transducers. Further, C-scan imaging using a pinducer can be used to study the beam profile and the quality of the radiated field of the ultrasonic transducer.

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## Application of ultrasonicated blended solvent for dewaxing of crude petroleum oil

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Wax deposition is a serious problem in the petroleum industry that results in plugging of flow strings, formation damage, loss of hydrocarbons, increased production cost. In order to overcome wax deposition problem, this study investigated the feasibility of acetone: n-pentane as a selective solvent to dewax a Nigerian heavy crude oil using ultrasonic technique. To understand the effectiveness of selected solvent mixture for dewaxing process, the ultrasonic velocity, density, viscosity are determined experimentally at temperature 30°C. From the experimental data the acoustic parameters such as adiabatic compressibility ( $\beta$ ), intermolecular free length ( $L_p$ ), acoustic impedance ( $Z$ ), interaction parameter ( $\eta$ ) and molar volume ( $V_m$ ) and their excess values are calculated. The variation of acoustic parameters and their excess values are discussed in terms of molecular interactions present in the solvent mixture which explains the physio-chemical behaviour and optimum feasibility condition of the blended solvent for dewaxing of the crude oil. Experiments were conducted on three solvent to crude oil ratios (10:1, 15:1 and 20:1) at 30°C. Each crude oil sample was weighed out and mixed with acetone: n-pentane at a predetermined mass ratio and then heated in a hot water bath and stirred until a thermal equilibrium was achieved. The results indicated that acetone: n-pentane dewaxing performance improved at 30°C temperatures.

**Keywords:** Crude oil, dewaxing, blending solution, ultrasonic velocity, acoustic parameter.

### Introduction

The presence of paraffin waxes in crude oils presents a multitude of problems to the producers. Petroleum production can be significantly affected by deposition of paraffin wax during crude production, with devastating economic consequences. Hence, predicting wax problems within the production tubing and flow lines that could decrease or halt production is essential in optimizing production and operating efficiency. Paraffin generally consist of straight and branched chain hydrocarbons and precipitates out of waxy crude when there is a slight change in equilibrium conditions, causing a loss of solubility of the wax in the crude. Deposition of wax in a region changes the thermo-dynamical equilibrium for which the temperature falls below the cloud point. As a result of which the oil becomes thicker and clogs fuel filters and injectors in engines. Thus a suitable solvent mixture is used which increases the cloud

point temperature. As a result of which, the interaction of solvent mixture with wax decreases the viscosity converting the non-Newtonian flow into Newtonian. Though there are many methods<sup>1-7</sup> available in the literature for study of suitability of solvent mixture for dewaxing but accurate no such method is highly valuable for this process. Thus this work is an attempt to study the compatibility of a suitable blend for the dewaxing of wax present in crude oil using ultrasonic technique. Being ultrasonic wave a sub category of acoustics it has high frequency - low intensity and low frequency - high intensity which makes it possible to obtain the information about the medium or to convey information through the medium through molecular interactions between the molecules of the medium. The wavelength of this wave changes from one medium to another medium due to which it can be reflected off with very small surfaces and provides information from atomic/subatomic level.

## Materials and Methods

High purity and analytical grade samples of n-pentane 99% (GC) and Acetone 99.0% (GC) are taken for experiment. The entire chemical used in the study are purified by standard procedure and redistilled before use. To minimize the contact of this deliquescent reagent with moist air, the product was kept in sealed bottles in desiccators. The purities of the sample were confirmed by GLC. The crude oil samples used in this study were from Niger Delta Oil field in Nigeria, having density in the range of 847-869 kg/m<sup>3</sup>.

Binary mixtures were prepared by mass in air tight bottles. The mass measurements were performed on high precision digital balance with an accuracy of + 0.001 mg. The uncertainty in mole fraction was + 0.001. The densities of pure liquids and their mixture were determined by using double arm pycnometer with accuracy of the order of + 0.01 kg/m<sup>3</sup>. The ultrasonic speed of the above liquids and their mixtures were measured using multi-frequency ultrasonic interferometer operating at different frequencies. The accuracy in the measurement of ultrasonic velocity was within + 0.01 ms<sup>-1</sup>. The temperature of the solution was controlled by circulating water at a desired temperature through the jacket of double walled cell within + 0.01 K using a constant temperature bath with an accuracy of + 0.001 K.

Experiments were conducted on three solvent to crude oil ratios (10:1, 15:1 and 20:1) for temperature 30°C for 45 min. Each crude oil sample was weighed out and mixed with acetone: n-pentane at a predetermined mass ratio and then heated in a hot water bath and stirred until a thermal equilibrium was achieved. The obtained extracts were kept in a sealed vial at 4°C. All extractions procedure was performed in triplicate with the standard deviation of obtained 0.7%. The ultrasonic bath Sonorex RK 52 operating at a frequency of 35 kHz was used with HF power of 60 W and ultrasonic peak output 240 W. In the second step, the solution of oil insolvent was then chilled to -17°C in a chiller for 30 minutes. The separated wax from the mother solution was filtered under vacuum suction through glass wool in a Gooch crucible, The flask containing the mother solution was rinsed with 20 ml of fresh solvent at -17°C to ensure that all components have been removed. The wax was then dried from any traces of oil by leaving it for 24 hours in a desiccators containing P<sub>2</sub>O<sub>5</sub> before being weighed. The yield point of the wax was calculated from the recovery of wax after the

treatment of solvent mixture by the following relation.

$$Y = \frac{W_e}{W_o} \times 100$$

where  $Y$  is the yield as a percentage of the total weight of the sample;  $W_e$  = the weight of the extract and  $W_o$  = the weight of the crude oil sample.

## Results and Discussion

For measurement of physio chemical properties the ultrasonic velocity was determined in pure acetone, n-pentane and their blended solution. The results shows that the ultrasonic velocity in pure n-pentane is high and then decreases with increase of acetone up to 0.70 mole fraction of solvent mixture. It may be due to the fact that strong hydrogen bonding between the long carbon chain of n-pentane and carbonyl group of acetone. As a result of which the motion of the molecules in the solvent mixture decreases which decreases the ultrasonic velocity. Since in acetone rich region the dispersive force is high between the similar molecules the ultrasonic velocity increases<sup>8</sup>.

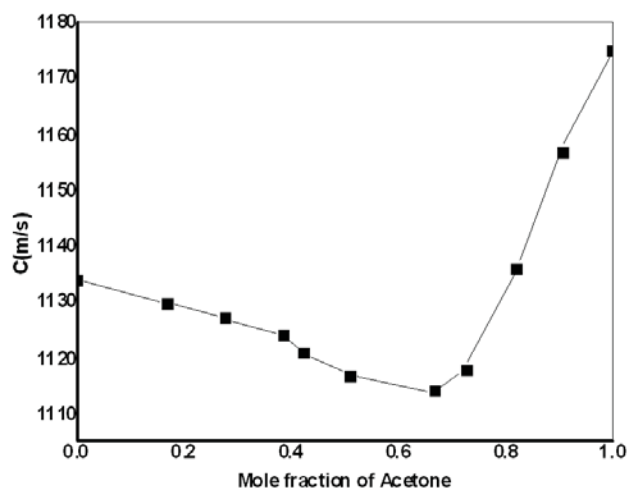


Fig. 1 Variation of ultrasonic velocity

The variation of excess adiabatic compressibility's and free length in Figs. 2-3 are found to be negative<sup>9</sup> for all mole fractions of acetone indicating the strong interactions between acetone and n-pentane. This is due to the fact that the formation of hydrogen bond in mixture is more than pure liquids of acetone and n-pentane. It shows that liquid mixture is highly compressed and it is possible only when unlike molecules are more tightly bound or interaction is high. Negative value of excess

