

Technical Report ARMET-TR-12039

Note for Sandia R&A:

This is an Army document. They (ARDEC) have requested permission to use Sandia-provided information. The purpose of this R&A is to review the Sandia material. The Sandia material is found in Figures 3, 4, 5, 6 and 7. Steve Goods, the Sandian who performed the testing and created the figures, has since retired.

Ken Black

Measurement of Elastic Modulus of Alumina and Barium Strontium Titanate wafers produced by Tape casting method

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Measurement of Elastic Modulus of Alumina and Barium Strontium Titanate wafers produced by Tape casting method

During a recent (2007-2011) program, there existed a need for a modeling and simulation study to determine the stresses inside a device fabricated using thin wafers of BST (Barium Strontium Titanate) and Alumina ceramic during a launch of a system. Sandia National Laboratory, California was given the task of Modeling and simulation. For such a simulation the values of young's Modulus for Alumina and Barium Strontium Titanate (BST) were essential requirements.

Literature search

Mechanical Properties of BST are very scarce and no data dealing with mechanical properties was found. However, since Barium and Strontium make complete solid solution, we presume therefore, that the properties of Barium Titanate (BT) will be similar to those of BST. Therefore, we searched the literature for values of BT. Pohanka & Smith (Ref. 1) listed Young's modulus for BT in J/m sq at 4.0 at 25 deg C and 2.5 at 150deg C. Another work by Scholz, Schneider et al (Ref 2) lists Youngs' Modulus for BT at 181 GPa. A A Wereszczak , L Riester et al (Ref 3) find BT Multilayer Capacitors Youngs' Modulus 193 GPa . It seems that there is a close agreement between these three studies.

Mechanical Properties of Alumina have been determined by several researchers and are listed in the literature. A value of Young's Modulus 390 GPa is listed in Fundamentals of Ceramics (Ref.4). "Mechanical Properties of Ceramics lists the value of Young's Modulus for Alumina at 297 GPa. Another study of modulus of Alumina as a function of its density lists the values between 260 to 410 GPa depending on its density (Ref.5) There is quite large variation between these two values. It is to be noted that sample preparation, Impurity levels and sample size may be the reason for such variations.

It is worth mentioning here that most of samples are prepared by ball milling powders, Calcining, crushing, milling, Isostating pressing and forming the sintered products which then are used to mechanically cut and polished to obtain the samples for measurements.

Because there was big variation in the published data and since we had samples of both BST and Alumina readily available to us, we therefore decided to perform the measurements of elastic modulus to confirm and verify the values we found in the published literature. These measurements done on actual materials will also provide us the most reliable data for our purpose

Measurements of Elastic Modulus

Two kinds of samples were used for BST and one kind for Alumina. For BST the first samples were obtained from ARL and were produced by standard method using powders of Barium and Strontium carbonates .The steps followed are summarized below:

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- Mix stoichiometric amounts of barium carbonate, strontium carbonate, and titanium (iv) oxide together
- Powder pressed into a 32mm dia. Pellet using a uniaxial press.
- Pellet heated in air for 10 hrs to 1100C
- Pellet reground into a fine powder with a mortar and pestle
- Powder isostatically pressed into a 32mm dia. Pellet with a pressure of 45-50kpsi
- Pellet heated to 1500C in air for 25 hrs

This resulted in sintered pellets which were sawed using fine diamond coated saws to prepare wafers of 25 X 8 X 0.5 mm.

The other samples of BST wafers as well as Alumina wafers were produced by a technique known as Tape casting. Tape casting is widely used in the production of paper, plastic, paint as well as in production of thin ceramics sheets. The basic principles of casting are described on flow chart depicted in Fig.1. The ceramic powders are added in the ball mill along with deflocculants/dispersants. The mixture is then ball milled till the proper dispersion of solid particles in the solvent is achieved. Next step consists in mixing the plasticizers and binders into the slurry produced in the ball mill. De-airing, usually by vacuum, is necessary to remove the entrapped air. This is accomplished in a simple device such as desiccators. For the most critical applications, such as a very thin tapes or very smooth thin –film substrates, a slip filtration step is usually needed just before casting step. The details of tape casting can be found in Ref.6 as well as in a technical report Ref.7

