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# Analysis of the elastic properties of an interpenetrating AlSi12-Al<sub>2</sub>O<sub>3</sub> composite using Ultrasound Phase Spectroscopy

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## Abstract

An interpenetrating composite fabricated by squeeze-casting a eutectic aluminium-silicon alloy into a porous alumina preform is studied in this work. The preform was fabricated by pyrolysis of cellulose fibres used as pore forming agent, pressing of the green ceramic body and subsequent sintering of alumina particles. The resulting preform had both micropores within the ceramic walls and macropores between those walls, which were infiltrated by the liquid metal. Composites with alumina contents varied in the range of 18-65 vol% were studied. Three longitudinal and three shear elastic constants of the composites were determined using ultrasound phase spectroscopy on rectangular parallelepiped samples. Complete stiffness matrix of one sample was determined by modifying the sample geometry by cutting at the corners of the sample and subsequent ultrasonic measurements. All composites exhibit a moderately anisotropic behaviour, which can be attributed to a non-random pore orientation distribution caused by uni-axial pressing of the preforms prior to sintering. The experimental results are compared with several theoretical micromechanical models.

## Keywords

A. Metal-matrix composites (MMCs), C. Anisotropy, C. Elastic properties, D. Ultrasonics, E. Liquid metal infiltration (LMI)

## 1. Introduction

Metal matrix composites (MMC) typically exhibit enhanced specific mechanical properties in comparison to monolithic metals of which they are made [1]. MMCs can be classified according to the form of the reinforcement (such as fibres, particles, whiskers or short fibres, interpenetrating etc). Among these, MMCs having an interpenetrating structure have been shown to possess better strength, toughness and wear resistance in comparison to other composite structures [2, 3]. Hence, much recent work has focussed on the development and analysis of interpenetrating metal matrix composites. These include MMCs fabricated by reaction synthesis [4, 5, 6], by melt infiltration in wood ceramics [7], in 3-D periodic preforms produced by robotic deposition [8], in porous ceramic preforms fabricated by freeze-casting [9, 10]. Recently, several studies have been carried out to analyse the elastic properties of composites having an interpenetrating structure [11, 12, 13]. Young's modulus and/or shear modulus of the composites were determined and the results were compared with numerous micromechanical models such as Hashin-Shtrikman (HS) bounds [14], Ravichandran (R) model [15], Tsuchinskii (T) model [16] and effective medium approximation (EMA) [13].

The purpose of the present study is to carry out in depth analysis of the elastic properties of an interpenetrating AlSi12-Al<sub>2</sub>O<sub>3</sub> composite. The composites were fabricated by direct squeeze-casting a eutectic aluminium-silicon (AlSi12) melt in an open porous alumina preform. The preform was fabricated by pyrolysis of cellulose fibres used as pore forming agent, pressing of the green ceramic body and subsequent

sintering of alumina particles. Previous studies [11, 12, 13] on the elastic properties of interpenetrating metal/ceramic composites considered the composite to be isotropic. In the current study, the three longitudinal and the three shear elastic constants of composites with alumina contents varying in the range of 18-65 vol% will be studied using ultrasound phase spectroscopy (UPS) [17]. As longitudinal and shear elastic constants are determined along all three directions, the extent of anisotropy can be investigated. The results are compared with several micromechanical models (HS-model, R-model, Feng (F) model, self consistent (SC) method and differential effective medium (DEM) approximation). These analyses show that the studied composites are not isotropic. Hence, assuming orthotropic symmetry, all nine elastic constants of the stiffness matrix of one sample will be determined. In a previous study, we investigated the mechanism of internal load transfer in this material under compression and tension [18]. The current study, along with this previous one helps to thoroughly understand the mechanics of this complex composite structure.

## **2. Experimental procedure**

### **2.1 Specimen material**

The composite material was fabricated by the Materials Research Team of Aalen University of Applied Sciences, Germany, following a processing route thoroughly described in Ref. [19]. Commercially available alumina powder (CL2500 from Almatix GmbH, Germany with particle size  $1.8 \mu\text{m}/d_{50}$  and nominal alumina content 99.8%) was used to prepare porous ceramic preforms. Cellulose fibres (Arbocel P290 from J. Rettenmaier & Söhne GmbH & Co. KG, Germany) having nominal average length of  $150 \mu\text{m}$  were added as pore forming agent. The alumina powder particles were first degglomerated by wet ball milling, mixed with the pore forming agent and freeze-dried.