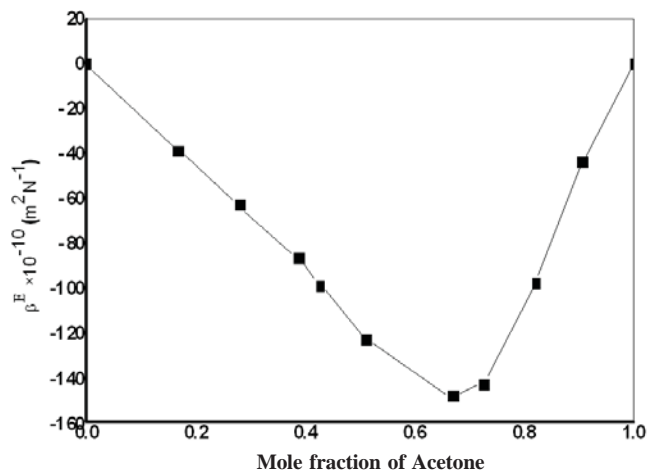


Fig. 2 Variation of excess isentropic compressibility

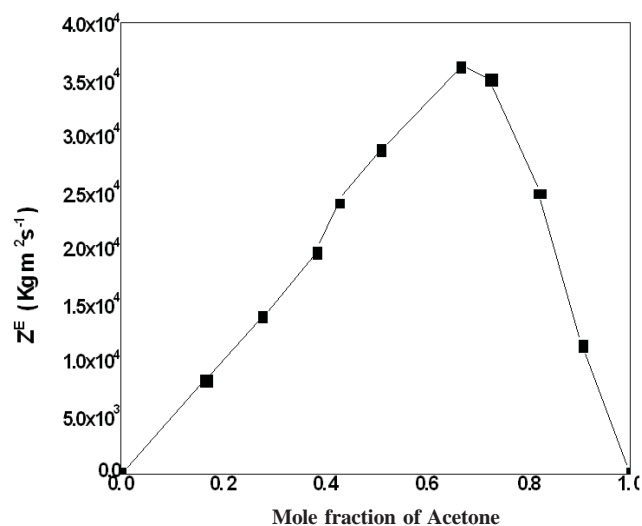


Fig. 4 Variation of excess acoustic impedance

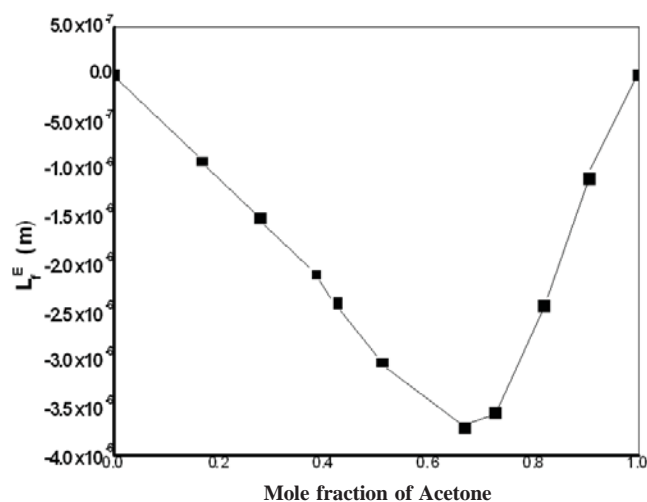


Fig. 3 Variation of excess inter molecular free length

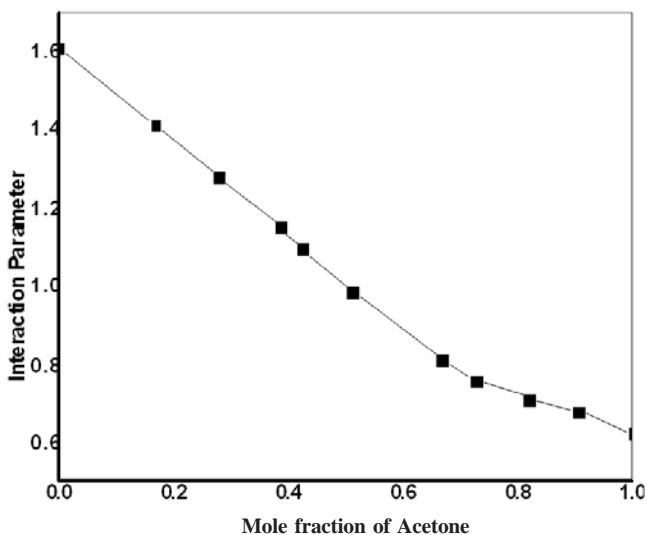


Fig. 5 Variation of interaction parameter

$L_f^E$  reflects strong interaction and it is due to the association of molecules.

Acoustic impedance is the opposition offered by the medium for the propagation of sound energy. In Fig. 4 positive deviation of excess acoustic impedance is maximum at 0.75 mole fraction of acetone, signifies strong interaction between polar and non-polar molecular. However, the magnitude of molecular association decreases as component of acetone (polar molecules) increases<sup>10</sup>. The interaction parameter ( $\chi$ ) decreases with the increase in acetone indicating association between solute and solvent is better than n-pentane rich region as shown in Fig. 5. From Fig. 6 it was observed that as the solvent added to the crude oil the extraction of wax produced increased<sup>11</sup> under the optimum conditions of acetone/n-pentane ratio 3:1 and

with change in the ratio of acetone/n-pentane to 10:1, 15:1 and 20:1 the amount of removal of wax increases with increasing the ratio of mixture.

It was observed that an increase in initial weight of crude oil from 5 g to 15 g, increases the amount of wax removed from 2.5 g to 17.8 g. This is ascribed to the fact that as the amount of crude oil increased, the amount of paraffinic wax removed was increased. Thus the produced wax after separation and filtration increased. Again, with the help of a light microscope (Metzer 5000 BM having resolving power 40X-1000X, with camera 3 Mega pixel CMOS SENSOR) analysis was done to observe the wax content before treatment and after treatment of solvent mixture.

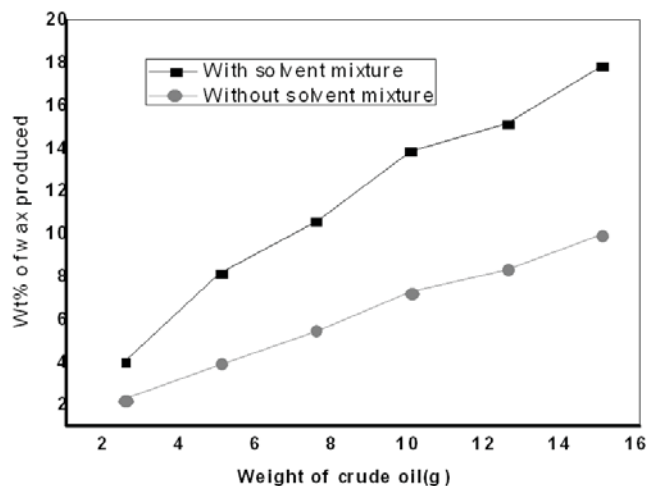


Fig. 6 Variation of wax produced with and without treatment of solvent mixture

Figures 7 and 8 shows crystals of wax that appear in Crude oil before and after the effects of solvent mixture at best conditions (30°C, 2 hr, ratio 3:1). As indicated above, the most important factor affecting the oil content of wax is solvent ratio which has the highest percentage of contribution. With an increase in the volume of the solvent, the viscosity of the blend decreases; this induces a crystal growth which consequently leads to better filtration, and thereby a decrease in the oil content. The volume of the solvent should not be more than an optimized volume because the cost of the solvent recovery could be too high, thereby increasing the total operational cost. The second important factor influencing the oil content is solvent composition. If the amount of

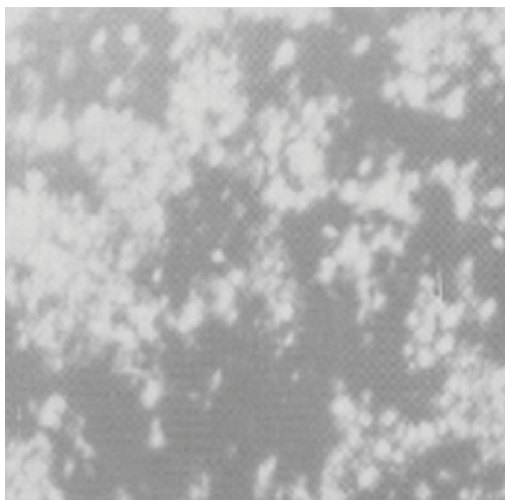


Fig. 7 Wax appearance in crude oil before treatment with solvent mixture

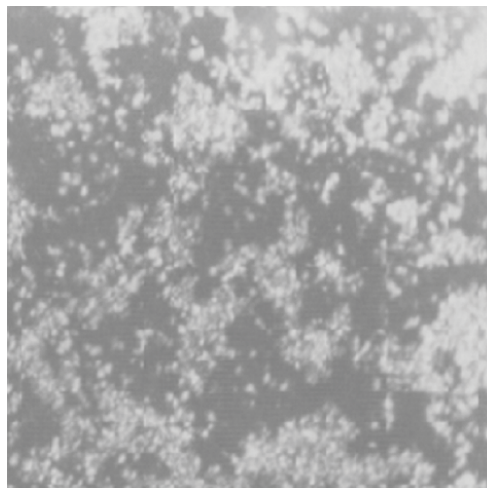


Fig. 8 Wax appearance crude after treatment with solvent mixture

acetone or n-pentane increases the proportion of anti-solvent improves the filtration rate, it leads to a drop in yield and the oil content of the wax produced.

## Conclusion

The suitability of solvent mixture taken for study of dewaxing of crude oil was established through the ultrasonic wave propagation. Variation of acoustic parameters indicates the presence of strong interactions in the solvent mixture. The treatment of each concentration of solvent mixture coagulates more wax for a particular solvent to oil ratio. From the experimental work it was concluded that the amount of wax produced increased, if the initial weight of the crude oil is increased and the solvent ratio increased but not beyond the optimized value. Therefore, we conclude that the amount of solvent composition should be determined in an economic manner that oil content and filtration rates are optimized. However, the proportion of anti-solvent can only be raised up to a certain point; otherwise phase separation occurs, which makes the filtration part impossible. The characteristics and behavior of solvent modified crude oils should help in addressing cost-effective solution to flow assurance problems in the industry.

## Acknowledgements

The authors are thankful to GAIL, Vijaipur, Guna, MP for giving the crude oil with chemical analysis and Vice Chancellor VSSUT, Sambalpur for giving permission to conduct the experimental work in the laboratory.

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## Elastic and ultrasonic properties of LaPn (Pn=N, P, As, Sb, Bi)

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Elastic and ultrasonic properties of lanthanum pnictides (LaPn) have been investigated along  $\langle 100 \rangle$ ,  $\langle 110 \rangle$ ,  $\langle 111 \rangle$  directions in the temperature range 100K to 300K. The second and third order elastic constants (SOECs and TOECs) have been computed using Coulomb and Born-Mayer potential using nearest neighbour distance and hardness parameter. Other parameters like Young's modulus, bulk modulus, shear modulus, Zener anisotropic factor, Poisson's ratio, toughness to fracture (G/B) ratio have also been found out for the future performance of LaPn. Additionally ultrasonic velocity has been evaluated with use of SOECs and TOECs. Obtained results are discussed in correlation with available mechanical and thermophysical properties of these materials.