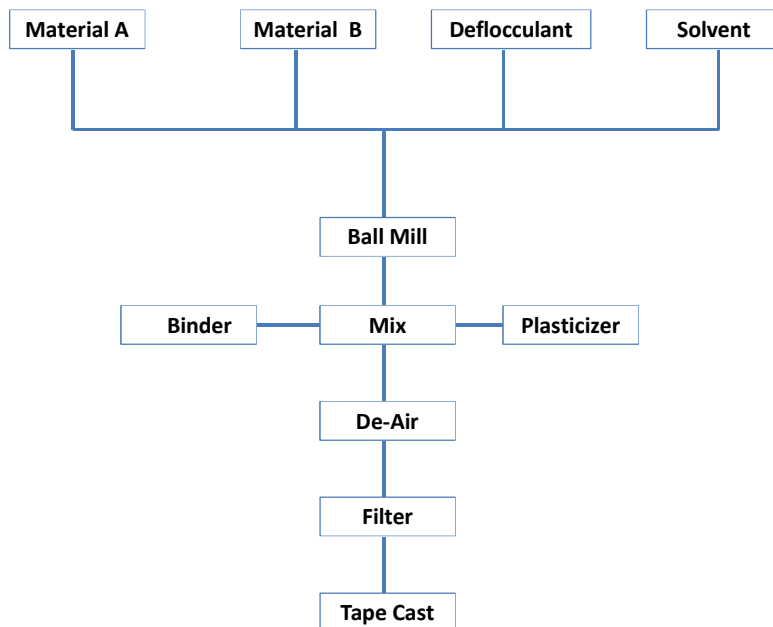


Fig.1. Schematic of tape casting method, Ref.6

The finished sintered wafers of BST and Alumina are shown in Fig.2

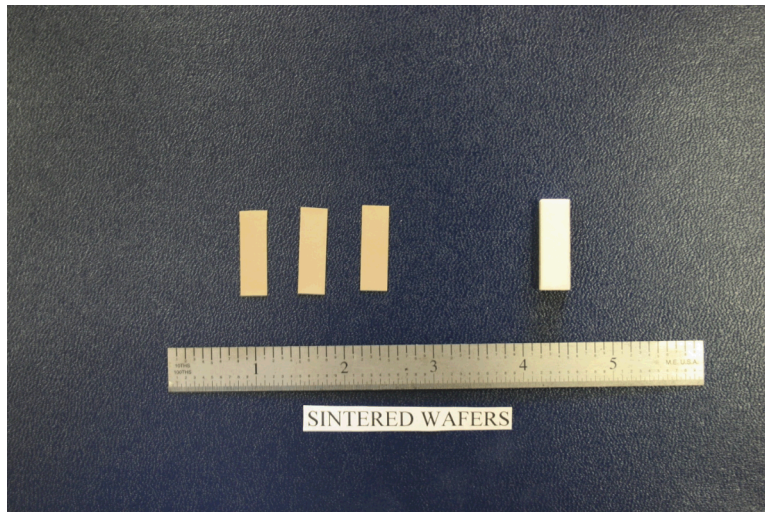


Fig. 2 Wafers of Tape Cast BST (dark color) and Alumina (light color)

Measurements of Elastic Modulus

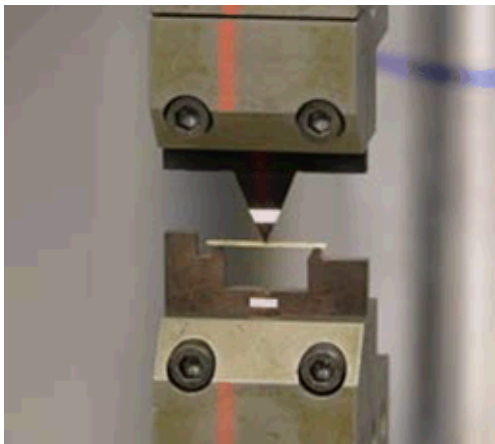
The most common method a fixture testing, so called three-point configuration testing was used to determine the elastic modulus. The measurements were made at The Sandia National Lab. Fig. 3 shows the experimental set up and the magnified view of the three point test. The formula used to calculate the modulus is indicated at the right hand corner of the Fig. 3. The dimensions of the wafers are explained in the diagram.