The powders were subsequently pressed uni-directionally under 100 MPa applied pressure into plates having nominal dimensions  $65 \times 45 \times 10 \text{ mm}^3$  and then sintered in an electrically heated tube furnace under an oxidizing atmosphere. The sintering temperature was  $1550 \text{ }^\circ\text{C}$  and during this process pyrolysis of the pore forming agents also occurred. Figure 1 shows a scanning electron microscopy (SEM) micrograph of the uninfiltreated ceramic preform with a nominal ceramic content of 41 vol.%. Several regions with traces of cellulose fibres can be observed in this image (macroporosity), and one such location is indicated by an arrow. Micropores visible within the ceramic rich regions result from incomplete sintering of the preform. The porous preforms were subsequently infiltrated with a eutectic aluminium-silicon (AlSi12) melt via direct squeeze-casting along the same direction as that of powder pressing before sintering. Prior to squeeze-casting, the preform and the melt were preheated to  $800 \text{ }^\circ\text{C}$ , while the tool was heated up to  $420 \text{ }^\circ\text{C}$ . 100 MPa infiltration pressure was applied.

The metal/ceramic composite samples were fabricated in the form of plates. Five different plates with varying alumina contents were available. In the following, these plates are named as Types A-E. For ultrasonic analysis, several rectangular parallelepiped samples were cut from each plate using a  $500 \text{ }\mu\text{m}$  diameter diamond coated steel wire saw. The samples had nominal dimensions in the range of 6-10 mm and the numbers of samples in each composite type are listed in Table 1. For each sample, the direction parallel to the direction of preform pressing and squeeze-casting was marked as direction 1, and the two directions orthogonal to it were marked as directions 2 and 3, respectively. Density of each sample was determined following Archimedes' principle by immersing it in distilled water. Alumina content of each sample was determined from the measured density and assuming absence of any

porosity. To calculate the alumina volume fraction, the density of alumina was taken as  $3.90 \text{ Mg.m}^{-3}$  [20], while the density of AlSi12 was determined in the current study to be  $2.66 \text{ Mg.m}^{-3}$ . The average density and the alumina volume fraction of each composite type, along with their standard deviations are given in Table 1.

## 2.2 Ultrasonic analysis

Ultrasonic phase spectroscopy (UPS) was used to determine the longitudinal and the shear wave velocities along all three directions in each sample. Only a brief overview of the method is given here and for thorough description we refer to Ref. [17]. In this technique continuous, sinusoidal, elastic waves are used for the measurements and the phase shift occurring as the wave passes through the specimen is recorded as a function of frequency. The measurements were accomplished using an electronic network analyser (Advantest, model R3754A) and two identical broadband ultrasonic longitudinal wave (Panametrics, model V122 with nominal central frequency 7.5 MHz and diameter 9.5 mm) and shear wave (Panametrics, model V155 with nominal central frequency 5 MHz and diameter 12.7 mm) transducers. These transducers were attached to the opposite sides of the rectangular parallelepiped samples with the help of a water soluble couplant. The phase and the amplitude spectra were recorded in the frequency range from 10 kHz to 15 MHz for longitudinal wave measurements and in the range from 10 kHz to 10 MHz for shear wave measurements, respectively. In a non-dispersive medium, the velocity of the propagating wave can be determined following the relation:

$$V = -2\pi L/m \quad (1)$$

where  $L$  is the sample length along the propagating direction and  $m$  is the slope of the straight line fitted to the phase-frequency spectrum. Once the wave velocity is measured, the respective elastic constants can be determined using the relation:

$$C = \rho V^2 \quad (2)$$

where  $\rho$  is the sample density. In case of the measurements with longitudinal waves, the direction of vibration of the particles of the medium is parallel to the direction of wave motion. Hence, for each longitudinal elastic constant  $C_{ii}$  ( $i=1-3$ ), only the wave velocity along the direction of wave propagation  $V_i$  needs to be measured. For shear waves, the direction of vibration of the particles of the medium is orthogonal to the direction of wave motion. Following Rose [21], each shear elastic constant  $C_{ii}$  ( $i=4-6$ ) can be determined by either of the two wave velocities  $V_{xy}$  (where the first suffix is the direction of wave motion and the second suffix is the direction of particle motion). The velocities corresponding to  $C_{44}$ ,  $C_{55}$  and  $C_{66}$  are  $V_{23}$  and  $V_{32}$ ,  $V_{13}$  and  $V_{31}$ , and  $V_{12}$  and  $V_{21}$ , respectively. Therefore, 9 different wave velocities (3 longitudinal wave velocities along 3 principal directions and 2 shear wave velocities for each of the 3 shear elastic constants) were measured for each sample.

To determine the three non-diagonal components of the stiffness matrix ( $C_{12}$ ,  $C_{13}$ ,  $C_{23}$ ), 45° cuts were made at the opposite corners along the edges of one sample of Type A.

Detail description of the sample geometry and the necessary methodology to determine these three constants are described in Ref. [22].

### 3. Results

Figure 2 shows the optical micrographs of the composite material with alumina content of approximately 35 vol%. Micrographs ‘a’ and ‘b’ correspond to the faces parallel to the direction of preform pressing and squeeze-casting. Micrograph ‘c’ shows the structure of the face perpendicular to the direction of preform pressing and squeeze-casting. All three images were taken from the same sample. In images ‘a’ and ‘b’, the direction of preform pressing and squeeze-casting is oriented along the vertical



direction. Texture caused by preform pressing is clearly visible in the shown images. Distribution of the phases in micrograph 'c' is mostly random, while the phases are mostly aligned along the direction transverse to the press direction in micrographs 'a' and 'b'. In these optical micrographs the darker phase is alumina while the brighter phase is AlSi12 alloy. The optical micrograph shows clusters rich with alumina while the regions away from the clusters are rich with the matrix alloy AlSi12. Figure 3 shows a SEM image of the composite material. In this image the regions marked as AlSi12 are the locations of the original cellulose fibres shown in Figure 1. Regions of metallic alloy visible within the alumina rich regions are the locations of the micropores within the sintered preform. Both these original micropores in the ceramic walls and the traces of the cellulose fibres between the ceramic walls are filled by the liquid metal during infiltration and give rise to the interpenetrating structure of the composite. Small amount of residual porosity remains in the MMC after infiltration.

Figure 4 shows typical phase-frequency spectra obtained from UPS using longitudinal and shear wave transducers. In the current study it was observed that in almost all samples, the phase spectra obtained with both longitudinal and shear transducers were mostly linear with a constant slope over a significant range of frequency. This indicates that the measured wave velocities were independent of frequency (absence of dispersion). The spectra shown in Figure 4 were obtained for wave propagation in the same sample along the same direction. Following Eqn. 1, as the shear wave propagation velocity in the material is significantly less than the longitudinal wave propagation velocity, the slope of the shear wave spectrum in Figure 4 is significantly higher.

In Table 1 the measured longitudinal and shear wave velocities, averaged over all the samples within a composite type are listed. For each sample, the three longitudinal and

the three shear elastic constants were determined following Eqn. 2. Two shear wave velocities were determined for each shear elastic constant and the average of these two velocities was then used to determine the actual shear elastic constant. In almost all samples and for each shear elastic constant, the two shear wave velocities were almost equal within experimental uncertainties, suggesting that the composite had orthotropic or higher symmetry [21]. Figure 5 and Figure 6 show the variation of the longitudinal and the shear elastic constants with ceramic content in all composite samples. In spite of the considerable scatter, these plots show that in general the elastic constants increase with increasing ceramic content. The scatter in experimental data points may arise due to structural heterogeneities and defects such as residual porosity, imperfect bonding between the phases, particle agglomeration, varying degrees of ceramic wall interconnections etc. The scatter in the data points for shear elastic constants is more than the data points for longitudinal elastic constants. This may be attributed to the enhanced experimental difficulty associated with shear wave measurements due to the additional constraint of aligning the sample properly along the direction of vibration of the particles of the medium. In individual samples,  $C_{11}$  was found to be marginally less than  $C_{22}$  and  $C_{33}$ , which themselves were almost equal to each other. The values of the average longitudinal-wave velocities in each composite type listed in Table 1 clearly show this. No such trend was observed in case of shear elastic constants, and Table 1 shows that the three average shear elastic constants in each composite type were equal to each other within experimental uncertainties.