**Keywords:** Lanthanum Monopnictides, elastic properties, ultrasonic properties.

### Introduction

Ultrasonics is a versatile tool in the fields of science and technology<sup>1,2</sup>. It is used to characterise the materials during processing and also after production by measuring ultrasonic properties. The rare earth monopnictides are the typical low carrier strongly correlated system. They have various anomalous physical properties because of partially filled f-orbitals. The partially filled f-electron of lanthanum ions are highly delocalized and interact strongly with lattice. The LaPn (Pn=N, P, As, Sb, Bi) generally crystallized in NaCl- structure. Many investigators studied on the physical properties of lanthanum monopnictides LaPn<sup>3-8</sup>.

Pagare *et al.*<sup>3</sup> found out the structural phase transition from their initial NaCl (B1) phase to body centered tetragonal (BCT) phase at high pressure of LaPn theoretically using interionic potential method. Deligoz *et al.*<sup>4</sup> investigated the structural, elastic, electronic, thermodynamical and vibrational properties of LaAs and LaP in the rock-salt (B1) structure by performing ab-initio calculations within the local-density approximation (LDA). Vaitheeswaran *et al.*<sup>5-6</sup> studied the electronic structure and structural properties of LaSb and LaBi by means of the self-consistent tight binding linear muffin

tin orbital method. The relative stabilities of LaSb and LaBi at high pressures in the rock salt primitive tetragonal and CsCl-structures are analysed. They also used the self-consistent tight binding linear muffin-tin orbital method to calculate the electronic structure and the relative stabilities of LaP and LaAs at high pressures in the rocksalt, primitive tetragonal and CsCl structures. Varshney *et al.*<sup>7</sup> discussed the pressure dependent first order phase transition, mechanical, elastic, and thermodynamical properties of NaCl-type (B1) to CsCl-type (B2) structure in LaPn. Gökoglu and Erkisi<sup>8</sup> deliberated the elastic and structural properties and lattice dynamics of LaN and LaBi systems in B1 phase using plane-wave pseudopotential method of the density functional theory (DFT) with generalized gradient approximation (GGA). In the present paper, temperature dependent elastic and ultrasonic properties of LaPn have been computed. We have projected the elastic properties like second and third order elastic constant (SOECs and TOECs) and mechanical properties such as Young's modulus (Y), bulk modulus (B), shear modulus (G), Poisson's ratio ( $\sigma$ ), G/B ratio. Then, ultrasonic velocity and Debye average velocity have computed along  $\langle 100 \rangle$ ,  $\langle 110 \rangle$ ,  $\langle 111 \rangle$  directions for longitudinal and shear mode of propagation.

## Theory

The SOECs and TOECs have been calculated following Brugger's definition of elastic constants at absolute zero<sup>9</sup>. The SOECs and TOECs at higher temperatures are obtained by the methods developed by Ghate<sup>10</sup> and Mori and Hiki<sup>11</sup> for NaCl type crystals. The SOEC and TOEC at a particular temperature can be obtained by adding vibrational energy contribution to the static elastic constants. The detailed expressions to find out SOECs and TOECs are given in our previous paper<sup>2</sup>.

Other mechanical constants like bulk modulus (B), shear or rigidity modulus (G), Young's modulus (Y), Poisson's ratio ( $\sigma$ ), Zener's anisotropy (A), tetragonal shear modulus, toughness to fracture ratio (G/B) are also calculated by using second order elastic constants in this paper. These parameters are responsible for determining strength, stability and hardness of the material<sup>12</sup>.

The expressions for the mechanical constants are given below:

$$B = \frac{C_{11} + 2C_{12}}{3}; Y = \frac{9GB}{G + 3B}$$

$$G = \frac{C_{11} - C_{12} + 3C_{44}}{3} + \frac{2.5(C_{11} - C_{12})C_{44}}{4C_{44} + 3(C_{11} - C_{12})}$$

$$= \frac{3B - 2G}{6B + 2G}; Y = \frac{9GB}{G + 3B}; C_s = \frac{C_{11} - C_{12}}{2}$$

When sound wave propagates in a solid medium, there are three modes of propagation one longitudinal acoustical and two transverse acoustical. Hence there exists three types of velocities, as one longitudinal ( $V_L$ ) and two shear ( $V_{s1}$ ,  $V_{s2}$ ) that depends on the direction of propagation of the wave. From ultrasonic velocity, the Debye average velocity along different crystallographic directions is calculated. From the values of Debye average velocity Debye temperatures were calculated which gives important information regarding crystal structure. The expression for ultrasonic velocity along the  $\langle 100 \rangle$ ,  $\langle 110 \rangle$ ,  $\langle 111 \rangle$  are given in literature<sup>2,12</sup>.

## Results and Discussion

The temperature dependent SOECs and TOECs have been calculated by using two basic parameters, lattice parameter and hardness parameter. The lattice parameter<sup>8</sup> for LaN, LaP, LaAs, LaSb, LaBi in NaCl structure are 5.30 Å, 6.00 Å, 6.13 Å, 6.46 Å, 6.56 Å, respectively and the hardness parameter was taken 0.313 Å for all LaPn. The computed values of SOECs and TOECs are

Table 1 – Temperature dependent SOECs and TOECs of LaPn [in  $10^{10}$  N/m<sup>2</sup>].

Material	Temp. [K]	C <sub>11</sub>	C <sub>12</sub>	C <sub>44</sub>	C <sub>111</sub>	C <sub>112</sub>	C <sub>123</sub>	C <sub>144</sub>	C <sub>166</sub>	C <sub>456</sub>	C <sub>44/C12</sub>	C <sub>144/C456</sub>	C <sub>166/C112</sub>
LaN	100	5.54	1.88	1.99	-84.33	-7.80	2.64	3.20	8.15	3.17	1.05	1.01	1.04
	200	5.71	1.84	1.99	-85.48	-7.60	2.51	3.21	-8.18	3.17	1.08	1.01	1.08
	300	5.92	1.81	2.00	-87.03	-7.44	2.38	3.23	-8.22	3.17	1.10	1.02	1.10
LaP	100	4.39	1.08	1.14	-72.00	-4.37	1.52	1.95	-4.67	1.93	1.05	1.01	1.07
	200	4.58	1.04	1.15	-73.51	-4.18	1.37	1.95	-4.69	1.93	1.10	1.01	1.12
	300	4.77	1.01	1.16	-75.18	-4.00	1.22	1.96	-4.72	1.93	1.15	1.02	1.18
LaAs	100	4.20	0.98	1.04	-69.83	-3.96	1.43	1.78	-4.23	1.77	1.06	1.01	1.07
	200	4.39	0.95	1.05	-71.46	-3.77	1.28	1.79	-4.26	1.77	1.10	1.01	1.13
	300	4.59	0.92	1.05	-73.17	-3.59	1.12	1.79	-4.28	1.77	1.10	1.01	1.19
LaSb	100	3.77	0.78	0.83	-64.66	-3.08	1.13	1.44	-3.34	1.43	1.06	1.01	1.08
	200	3.95	0.75	0.84	-66.32	-2.89	0.97	1.45	-3.36	1.43	1.12	1.01	1.16
	300	4.14	0.74	0.84	-68.03	-2.70	0.81	1.46	-3.39	1.43	1.14	1.02	1.26
LaBi	100	3.65	0.73	0.77	-61.68	-2.86	1.06	1.35	-3.12	1.35	1.05	1.01	1.09
	200	3.83	0.70	0.78	-64.56	-2.67	0.90	1.36	-3.14	1.35	1.12	1.01	1.18
	300	4.02	0.66	0.79	-66.57	-2.48	0.73	1.37	-3.15	1.35	1.20	1.01	1.27

presented in Table 1.

Table 1 depicts that out of nine elastic constants,  $C_{11}$ ,  $C_{14}$ ,  $C_{111}$ ,  $C_{144}$ ,  $C_{166}$ , are increasing with temperature  $C_{12}$ ,  $C_{112}$ ,  $C_{123}$  is decreasing with temperature while  $C_{456}$  is not changing with temperature. This type of behaviour of LaPn is similar to lanthanum monochalcogenides<sup>13</sup>.

The obtained results of SOECs and TOECs are applied to compute the bulk modulus (B), shear or rigidity modulus (G), Young's modulus (Y), Zener's anisotropy (A), tetragonal shear modulus (Cs) toughness to fracture ratio (G/B). These values are given in Table 2.

Pugh<sup>14</sup> proposed a simple relationship, empirically connecting plastic properties with elastic moduli of the material. Larger value of G/B indicated a tendency towards ductility, while brittleness can be predicted by its smaller value. The separation of ductile material from brittle one is calculated by a critical value equal to 0.571. The G/B ratio is found to be 0.60-0.64 for all LaPn, satisfy its brittle character. As of LaPn fulfilled the Born criterion  $B_T = (C_{11} + 2C_{12})/3 > 0$ ,  $C_{44} > 0$ ,  $C_S = (C_{11} - C_{12})/2 > 0$ , so we can say that these materials are stable. The Zener anisotropy ratio is found to be less than one for all LaPn compounds. This indicates that all LaPn compounds are anisotropic materials.

The values of second order elastic constants of LaPn have been used to find out the ultrasonic velocities for longitudinal and shear waves. These velocities in turn are used to evaluate Delye average velocity. The evaluated values are presented in Table 3. It is obvious from Table 3 that values of wave velocities is highest for LaN. This indicates that LaN is more applicable material in comparison to others.

## Conclusion

Summarizing the points of the above discussion following points are drawn.