The details of the measurements and the results are shown in the Fig. 4 and summarized in Fig. 5. These measurements were made on the samples produced at ARL and by the method used as described above (pelletizing method). An average value of the elastic modulus of around 105 GPa was calculated using several samples as described on the figure5.

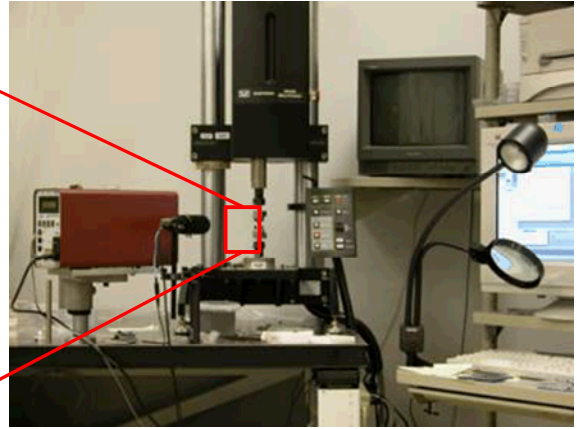
The measurements made for the BST wafers produced by Tape Casting method are shown in the Fig. 6. Samples of two different wafer thicknesses (0.5 and 0.3 mm) were used and details of the measurements and the results are shown in the Fig.6. Results of many tests using the both thicknesses show very consistent values of 161 GPa.

The values of modulus for the tape cast material is significantly higher than the ones made by standard powder pressing, Calcining, grinding and pelletizing method. The wafers made by this method were less uniform as compared to tape cast material. It is also to be noted that the study of the microstructure had shown that there was more porosity in samples made at ARL with standard method. On the other hand the wafers made by tape casting were more uniform in thickness and of more uniformly distributed porosity of smaller size. The value of 161 GPa is although different from the value for BT but are not too far from those 161 Vs 180-190 GPa. It is to be noted that these values are for different materials and such variation is expected.

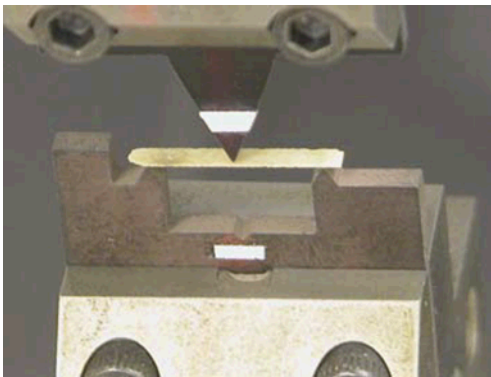
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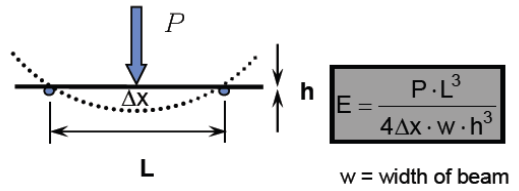
(a)



(b)



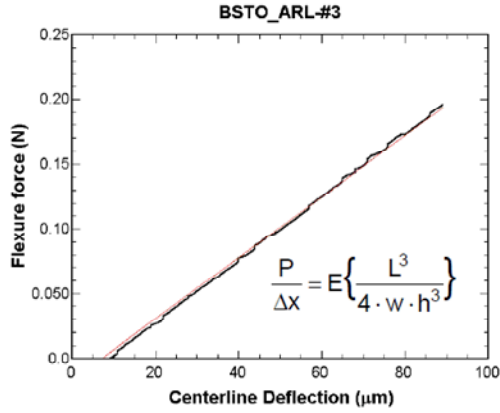
(c)



Formula used for calculations

Figure 3. The three point test equipment (a), (b) ,(C) and formula used for calculations