Figure 7 and Figure 8 show the correlation of the experimentally determined average elastic constants in each composite type with the theoretical micromechanical models. As  $C_{22}$  and  $C_{33}$  were almost equal, their average is plotted in Figure 7. Average of the

three shear elastic constants in each composite type is plotted in Figure 8. Error bars in these plots correspond to standard deviation in volume fraction and the respective elastic constant within all samples of a particular composite type. Thorough description of the used micromechanical models is beyond the scope of this work and they can be found in the following Refs. ([14] for HS bounds, [15] for R bounds, [23] for F model, [24] for DEM model and [25] for SC model). Among the models, the HS bounds, the DEM and the SC models predict the bulk modulus and the shear modulus of a linear elastic isotropic spherical particle reinforced composite. The F model is developed for multiphase interpenetrating composites while the R model considers isolated cubic inclusions in a continuous matrix. These two models give expressions for the Young's modulus and the shear modulus of the composite. Isotropy has been assumed to calculate the model predictions in the present work. The actual mathematical formulations for the model calculations can be found in Refs. [11, 26]. To calculate the model predictions, the Young's modulus and the Poisson's ratio of alumina were taken as 390 GPa and 0.24, respectively [27]. The Young's modulus and the Poisson's ratio of AlSi12 were measured during the present study and they were 82 GPa and 0.326, respectively. Other elastic constants were determined from these values assuming isotropy. For isotropic behavior, the models predicted the technical constants of the composite (Young's modulus ( $E$ ), bulk modulus ( $K$ ), shear modulus ( $G$ ) and Poisson's ratio ( $\nu$ )). The expression for the shear modulus is identical to the expression for the shear elastic constant. The expression for the longitudinal elastic constant of the composite was determined using the relation:

$$C_{ii(i=1-3)} = \frac{(3K + 2G)(6K + 2G)}{6(3K + G)} \quad (3)$$

9 elastic constants of one sample of Composite Type A were measured to determine the complete stiffness matrix assuming orthotropic symmetry. The results are shown in Table 2. The compliance matrix for this sample was subsequently determined by matrix inversion. The technical constants (Young's moduli, shear moduli and Poisson's ratios) were finally determined using the standard relation for orthotropic symmetry [22]. These technical constants are listed in Table 3.

#### 4. Discussions

Ceramic content in each sample was determined assuming no porosity. However, Figure 3 shows presence of small amount of residual porosity in the composite. As shown by Weidenmann et al. [28], presence of porosity may lead to a deviation in the calculated ceramic volume content. They attributed the residual porosity in the MMC to stronger shrinkage of the matrix than the ceramic particles during cooling from the solidification temperature and to incomplete infiltration due to insufficient infiltration pressure. As 100 MPa pressure was applied during squeeze-casting to fabricate the MMC in the present study, the second term in their calculation has been neglected here. Following the modified expression in Equation 4 in Ref. [28], the amount of residual porosity in the studied MMC samples was estimated to lie in the range of 0.1-0.5 vol%. This may shift the calculated alumina content in the composite samples to a value approximately 0.2-1.1 vol% higher than under the assumption with no porosity.

Due to the random structure of the composite, the material is expected to behave isotropically. The observed marginally reduced stiffness along 1 direction may result from either powder pressing or squeeze-casting. UPS on uninfiltreated ceramic preforms showed that the longitudinal wave velocity along 1 direction was about 30-50% less than the longitudinal wave velocities along directions 2 and 3, which were similar.