Table 3 – Ultrasonic velocities ( $V_L$ ,  $V_S$  and  $V_D$  in  $10^3$  m/s) of LaPn at room temperature

Material		Direction		
		<100>	<110>	<111>
LaN	$V_L$	3.07	2.93	2.93
	$V_{S1}$	1.71	1.71	1.73
	$V_{S2}$	1.71	2.46	1.73
LaP	$V_L$	3.02	2.79	2.70
	$V_{S1}$	1.49	1.49	1.77
	$V_{S2}$	1.49	2.68	1.77
LaAs	$V_L$	2.72	2.48	2.39
	$V_{S1}$	1.30	1.30	1.59
	$V_{S2}$	1.30	2.43	1.59
LaSb	$V_L$	2.54	2.26	2.15
	$V_{S1}$	1.15	1.14	1.49
	$V_{S2}$	1.15	2.31	1.49
LaBi	$V_L$	2.22	1.95	1.86
	$V_{S1}$	0.98	0.98	1.30
	$V_{S2}$	0.98	2.03	1.30
		$V_{D<100>}$	$V_{D<110>}$	$V_{D<111>}$
LaN		1.90	2.14	1.92
LaP		1.67	1.96	1.94
LaAs		1.47	1.72	1.74
LaSb		1.29	1.53	1.62
LaBi		1.11	1.31	1.41

L, S and D stand for longitudinal, shear and Debye respectively.

- The elastic constant of LaN is highest so LaN will be more promising for mechanical applications.
- The toughness to fracture ratio, G/B ratio of all compounds is more than 0.571, hence the materials are brittle in nature. LaN is found to be more brittle as compare to other LaPn compounds.
- The anisotropic ratio A is not equal to one, it is

Table 2 – Y, B, G, Cs (all in  $10^{10}$  N/m<sup>2</sup>), A and B/G of LaPn at room temperature.

Material	B	A	G	G/B	Y	Cs
LaN	3.18	0.97	2.02	0.64	5.01	2.06
LaP	2.27	0.61	1.41	0.62	3.50	1.88
LaAs	2.14	0.57	1.31	0.62	3.28	1.83
LaSb	1.86	0.49	1.12	0.60	2.80	1.71
LaBi	1.78	0.47	1.07	0.60	2.67	1.68

less than one for all LaPn compounds. This shows anisotropic behavior of the materials.

- Ultrasonic velocity is found to be highest for LaN along all direction. So it is more suitable for wave propagation.

Preliminary results obtained in this investigation will be very helpful for finding their futuristic applications of these promising lanthanum monopnictides.

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## Ultrasonic speed-density-surface tension correlation for ionic and isotopic liquids

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For the first time ultrasonic velocity- density correlations have applied to ionic and isotopic liquids. Familiar Auerbach relation, Altenburg relation and temperature dependent correlation were employed to calculate surface tension of N-octyl-3-methyl pyridiniumtetrafluoroborate[o3mpy][BF<sub>4</sub>] and five pairs of isotopic liquids named, H<sub>2</sub>O-D<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>-C<sub>6</sub>D<sub>6</sub>, C<sub>6</sub>H<sub>12</sub>-C<sub>6</sub>D<sub>12</sub>, CH<sub>3</sub>OH-CH<sub>3</sub>OD and C<sub>2</sub>H<sub>5</sub>OH-C<sub>2</sub>H<sub>5</sub>OD. All the correlations yield excellent results except Auerbach relation. For the applicability of this relation we have modified it. The modified Auerbach relation also gives good agreement with the experimental values of surface tension.

**Keywords:** Surface tension, ionic liquid, isotopic liquid, ultrasonic velocity.

### Introduction

Since experimental techniques involved in the measurements of density and sound speed are very simple and economical, it is worthwhile to correlate these properties with other equilibrium and transport properties (viscosity, thermal conductivity and diffusion coefficient) of liquids which are cumbersome for experimental measurements. Marcus<sup>1</sup> correlated a number of thermodynamic properties of pure liquids with transport properties and applied to more than 300 pure organic liquids at ambient conditions. In the year 2013, a critical study of some speed-density-surface tension correlation has been presented and tested successfully for pure and ternary liquid mixtures<sup>2</sup>. A comparative study of various correlations as well as with Flory statistical theory has been made.

As far as our knowledge is concerned not such correlations were applied to ionic and isotopic liquids due to scarcity of experimental data. In the present communication we have applied various correlation equations to one ionic liquid and five pairs of isotopic liquids. A modification of most widely used equation is also suggested. The ionic liquid considered as N-octyl-3-methyl pyridinium tetrafluoroborate [o3mpy] [BF<sub>4</sub>].

Ultrasonic speed ( $u$ ), density ( $\rho$ ) and surface tension ( $\sigma$ ) of this liquid has been very accurately and precisely measured by Royo *et al.*<sup>3</sup> over a temperature range of 278.15K to 328.15K. The experimental data for the isotopic liquids are taken from different sources<sup>4-6</sup>. The pairs of isotopic liquids considered for the present study are: H<sub>2</sub>O-D<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>-C<sub>6</sub>D<sub>6</sub>, C<sub>6</sub>H<sub>12</sub>-C<sub>6</sub>D<sub>12</sub>, CH<sub>3</sub>OH-CH<sub>3</sub>OD and C<sub>2</sub>H<sub>5</sub>OH-C<sub>2</sub>H<sub>5</sub>OD.

### Theory

The first interrelationship between ultrasonic velocity and surface tension ( $\sigma$ ) of liquid was proposed by Auerbach<sup>7</sup> in 1948. This relationship has been widely employed upto the very recent years<sup>8-11</sup>. This relationship may be written as :

$$\sigma = 6.4 \times 10^{-4} u \rho^{1/2} \quad (1)$$

where  $\rho$  is the density.

Another relationship between  $\rho$  and  $u$  has been worked out by Sette<sup>12</sup> in 1950. This relation may be expressed as :

$$\sigma = \frac{u \rho^{1/3} M^{1/6}}{5.663 L^{1/6}} \quad (2)$$

where  $M$  is the molar mass and  $L$  the Loschmidt number  $= 2.7 \times 10^9$ . Altenberg relation has been used by few workers<sup>10-12</sup> with successful results for multicomponent liquid systems. Recently Blairs<sup>9</sup> applied the Auerbach relation for a number of liquid metals and suggested the appropriate results as :

$$\log u = 0.5526 \log (\sigma / \rho) + 5.4364 \quad (3)$$

Another empirical relation can be obtained from McGowan equation<sup>13</sup> between isothermal compressibility  $[\beta_t]$  and surface tension as :

$$\beta_t \sigma^{3/2} = 1.33 \times 10^{-8}$$

Since  $\frac{\beta_t}{\beta_s} = \gamma = \frac{C_p}{C_v}$

and  $\beta_s = (u^2 \rho)^{(-1)}$

hence  $\beta_t = \gamma \beta_s = \gamma (\rho u^2)^{-1} = \frac{\gamma}{u^2 \rho} \quad (4)$

where  $\frac{\gamma}{u^2 \rho} \sigma^{3/2} = 1.33 \times 10^{-8}$

So that  $\gamma \sigma^{3/2} = 1.33 \times 10^{-8} u^2 \rho \quad (5)$

It is apparent that all the  $u$ - $\sigma$ - $\rho$  interrelations vide Eqs. (1) to (5) do not contain the temperature of liquid. It is well known that surface tension, ultrasonic velocity and density of liquid vary with temperature. By considering the temperature dependence of  $u$ ,  $\sigma$  and  $\rho$ , Singh<sup>14</sup> obtained a new relation between surface tension-ultrasonic velocity-density of liquid using dimensional analysis. A critical analysis of all the surface tension-ultrasonic velocity-density relations proposed earlier, has been recently<sup>2</sup> reviewed and applied to varieties of liquid

Table 1 – Calculated values of surface tension of ionic liquid N-octyl-3-methyl Pyridinium tetrafluoroborate at different temperatures.

T (K)	(g cm <sup>-3</sup> )	u (ms <sup>-1</sup> )	$\sigma$ (dyne cm <sup>-1</sup> )			
			Eq (1)	Eq (2)	Eq (6)	Eq (7)
278.15	1.1064	1571.90	105.82	39.34	36.94	39.30
280.65	1.1050	1564.90	105.28	39.15	36.75	38.99
283.15	1.1036	1557.70	104.73	38.95	36.55	38.67
285.65	1.1021	1549.20	104.09	38.72	36.30	38.31
288.15	1.1007	1541.20	103.48	38.51	36.07	37.96
290.65	1.0994	1533.50	102.91	38.30	35.86	37.63
293.15	1.0977	1526.30	102.34	38.10	35.65	37.31
295.65	1.0963	1518.70	101.77	37.89	35.43	36.98
298.15	1.0945	1511.40	101.20	37.69	35.21	36.66
300.65	1.0933	1505.40	100.74	37.53	35.06	36.40

Table 2 – Experimental values of ionic liquid N-octyl-3-methyl pyridinium tetrafluoroborate at different temperatures and percentage deviations of calculated  $\sigma$  values alongwith APD values.

T	$\sigma$ (dyne cm <sup>-1</sup> )				% Deviation		
	Eq (2)	Eq (6)	Eq (7)	Exp	Eq (2)	Eq (6)	Eq (7)
278.15	39.34	36.94	39.30	39.82	1.20	7.24	1.30
280.65	39.15	36.75	38.99	39.28	0.33	6.45	0.74
283.15	38.95	36.55	38.67	38.70	-0.66	5.56	0.07
285.65	38.72	36.30	38.31	38.30	-1.11	5.22	-0.01
288.15	38.51	36.07	37.96	37.91	-1.58	4.85	-0.13
290.65	38.30	35.86	37.63	37.52	-2.08	4.43	-0.30
293.15	38.10	35.65	37.31	37.19	-2.45	4.15	-0.32
295.65	37.89	35.43	36.98	36.81	-2.95	3.75	-0.47
298.15	37.69	35.21	36.66	36.51	-3.24	3.55	-0.40
300.65	37.53	35.06	36.40	36.23	-3.58	3.24	-0.47
303.15	37.33	34.84	36.08	35.94	-3.86	3.07	-0.38

and liquid mixtures. Temperature dependent relation as obtained by Singh<sup>14</sup>, can be expressed as :

$$\sigma = 10^{-4} T^{1/3} u^{3/2} \rho \quad (6)$$

Any of these relations were never applied to ionic and isotopic liquids due to scarcity of experimental data of  $\sigma$ ,  $u$  and  $\rho$ . It is purpose of the present paper to apply these relations for ionic and isotopic liquids.