Therefore, it can be concluded that powder pressing prior to sintering of the preform caused the observed marginal reduction in  $C_{11}$ . Table 1 however shows that in the composite the velocity  $V_1$  is only 2-5% less than the velocities  $V_2$  and  $V_3$ . This suggests that as a result of infiltration, the composite exhibits much less pronounced anisotropy in comparison to the uninfiltreated preform. The continuous compliant AlSi12 phase increases the stiffness of the porous preform along all directions; however, this increase is most pronounced along the most compliant direction of the preform. Figure 7 and Figure 8 show that the measured average longitudinal and shear elastic constants lie within the HS upper and lower bounds. The predictions of R bounds lie within the HS bounds. The extent to which the individual theoretical models predict the experimentally determined elastic constant may be expressed as [11]:

$$\%D = \frac{C_{ii}^{model} - C_{ii}^{experimental}}{C_{ii}^{model}} \times 100\% \quad (4)$$

This quantity is listed in Table 4 for individual models. The table clearly shows that when all the elastic constants are considered, the predictions from the SC model are most accurate, the maximum deviation being approximately 6% for the shear elastic constant in Composite Type D. The predictions from DEM model lie between the predictions from SC model and HS lower. It predicts the composite behavior most accurately when the elastic constant  $C_{11}$  is considered. In case of shear elastic constants, the accuracy of the DEM model is close to the SC model, however, it is rather inaccurate when elastic constants  $C_{22}$  and  $C_{33}$  are considered. Although F model is specially designed for interpenetrating composites, the table shows that apart from the elastic constants  $C_{22}$  and  $C_{33}$ , its accuracy for the studied material is significantly less in comparison to SC and DEM models. Table 4 typically shows that with increasing alumina content, the  $\%D$  progressively becomes less positive for HS upper and it

becomes more negative for HS lower. This trend is similar for all elastic constants. It suggests that as the alumina content of the composite increases, the elastic constants of the composite are predicted more accurately by the HS upper bound than the HS lower bound. This can be explained by the fact that the HS model considers an isolated spherical phase coated with a concentric matrix phase. Diameters of the spheres depend upon the volume fraction of the individual phases. As discussed by Torquato et al. [29], in case of the lower bound, the compliant matrix is the outer phase and the arrangement of the stiffer phase can be regarded to be most disconnected. In case of the upper bound, the compliant phase is the inner phase and the arrangement of the stiffer phase can be regarded to be most connected. In the interpenetrating composite under study, both phases are continuous, and hence the simplistic HS model is not ideally suited to predict the elastic behavior of such composites. In spite of this limitation, comparison of the experimental results of the current study with theoretical HS bounds provides valuable insight because of two reasons. Firstly, in a previous study, Young's modulus of an interpenetrating MMC over a range of ceramic content was found to lie close to the HS lower bound [30]. Secondly, the HS bounds are much more rigorous than the simple rule of mixtures models, which are considered to be strict upper and lower bounds. However, the proximity of the experimentally determined elastic constants at high ceramic content to the HS upper bound contradicts the results obtained by Moon et al. [11]. For Al-Al<sub>2</sub>O<sub>3</sub> interpenetrating composites they found that even at ceramic content in the range of 73-97 vol% the Young's modulus was closer to the HS lower bound. Table 2 - Table 3 shows that although 9 elastic constants of one sample were determined assuming orthotropic symmetry of the composite, the elastic constants were similar along directions 2 and 3. Hence, the composite behaves more like a material

with transverse isotropic symmetry with respect to 1 direction. Determination of the complete stiffness matrix also allows predicting the off-axis behavior of the material. Following Jones [31], the Young's moduli and Poisson's ratios of an orthotropic material must satisfy the following relations:

$$\frac{E_i}{\nu_{ij}} = \frac{E_j}{\nu_{ji}} \quad i, j = 1, 2, 3 \quad (5)$$

$$\text{and } \Delta = 1 - \nu_{12}\nu_{21} - \nu_{23}\nu_{32} - \nu_{31}\nu_{13} - 2\nu_{21}\nu_{32}\nu_{13} > 0 \quad (6)$$

In the present study  $\Delta$  was equal to 0.757, while Table 5 shows that Eqn. 5 is also valid within slight uncertainties. These suggest that the technical constants determined in the current study are accurate and theoretically valid.

## 5. Conclusions

In depth elastic analysis of an interpenetrating AlSi12-Al<sub>2</sub>O<sub>3</sub> composite was carried out in this study using ultrasound phase spectroscopy. Following conclusions can be drawn:

- Preform pressing prior to sintering marginally reduces the longitudinal elastic constant of the composite along the press direction. The composite behaves like a transverse isotropic material with respect to this direction. The compliant metallic alloy functions as a stiff backbone to stiffen the composite along the press direction in comparison to uninfiltated preform.
- Results show that the experimentally determined longitudinal and shear elastic constants lie within the Hashin-Shtrikman bounds. At low ceramic contents the values are closer to the Hashin-Shtrikman lower bound, while at high ceramic content the Hashin-Shtrikman upper bound is more accurate.

- Among the micromechanical models considered, self consistent model predicted the average elastic constants of the composite over the complete ceramic vol% range most accurately.
- Complete stiffness matrix of one sample was determined and the technical constants were determined there from.

### Figure captions

**Figure 1:** SEM image of the ceramic preform with nominal ceramic content of 41 vol%. The micrograph shows the face perpendicular to the direction of preform pressing and squeeze-casting. Trace of a cellulose fibre is shown by the arrow.

**Figure 2:** Optical micrographs of the composite with alumina volume fraction  $V_f = 0.35$ . Micrographs (a-b) show the structure of the faces parallel to the direction of preform pressing and squeeze-casting, which are oriented along vertical direction in the images shown. Micrograph (c) shows the structure of the face perpendicular to the direction of squeeze-casting and preform pressing.

**Figure 3:** SEM image of the composite material.

**Figure 4:** Typical phase-frequency spectra obtained from ultrasonic analysis using longitudinal and shear-wave transducers.

**Figure 5:** Plot showing the variation of the longitudinal elastic constants with ceramic content

**Figure 6:** Plot showing the variation of the shear elastic constants with ceramic content

**Figure 7:** Plot showing the correlation between the experimentally determined average longitudinal elastic constant of each composite type with theoretical models. Average of  $C_{22}$  and  $C_{33}$  is plotted.



**Figure 8:** Plot showing the correlation between the experimentally determined average shear elastic constant of each composite type with theoretical models. Experimental values show the average between  $C_{44}$ ,  $C_{55}$  and  $C_{66}$ .

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Table 1: Densities, ceramic contents and longitudinal and shear wave velocities of each composite type. Values for each type are averaged over the number of samples in each type mentioned in the table.

Type	Density (Mg.m <sup>-3</sup> )	Sample No.	Ceramic content (vol. fr.)	$V_{11}$ (m.s <sup>-1</sup> )	$V_{22}$ (m.s <sup>-1</sup> )	$V_{33}$ (m.s <sup>-1</sup> )	$V_{23}$ (m.s <sup>-1</sup> )	$V_{32}$ (m.s <sup>-1</sup> )	$V_{13}$ (m.s <sup>-1</sup> )	$V_{31}$ (m.s <sup>-1</sup> )	$V_{12}$ (m.s <sup>-1</sup> )	$V_{21}$ (m.s <sup>-1</sup> )
A	3.10	20	0.35	7583	7789	7776	4106	4079	4094	4146	4006	4060
	±0.06		±0.05	±208	±294	±263	±327	±293	±143	±222	±175	±242
B	3.22	13	0.45	8006	8186	8239	4483	4467	4375	4417	4342	4421
	±0.06		±0.05	±126	±113	±85	±166	±153	±197	±176	±195	±188
C	3.28	13	0.50	8199	8537	8507	4732	4645	4548	4669	4408	4627
	±0.09		±0.08	±125	±190	±133	±308	±348	±81	±107	±230	±287
D	3.31	20	0.53	8332	8513	8583	4588	4593	4575	4589	4589	4629
	±0.06		±0.05	±121	±81	±137	±244	±197	±276	±298	±112	±189
E	3.39	20	0.59	8692	8956	9008	5040	4946	4790	4866	4901	4960
	±0.07		±0.06	±190	±183	±162	±247	±228	±275	±320	±147	±144

Table 2: Complete stiffness matrix of one sample of Composite Type A.