## Results and Discussion

All the aforesaid relations have been applied to ionic liquid N-octyl-3-methylpyridinium tetrafluoroborate [o3mpy][BF<sub>4</sub>]. The relevant data of density ( $\rho$ ), ultrasonic velocity ( $u$ ) and surface tension ( $\sigma$ ) were taken from recent paper<sup>3</sup>. The values of  $\sigma$  were calculated from Eqs. (1), (2) and (6) using the experimental values of  $\rho$  and  $u$  in the temperature range 278.15K - 300.65K. The calculated values of  $\sigma$  are recorded in Table 1.

As can be seen from Table 1, that the calculated values of  $\sigma$  from original Aurbach relation {Eq(1)} are too high in composition to experimental values. In such case Eq

(1) is modified as :

$$\sigma = 5.7 \times 10^{-4} \rho u^{3/2} \quad (7)$$

Calculated values of  $\sigma$  from Eq. (7) are also recorded in Table 1. Table 2 enlists the percentage deviations of calculated  $\sigma$  values from the experimental one along with average percentage (APD) values. It can be clearly seen that the APD values from Eqs. (2), (6) and (7) are fairly in excellent agreement with the experimental values. Out of all the correlations, viz. Altenberg<sup>12</sup>, Pandey *et al.*<sup>6</sup> and modified Auerbach<sup>7</sup>, the later equation yields the best results. Keeping in view the empirical nature, all the present correlations, Eqs. (2), (6), (7) give excellent results for the ionic liquid [o-3mpy][BF<sub>4</sub>].

Also the correlation equations have been applied to five pairs of isotopic liquids at four different temperatures (293.15K and 303.15K). The pairs of isotopic liquids selected for the present investigations are H<sub>2</sub>O-D<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>-C<sub>6</sub>D<sub>6</sub>, C<sub>6</sub>H<sub>12</sub>-C<sub>6</sub>D<sub>12</sub>, CH<sub>3</sub>OH-CH<sub>3</sub>OD and C<sub>2</sub>H<sub>5</sub>OH-C<sub>2</sub>H<sub>5</sub>OD. The experimental data of  $\rho$  and  $u$  were taken from different sources<sup>1-6</sup>.

Table 3 – Calculated values of  $\sigma$  (dyne cm<sup>-1</sup>) for some isotopic liquids.

Isotopic Liquid	T (K)	$\rho$ (gcc <sup>-1</sup> )	$u$ (ms <sup>-1</sup> )	$\sigma$ (dyne cm <sup>-1</sup> )			
				Eq (1)	Eq (2)	Eq (6)	Eq (7)
H <sub>2</sub> O	293.15	0.9982	1483.00	94.83	24.46	37.87	35.92
H <sub>2</sub> O	303.15	0.9966	1511.00	96.54	24.91	39.32	36.88
D <sub>2</sub> O	293.15	1.1076	1386.00	93.35	24.09	37.97	36.00
D <sub>2</sub> O	303.15	1.1044	1413.00	95.04	24.53	39.41	36.96
C <sub>6</sub> H <sub>6</sub>	293.15	0.8791	1327.00	79.63	26.79	28.23	26.77
C <sub>6</sub> H <sub>6</sub>	303.15	0.8670	1280.00	76.28	25.72	26.67	25.01
C <sub>6</sub> D <sub>6</sub>	293.15	0.9431	1246.00	77.44	26.07	27.55	26.13
C <sub>6</sub> D <sub>6</sub>	303.15	0.9318	1201.00	74.20	25.03	26.05	24.43
C <sub>6</sub> H <sub>12</sub>	293.15	0.7785	1280.00	72.28	25.12	23.68	22.46
C <sub>6</sub> H <sub>12</sub>	303.15	0.7695	1230.00	69.05	24.05	22.30	20.91
C <sub>6</sub> D <sub>12</sub>	293.15	0.8902	1153.00	69.62	24.20	23.15	21.96
C <sub>6</sub> D <sub>12</sub>	303.15	0.8793	1110.00	66.62	23.20	21.85	20.49
CH <sub>3</sub> OH	293.15	0.7912	1122.00	63.87	18.85	19.75	18.73
CH <sub>3</sub> OH	303.15	0.7829	1089.00	61.67	18.23	18.90	17.73
CH <sub>3</sub> OD	293.15	0.8082	1103.00	63.46	18.76	19.67	18.65
CH <sub>3</sub> OD	303.15	0.7998	1071.00	61.30	18.15	18.83	17.66
C <sub>2</sub> H <sub>5</sub> OH	293.15	0.7904	1167.00	66.40	20.82	20.93	19.85
C <sub>2</sub> H <sub>5</sub> OH	303.15	0.7806	1133.00	64.06	20.13	20.00	18.75
C <sub>2</sub> H <sub>5</sub> OD	293.15	0.8063	1145.00	65.80	20.64	20.75	19.68
C <sub>2</sub> H <sub>5</sub> OD	303.15	0.7974	1111.00	63.49	19.95	19.84	18.60

Table 4 – Some of the experimental values of  $\sigma$  available at given temperature.

T°C	Liquid	$\sigma$ (dyne cm <sup>-1</sup> )
20	C <sub>6</sub> H <sub>6</sub>	28.88
20	C <sub>6</sub> D <sub>6</sub>	28.30
20	C <sub>6</sub> H <sub>12</sub>	24.94
20	C <sub>6</sub> D <sub>12</sub>	24.62
100	H <sub>2</sub> O	58.60
100	D <sub>2</sub> O	58.20
200	H <sub>2</sub> O	37.80
200	D <sub>2</sub> O	36.60

Calculated values of  $\sigma$  for all the isotopic liquids mentioned from Eqs (1), (2), (6) and (7) are recorded in Table 3. No experimental values of  $\sigma$  for these liquids are available at the desired temperature, so comparison could not be made. However we have collected some experimental values of  $\sigma$  for some isotopes available in literature <sup>4</sup>. These values are presented in Table 4.

It is apparent from the experimental values of  $\sigma$  that surface tension of isotopic liquids increases by increasing the temperature which is also revealed from the calculated values from all the relations (Table 3).

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# Ultrasonic wave propagation through calcium oxide single crystal in high temperature range

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The calcium oxide crystal possesses well developed fcc structure and is divalent in nature. Oxides and silicates make up the bulk of the Earth's mantle and crust, and thus it is important to predict their behavior for the study of seismic and volcanic activities. The second order elastic constants (SOECs) were computed using Coulomb and Born-Mayer potential in the temperature range 100-1000 K. The ultrasonic velocities were calculated using these values of second order elastic constants,  $C_{ij}$  and density  $\rho$ . In this study temperature dependent velocities in calcium oxide have been calculated along  $\langle 100 \rangle$ ,  $\langle 110 \rangle$  and  $\langle 111 \rangle$  crystallographic directions and the results are presented graphically and discussed in depth.

**Keywords:** Micro structural properties, Seismic and volcanic activities, acoustic wave velocities, second order elastic constants.

## Introduction

Ultrasonics offer the possibility to characterize materials behaviour based on the physical mechanism to predict potential performance of the materials<sup>1,2</sup>. Calcium oxide is a key **ingradient** in manufacture of building materials, refractory lining dehydrating agent, etc. and has many other applications. Various studies have been made by scientists on calcium oxides<sup>3</sup>. Quasri *et al.*<sup>3</sup> used waste shell as a bio resource of calcium oxide in catalyzing a transesterification to produce biodiesel. The effect of nanotitanium oxide dosage, Ca/S molar ratio, temperature on calcium oxide combustion desulfurization efficiency was studied by Wang and Zhaq<sup>4</sup>. But by the knowledge of authors, no ultrasonic study has been made on calcium oxide. This motivated to authors to present ultrasonic properties on calcium oxide.

In present study, we first computed second order elastic constants in the temperature range 100 K - 1000 K. These elastic constants are utilized to compute ultrasonic velocities along  $\langle 100 \rangle$ ,  $\langle 110 \rangle$  and  $\langle 111 \rangle$  direction in the same temperature regime. The results are discussed

and compared with similar type of materials.

## Theoretical Approach

The calcium oxide possesses face centered cubic crystal structure. The potential used for evaluation of second elastic constants (SOECs) is taken as the sum of Coulomb and Börn-Mayer potentials.

$$\begin{aligned}\phi(r) &= \phi(C) + \phi(B) \\ &= \{\pm (e^2/r)\} + A \exp(-r/q)\end{aligned}\quad (1)$$

where  $\phi(C)$  is the Coulomb potential and  $\phi(B)$  is the Börn-Mayer potential,  $e$  is the electronic charge,  $r$  is the nearest-neighbour distance,  $q$  is the hardness parameter and  $A$  is the strength parameter. The elastic energy density for a crystal of a cubic symmetry can be expanded up to quartic terms as shown below :

$$\begin{aligned}U_0 &= U_2 + U_3 + U_4 \\ &= [1/2!] C_{ijkl} \alpha_{ij} \alpha_{kl} + [1/3!] C_{ijklmn} \alpha_{ij} \alpha_{kl} \alpha_{mn} + [1/4!] \\ &\quad C_{ijklmnpq} \alpha_{ij} \alpha_{kl} \alpha_{mn} \alpha_{pq}\end{aligned}\quad (2)$$

where  $C_{ijkl}$ ,  $C_{ijklmn}$  and  $C_{ijklmnpq}$  are the SOECs, TOECs and FOECs in tensorial form;  $\alpha_{ij}$  are the Lagrangian strain

components;  $C_{IJ}$ ,  $C_{IJK}$  and  $C_{IJKL}$  are the SOECs, TOECs and FOECs in Brügger's definition and Voigt notations.

Following Brügger's<sup>5</sup> definition of elastic constants at absolute zero, second order elastic constants (SOECs) are obtained. According to lattice dynamics developed by Leibfried and Hahn<sup>6</sup>, lattice energy changes with temperatures. Hence adding vibrational energy contribution to the static elastic constants, one gets second order elastic constants ( $C_{ij}$ ) at the required temperature.

$$C_{ij} = C_{ij}^0 + C_{ij}^{vib} \quad (3)$$

There are three types of ultrasonic velocities one longitudinal ( $V_l$ ) and two shear ( $V_{s1}$  and  $V_{s2}$ ) for each direction of propagation in cubic crystals<sup>7,8</sup>. The ultrasonic velocities can be calculated using calculated values of second order elastic constants  $C_{ij}$ 's and density  $\rho$ . The expressions for velocities are as follows :

Along the  $\langle 100 \rangle$  crystallographic direction

$$V_l = \sqrt{\frac{C_{11}}{\rho}} \quad V_{s1} = V_{s2} = \sqrt{\frac{C_{44}}{\rho}} \quad (4)$$

Along the  $\langle 110 \rangle$  crystallographic direction

$$V_l = \sqrt{\frac{C_{11} + C_{12} + 2C_{44}}{2\rho}} \quad V_{s1} = \sqrt{\frac{C_{44}}{\rho}} \quad V_{s2} = \sqrt{\frac{C_{11} - C_{12}}{\rho}} \quad (5)$$

Along the  $\langle 111 \rangle$  crystallographic direction

$$V_l = \sqrt{\frac{C_{11} + 2C_{12} + 4C_{44}}{3\rho}} \quad V_{s1} = V_{s2} = \sqrt{\frac{C_{11} - C_{12} + C_{44}}{3\rho}} \quad (6)$$

and the Debye's average velocity is given by

$$\bar{V}_D = \left[ \frac{1}{3} \left( \frac{1}{V_l^3} + \frac{2}{V_s^3} \right) \right]^{-\frac{1}{3}} \quad (7)$$

## Results and Discussion

Second and third order elastic constants (SOECs and TOECs) for calcium oxide are calculated (taking  $r_0 = 2.3925 \times 10^{-8}$  cm and  $b = 0.345 \times 10^{-8}$  cm)<sup>9,10</sup> at different temperatures (100 – 1000 K) using the theory described

Table 1 – The SOECs in  $10^{10}$  N/m<sup>2</sup> for CaO

Temp. (K)	$C_{11}$	$C_{12}$	$C_{44}$
100	19.96	12.74	12.85
200	20.20	12.65	12.79
300	20.67	12.52	12.71
400	21.23	12.38	12.63
500	21.84	12.24	12.55
600	22.47	12.10	12.47
700	23.12	11.97	12.39
800	23.78	11.85	12.33
900	24.44	11.73	12.27
1000	25.11	11.62	12.21

in section 2.1. Also the ultrasonic velocities along different crystallographic directions are calculated from 100 K – 1000 K. The calculated values of second and third order elastic constants are given in Table 1.

The temperature variation of ultrasonic velocities for longitudinal and shear waves and the Debye average velocity for different crystallographic directions are presented in Table 2.

Table 2 – The longitudinal, shear wave and Debye average velocities in  $10^3$  m/s for CaO crystal along different crystallographic directions.

Temp (K)	$\langle 100 \rangle$			$\langle 110 \rangle$				$\langle 111 \rangle$		
	$V_L$	$V_{S1} = V_{S2}$	$V_D$	$V_L$	$V_{S1}$	$V_{S2}$	$V_D$	$V_L$	$V_{S1} = V_{S2}$	$V_D$
100	7.48	6.20	6.53	9.20	6.20	3.19	6.77	9.71	4.39	4.95
200	7.49	6.19	6.52	9.17	6.19	3.23	6.76	9.66	4.44	5.00
300	7.52	6.17	6.51	9.12	6.17	3.38	6.73	9.60	4.50	5.07
400	7.56	6.15	6.50	9.08	6.15	3.53	6.71	9.53	4.58	5.14
500	7.59	6.13	6.49	9.04	6.13	3.69	6.69	9.47	4.65	5.22
600	7.63	6.11	6.48	9.00	6.11	3.84	6.66	9.41	4.72	5.29
700	7.67	6.09	6.47	8.96	6.09	3.98	6.64	9.34	4.79	5.36
800	7.72	6.08	6.47	8.91	6.08	4.12	6.62	9.28	4.86	5.43
900	7.76	6.06	6.46	8.88	6.06	4.25	6.61	9.23	4.93	5.50
1000	7.80	6.05	6.46	8.85	6.05	4.38	6.59	9.17	5.00	5.57

It is obvious from Table 2 that along  $\langle 100 \rangle$  direction longitudinal wave velocity increases while shear wave velocity decreases and also the Debye average velocity decreases as temperature increases. Along  $\langle 110 \rangle$  direction the situation is little different where longitudinal wave velocity decreases, shear wave velocity for direction of polarisation  $\langle 001 \rangle$  decreases while shear wave velocity for direction of polarization  $\langle 1\bar{1}0 \rangle$  increases also the Debye average velocity decreases as temperature increases. Along  $\langle 111 \rangle$  direction longitudinal wave velocity decreases while shear wave velocity increases also the Debye average velocity increases as temperature increases.

As experimental values are not available for all crystallographic directions, we therefore compare our results with other NaCl-type materials such as rock forming oxides (CdO, CoO and FeO)<sup>13</sup>.

## Conclusion

From the ultrasonic velocity values along different directions it is evident that the ultrasonic velocity is different along different directions *i.e.* varies with the orientation of the crystal, similar behaviour has been observed by some investigators in other crystals.

Ultrasonic wave velocity is higher along  $\langle 111 \rangle$  direction and hence this crystallographic direction is more suitable for acoustic purpose of the crystal. All these discussions are sufficient to conclude that the ultrasonic wave velocity is important and characteristic property of any substance.

This work presents a method for determining various elastic properties of a solid its with dependence on its internal crystallographic structure and nature.

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## Thermo-acoustical characteristics of binary mixture of acetone and aniline in pyridine

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The two binary systems : acetone and pyridine and aniline and pyridine at different temperatures have been studied to determine the different ultrasonic parameters. The measured values of density, ultrasonic velocity and coefficient of viscosity shows increasing trend with the increase in concentration of acetone in the mixture of pyridine. The nature of the curves of compressibility, free length, free volume show decreasing trend with that of increase in the concentration of acetone or aniline in their respective mixture. The internal pressure of both the binary mixture is found to increase with the corresponding increase in concentration of the acetone or aniline in their binary mixture. The excess values of the above parameters such as, excess velocity  $v^E$ , excess adiabatic compressibility  $\kappa_\alpha^E$ , excess free length  $L_f^E$ , excess free volume  $V_f^E$  and excess internal pressure  $\pi_i^E$  have also been evaluated and the results presented graphically. The data confirms the existence of interaction between the components of the binary mixtures.

**Keywords:** Molecular interaction, free volume, ultrasonic velocity, internal pressure, excess parameters.

### Introduction

The knowledge of behaviour of the molecules in the liquid and its mixture is very important for the application of that liquid in pure and applied fields, such as biological, automobile, pharmaceutical, chemical, industrial, and other research areas. It is a well known fact that in spite of many other studies used to understand the nature of intermolecular interaction the ultrasonic studies have played an important role. The dependence of ultrasonic velocity of liquid on the bonding forces between the atoms and molecules provides the knowledge about the nature of molecular interactions occurring in the liquids and their mixtures<sup>1-5</sup>.

The liquids acetone, aniline and pyridine are very useful liquids as they have wide applications in different areas. Pyridine is used currently in the extraction process for coal to analyse its compounds and in the manufacture of vitamin B6 and other drugs. Similarly Acetone and Aniline are also important in many applications such as bio-medical, chemical and pharmaceuticals. These

liquids and their mixtures are of interest to organic chemists to know about the type of bond and the complexes<sup>6-8</sup>.

The different ultrasonic parameters are to be discussed in terms of their excess values rather than actual in order to understand the nature of molecular interactions between the components of the liquid mixtures.

### Materials and Methods

The chemicals used here Pyridine, Acetone and Aniline with purity of 99.5% of AR grade were obtained commercially and used without further purification. The first binary mixture was prepared with the addition of pyridine in the liquid acetone for different mole fraction and second binary mixture was prepared with the addition of aniline in the liquid pyridine for different mole fraction and stored in specially designed containers to avoid air contact. All the mixtures prepared were utilized within the 24 hours of its preparation. The Ultrasonic velocity ( $v$ ) measurements for different concentrations of pure

liquids and binary mixtures were carried out by using Ultrasonic Interferometer at 1 MHz. The densities were measured using specific gravity bottle. The coefficients of viscosities were measured using the suspended level viscometer. The density ( $\rho$ ), ultrasonic velocity ( $v$ ) and coefficient of viscosity ( $\eta$ ) are used to evaluate various parameters by standard relations<sup>9,10</sup>.