$C_{11}$ (GPa)	$C_{22}$ (GPa)	$C_{33}$ (GPa)	$C_{44}$ (GPa)	$C_{55}$ (GPa)	$C_{66}$ (GPa)	$C_{23}$ (GPa)	$C_{13}$ (GPa)	$C_{12}$ (GPa)
176	190	189	62	54	49	61	67	68

Table 3: Technical constants of one sample of Composite Type A

E1 (GPa)	E2 (GPa)	E3 (GPa)	G23 (GPa)	G13 (GPa)	G12 (GPa)	$\nu_{21}$	$\nu_{31}$	$\nu_{12}$	$\nu_{32}$	$\nu_{13}$	$\nu_{23}$
140	156	156	62	54	49	0.305	0.298	0.272	0.214	0.267	0.215

Table 4: Table showing the deviation between the experimentally determined elastic constants and model predictions

Type	Expt.	HS <sub>U</sub>	%D	HS <sub>L</sub>	%D	R <sub>U</sub>	%D	R <sub>L</sub>	%D	F	%D	SC	%D	DEM	%D
<i>C<sub>11</sub></i>															
A	178±10	204.5	12.9	177.1	-0.5	200.3	11.1	182.3	2.4	192.8	7.7	185.1	3.8	180.1	1.1
B	206±7	233.7	11.9	199.9	-3.1	226.0	8.9	208.8	1.3	219.7	6.3	213.7	3.6	205.0	-0.5
C	220±10	249.4	11.8	212.8	-3.4	239.9	8.3	223.7	1.6	234.4	6.1	230.0	4.3	219.3	-0.3
D	232±8	259.1	10.5	221.1	-4.9	248.7	6.7	233.2	0.5	243.5	4.7	240.4	3.5	228.5	-1.5
E	256±13	279.5	8.4	239.2	-7.0	267.3	4.2	253.5	-1.0	262.7	2.6	262.8	2.6	248.3	-3.1
Average of <i>C<sub>22</sub></i> & <i>C<sub>33</sub></i>															
A	188±13	204.5	8.0	177.1	-6.1	200.3	6.2	182.3	-3.1	192.8	2.5	185.1	-1.6	180.1	-4.4
B	218±7	233.7	6.9	199.9	-8.8	226.0	3.8	208.8	-4.2	219.7	1.0	213.7	-1.8	205.0	-6.1
C	238±12	249.4	4.6	212.8	-11.9	239.9	0.8	223.7	-6.4	234.4	-1.5	230.0	-3.5	219.3	-8.5
D	244±9	259.1	5.8	221.1	-10.4	248.7	1.9	233.2	-4.7	243.5	-0.2	240.4	-1.5	228.5	-6.8
E	274±11	279.5	2.2	239.2	-14.3	267.3	-2.3	253.5	-7.9	262.7	-4.1	262.8	-4.1	248.3	-10.1
Average of <i>C<sub>44</sub></i> , <i>C<sub>55</sub></i> & <i>C<sub>66</sub></i>															
A	53±5	62.0	15.0	50.6	-4.1	58.6	10.1	51.0	-3.4	58.0	9.2	54.0	2.5	51.9	-1.5
B	63±5	72.8	13.4	58.6	-7.5	67.3	6.5	60.0	-5.0	67.7	6.9	64.6	2.4	60.9	-3.4
C	70±7	78.6	11.3	63.2	-10.3	72.2	3.5	65.2	-6.9	72.9	4.4	70.6	1.3	66.1	-5.4
D	70±6	82.2	14.4	66.2	-6.3	75.3	6.5	68.6	-2.6	76.2	7.7	74.5	5.6	69.4	-1.3
E	82±8	89.7	8.3	72.7	-13.3	81.9	-0.6	75.9	-8.5	83.1	0.9	82.9	0.7	76.8	-7.3

Table 5: Table showing the accuracy of the measured technical constants

$E_1/\nu_{12}$ (GPa)	$E_2/\nu_{21}$ (GPa)	$E_1/\nu_{13}$ (GPa)	$E_3/\nu_{31}$ (GPa)	$E_2/\nu_{23}$ (GPa)	$E_3/\nu_{32}$ (GPa)
514.71	511.47	524.34	523.49	725.58	728.97

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