## Results and Discussion

The first binary mixture under investigation is prepared by increasing the molar concentration of the liquid pyridine in the liquid acetone and second binary mixture is prepared by increasing the molar concentration of the liquid aniline in the liquid pyridine. As the density of pyridine is more than the density of acetone any increase in the concentration of pyridine in the mixture increases the density ( $\rho$ ) of the 1st binary mixture also the density of aniline is more than the density of pyridine any increase in the concentration of aniline in the mixture increases the density of the second binary mixture which is confirmed from the observations. The value of ultrasonic velocity ( $v$ ) increases in both binary mixtures with the increase in concentration of pyridine in the 1<sup>st</sup> mixture and aniline in the second binary mixture. The similar trend is also found in the values of coefficient of viscosity ( $\eta$ ), but the adiabatic compressibility ( $\beta_\alpha$ ), Free length ( $L_f$ ) and free volume shows the opposite trend at the three temperatures 298, 303 and 308 K, here the values internal pressure ( $\pi_i$ ) are increasing with the increase in concentration of pyridine in first and aniline in the second binary mixture.

The coefficient of viscosity showing increasing trend is the indication of existence of frictional resistive forces that may be due to a change in effective molecular area by the cohesive or adhesive forces. It is clear that intermolecular free length depends upon intermolecular attractive and repulsive force<sup>10</sup>. The decrease in free length is responsible for the decrease in free volume which ultimately increases the internal pressure of the system. This result is confirmed from the plots of these parameters. The plots of these parameters with concentrations and temperature conforms the non linear variation (Figs. 1 and 3). The decrease in the values of free length and free volume with increasing concentration can be concluded as there is significant interaction between the two liquids in both the cases<sup>11-16</sup>. This suggests the close packing of the molecules, which may be due to the increasing magnitude of the interaction.

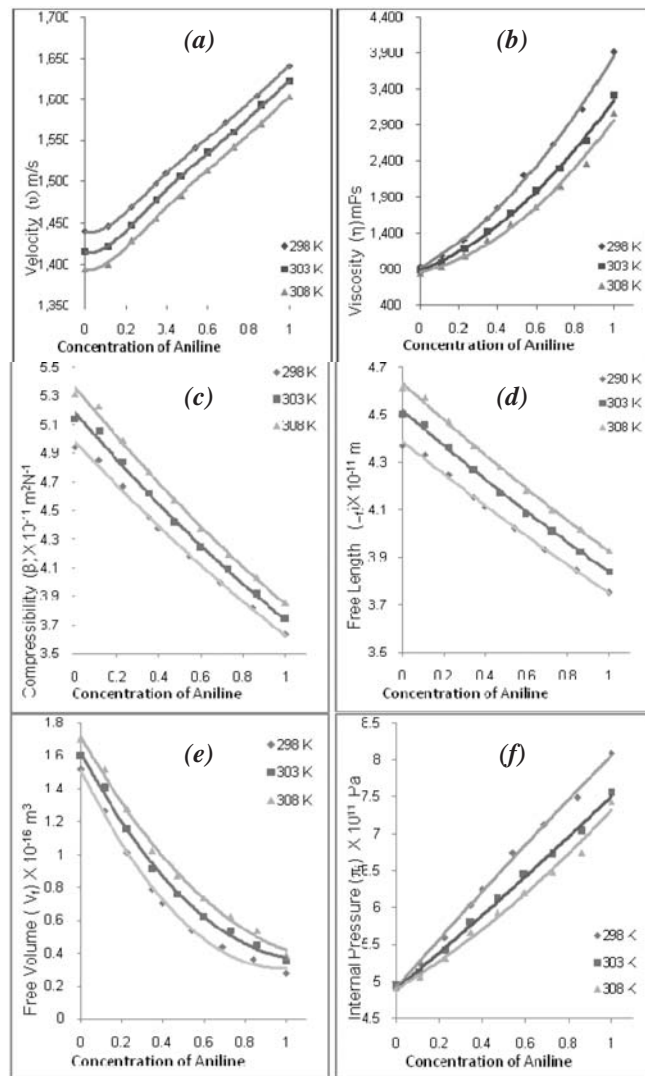


Fig. 1 Ultrasonic velocity, viscosity, adiabatic compressibility, free length, free volume and internal pressure for varying concentration of aniline at 298, 303 and 308 K temperatures.

The extent of deviation and sign of excess values of the thermo dynamical parameters depends on the strength of interaction between unlike molecules<sup>17,18</sup>. The plots of excess values of different parameters for varying concentration of binary mixture at different temperature are shown in Figs. 2 and 4. The excess velocity is seen to be negative which can be concluded as the making and breaking of a structure. At lower temperature the excess velocity is more negative. This means the strength of interaction decreases at higher temperature. The excess values of coefficient of viscosity are observed to be negative. The similar trend is also seen in the excess

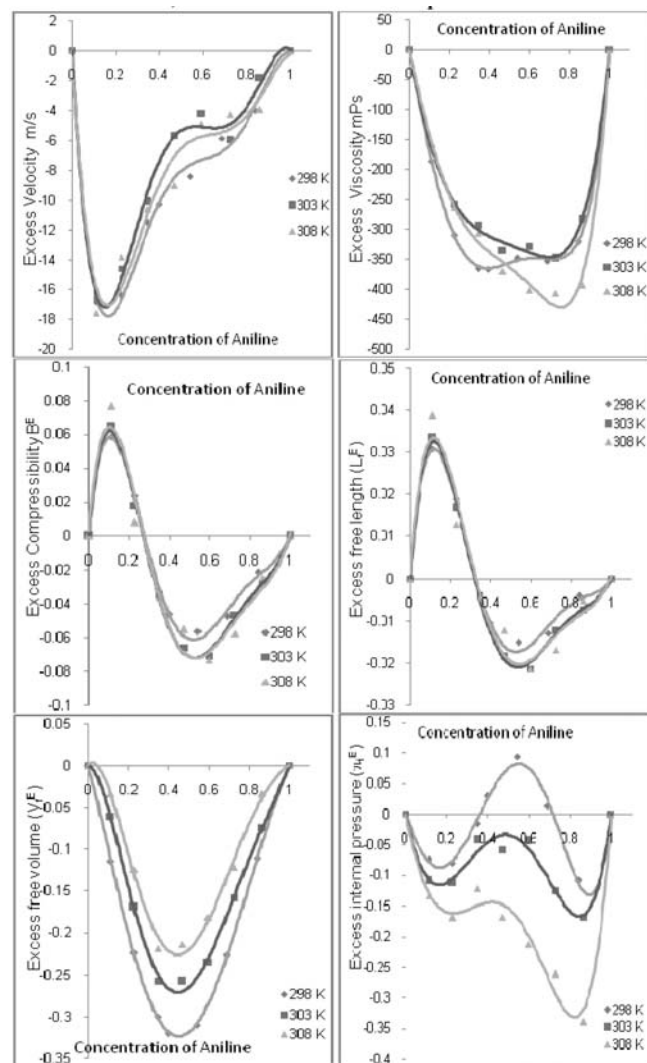


Fig. 2 The excess values of ultrasonic velocity, viscosity, adiabatic compressibility, free length, free volume and internal pressure for varying concentration of aniline at 298, 303 and 308 K temperatures.

free volume which indicates the presence of strong interaction between the components of the mixture. The sign of excess free length plays a vital role in assessing compactness due to molecular interaction through dipole-dipole interaction<sup>19,20</sup> the increase in compactness enhances structure making and excess free length tends to negative values. Excess compressibility and free length are negative after the 0.3 concentration of aniline in the binary mixture. This clearly indicates the weak and strong interactions are prevailing in the binary mixture. The more negative values means the strong attractive or repulsive forces are acting between the different components of both the mixtures.

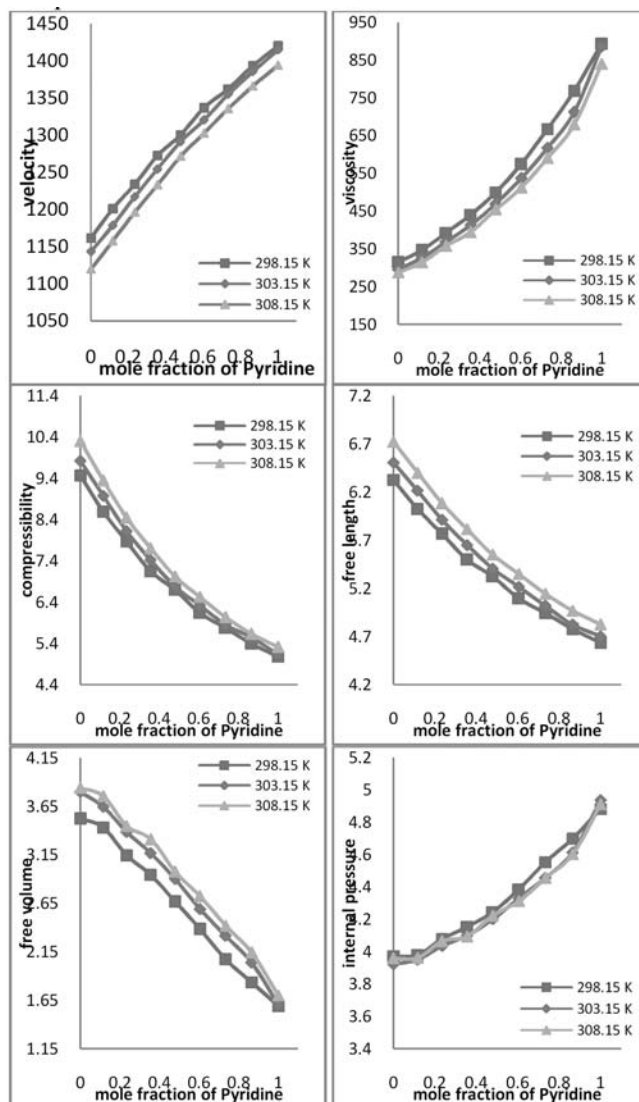


Fig. 3 The ultrasonic velocity, viscosity, adiabatic compressibility, free length, free volume and internal pressure for varying concentration of pyridine in acetone at 298, 303 and 308 K temperatures.

## Conclusion

The first binary mixture pyridine and acetone and second mixture of aniline and pyridine are under investigation for the molecular interaction study at different temperatures. In both the mixtures, the increase in ultrasonic velocity, density, coefficient of viscosity and internal pressure with the increase in molar concentration of pyridine in first and aniline in the second mixture and the decrease in adiabatic compressibility, intermolecular free length and free volume confirms the existence of intermolecular interaction between the components of the mixture. The negative values of excess

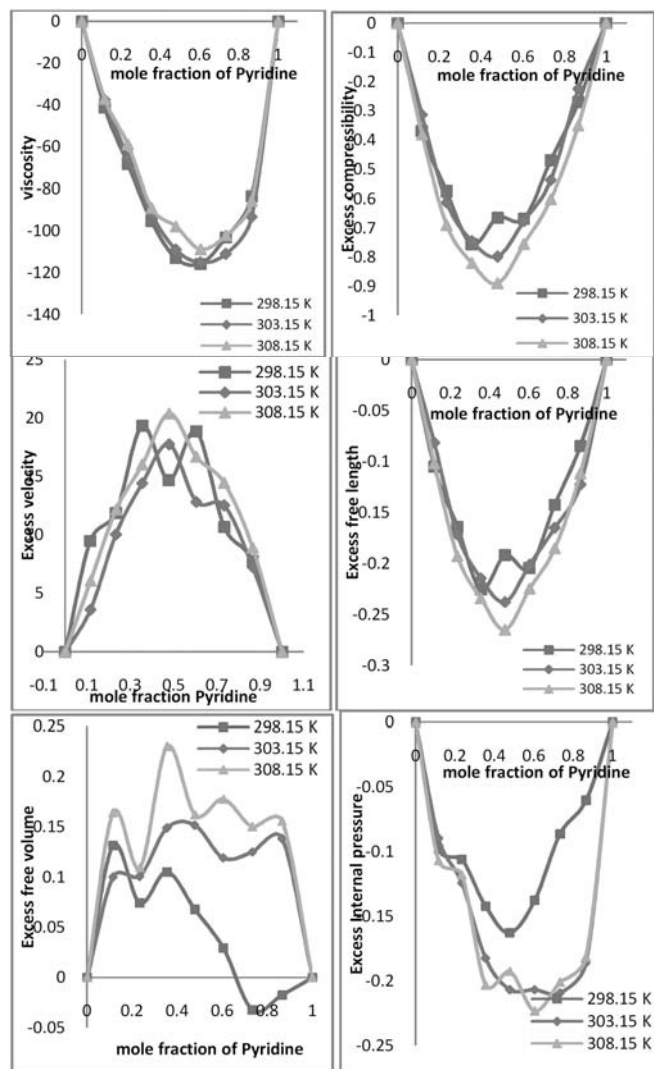


Fig. 4 Plots of excess values of ultrasonic velocity, viscosity, adiabatic compressibility, free length, free volume and internal pressure for varying concentration of pyridine at 298, 303 and 308 K.

ultrasonic velocity ( $v^E$ ), excess viscosity ( $\eta^E$ ), excess free volume ( $V_f^E$ ) and the negative excess compressibility ( $\beta_\alpha^E$ ) and excess free length ( $L_f^E$ ) after the 0.3 molar concentration of aniline in the mixture also non linear but more negative excess internal pressure ( $\pi_i^E$ ) for the different concentration of the aniline in the binary mixture at temperatures 298, 303, 308 K confirms the presence of strong dispersive interaction between the components of molecule than the binary mixture of pyridine in acetone. The non-linearity of the curve is common for all compositions. The non linear variation of all the parameters indicates existence of dipole-dipole interaction between the different molecules of the compounds in the mixture.

## Acknowledgements

Authors are thankful to Principals of their respective institutes for helping in carrying out the research work.

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## Symposium Report on XXI National Symposium on Ultrasonics (NSU 2016)

The Symposium and the General Body Meeting of the Ultrasonics Society of India, was held in S. N. Bose National Centre for Basic Sciences (SNBNCBS), Kolkata, from 08 to 10<sup>th</sup>, November, 2016. Prof. P. K. Mukhopadhyay of this centre was Convenor and Prof. B. Pal, S.N. Bose Institute of Nuclear Physics (SINP) was the Co-Convenor of the Symposium Organizing Committee. Other members from SNBNCBS were Prof. S. Mukherjee, Dr. T. Paramanik (Secretary), Dr. A. Dutta (Treasurer) and Ms. D. Bhattacharyya. The members from SINP were Prof. S. Banerjee, Prof. M. Roy, and Prof. S. Mukherjee, I.I.E.S.T. The website of the Symposium was kept continuously updated with relevant information for the benefit of participants. User-friendly arrangements were made for invited speakers and members for their transportation and stay.

The symposium was inaugurated by Prof. Samit Kumar Roy, Director of SNBNCBS and Prof. Vikram Kumar, President of USI. Prof. E.S.R. Gopal, Emeritus Professor, IISc, Bangalore, delivered a thought provoking keynote address on the use of ultrasonics to get more fundamental definition of temperature. Besides the Key Note Address, there were two more Plenary Talks by Dr. Krishnan Balasubramanian, IIT, Madras, on "Ultrasonic Waveguide Based Sensing and Measurements" and by Prof. S Bhattacharya, TIFR, on "Amorphous to Amorphous Transition in Athermal Particle Rafts". There were other invited talks, oral presentations and poster sessions in all the three days.

A GB meeting was arranged in the evening of the first day, chaired by the President of USI, Prof. Vikram Kumar. Many deliberations, including functioning and membership issues took place. An important decision was taken to hold in the next NSU at Central University of Himachal Pradesh, Sahapur, Dharamshala, Kangra 176206 in 2017 under guidance of Dr. O.S.K.S. Sastri, Dean School of Physics and Material Science.

The second day a symposium dinner was organized in a nearby place, with family members of some of the other faculty members of S.N.B.N.C.B.S. also joining in. It was enjoyed by all. In the final session of the third day, there was a result announcement for two best poster prizes and one for oral presentation - all for younger participants only. The symposium ended with a valedictory and concluding session. The highlights of this session were the following awards.

**M. Pancholy Award :** Minati Kumar Sahu, J Swaminathan, N R Bandyopadhyay for their Oral Presentation on "Application of Second Harmonic Based Ultrasonic Signal Analysis for Evaluation of Micro-structural Changes due to Creep Damage in Ni-based Super Alloy".

**Best Poster Award :** Md Sarower Hossain, Animesh Basak, Barnana Pal and P K Mukhopadhyay for their Paper on "Incidence of Ultrasonic Wave through Newtonian & Non-Newtonian Fluids".

**Second Best Poster Paper :** Sangeeta Sagar, Laxmi Kumari and Manisha Gupta for their paper on "Thermoacoustical Analysis of Binary Mixtures of N-N-Dimethyl formamide (DMF) with BAE and 1-BuOH".

**Best Poster Paper Award from the Organizers :** Matukumilli V D Prasad and Baidurya Bhattacharya for their Paper on "Acoustic Phonon Propagation and Interfacial Scattering in Nano structures".

**Dr. T K Saksena Award - 2015 :** Dr. Govind Kumar Sharma, Indira Gandhi Centre for Atomic Research (IGCAR), Kalpakkam, for his Thesis on "Ultrasonic NDE of Type 316 Austenitic Steel by Time Frequency Analysis".

**Dr. T K Saksena Award - 2016 :** Dr. Deep Gupta, Viveswaraya National Institute of Technology (VNIT), Nagpur, for his Thesis on "Denoising and Segmentation of Ultrasound Medical Images".

As a summary of numbers, there was a total of 97 participants in the meeting, out of which there were 30 students. There were 21 invited talks, 20 contributory orals and 36 posters. There were 8 technical sessions and two poster sessions. Six eminent medical practitioners from medical colleges and renowned hospitals in Kolkata gave excellent exposition of medical uses of diagnostic ultrasonics in health care, like echo cardiography, abdominal ultrasonogram and ophthalmic uses of USG. The participants came in from all parts of India, like IISc and JNCSC, Bangalore, NPL, Delhi, NML and Tata Steel, Jamshedpur, universities and colleges from around Allahabad, Nagpur, Lucknow, Cuttack, Sambalpur, Tuticorin, IITs and IISERs like KGP, Mohali, Madras *etc.*, came in large numbers. Finally, we also thank the partial support received from various funding agencies and vendors. These include SINP, Kolkata, SERB, CSIR, DRDO and ICMR, New Delhi, as well as M/s. Bosssa Nova Technologies, USA, M/s. ICON Analytical and M/s. Bansal Diagnostics from Kolkata.

Last but not the least the convenor placed on record **in** thanks to USI for whole hearted support by allowing us to hold the symposium under its banner and for all other cooperation that we required from time to time. In particular we thank the support of Prof. R. R. Yadav who was instrumental in getting the meeting take place in S.N.B.N.C.B.S. and Dr. Y. K. Yadav whom we called up time and again for help in securing support for liaising with various financial institutions, and general advice about contacting the members and many other trivial and nontrivial issues.

**Prof. P. K. Mukhopadhyay**

Convener-NSU-2016

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We are highly thankful to the reviewers of the published articles in the Journal of Pure & Applied Ultrasonics, Vol. 38, Nos. (1-4), 2016 for giving constructive comments to improve the quality of the articles.

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# Journal of Pure and Applied Ultrasonics

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**Units and Abbreviations** - Authors should use SI units wherever possible.