Modulus measurement- multiple tests on single beam



	Modulus (GPa)	Max Load (mN)	Max Stress (MPa)	Max CL disp (µm)
1	-	124.72	37.93	49.5
2	104.45	197.67	60.12	89.8
3	104.03	195.95	59.6	89.4
4	104.65	195.48	59.46	89.4
5	104.58	197.68	60.12	90.4
6	104.76	197.03	59.93	89.8
7	105.64	194.45	59.14	89.5
8	103.98	196.55	59.78	90.6
9	104.02	196.3	59.7	90.4
*10	104.43	229.3	*70.01	105.22
Mean	104.5	169.63	58.58	105.2
Standard Deviation	0.51	63.58	7.95	63.9
Minimum	103.98	0.43	37.93	49.5
Maximum	105.64	197.68	70.01	283.5
Range	1.65	197.25	32.08	233.9

- For any beam, mod. measurements are consistent
- 104-105 GPa modulus has been measured in 1st beam tested.
- Beam flexed to failure:
 - Failure stress = 70 MPa

*Test 10: beam taken to failure

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Figure 4. Multiple tests measurements on a single beam of wafer from ARL material of BST

Summary

- Modulus differences between samples are likely the result of:
- Dimensional variability in machining
 - Local porosity and defects

	Sample #		
	2	3	4*
Avg Mod (GPa)	99.3	104.5	113.9
Std Dev	1.2	0.3	0.8
Max	100.3	104.8	114.6
Min	97.6	104.0	112.7

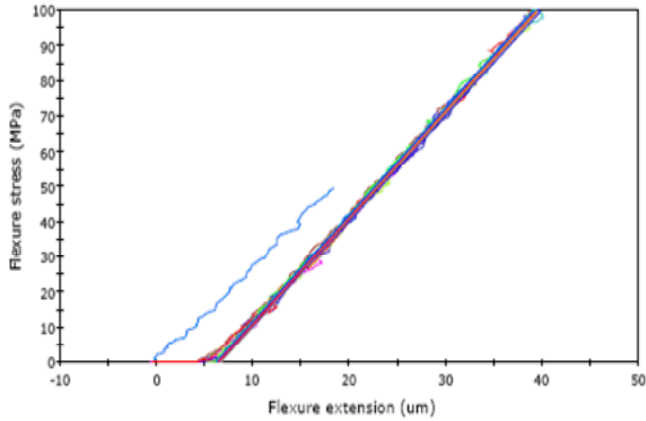
* 4 measurements only

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Figure5. Summary of measurements performed on 3 different samples of wafers from ARL

Results –Tape Cast BST Wafers



- Modulus
 - 0.5 mm thick (.47)
 - 161.4 +/- 0.6 GPa
 - 165.5 +/- 1.8
 - 0.3 mm beams (.28)
 - 163.6 +/- 1.1 GPa
 - 157.9 +/- 0.9
- Failure flexure stress
 - two beams @0.3 mm
 - 98/115 MPa

	Specimen label	Flex Modulus (GPa)	Maximum Load (mN)	Maximum Stress (MPa)	Max CI disp (um)
1	BST Thick #1-1	-----	1174.26	49.57	18.42
2	BST Thick #1-2	-----	1880.51	79.38	33.18
3	BST Thick #1-3	161.16	2368.94	100.00	39.38
4	BST Thick #1-4	161.62	2366.71	99.90	39.92
5	BST Thick #1-5	161.37	2365.33	99.84	39.71
6	BST Thick #1-6	161.54	2364.46	99.81	39.55
7	BST Thick #1-7	162.21	2363.63	99.77	39.22
8	BST Thick #1-8	160.26	2362.49	99.72	39.54
9	BST Thick #1-9	161.88	2361.32	99.67	38.67
Mean		161.43	2178.63	91.96	36.29
Standard Deviation		0.61933	409.27296	17.27603	7.00680
Minimum		160.26	1174.26	49.57	18.42
Maximum		162.21	2368.94	100.00	39.71
Range		1.95	1194.69	50.43	21.30

Fig. 6 Summary of measurements made on BST wafers produced by Tape Casting Method

Measurements done on Al₂O₃ Wafers

- Density of Al₂O₃ (99.9%) ≈ 3.96 g/cm³
 - Estimated density of coupon: 3.62 g/cm³
 - Relative density = 91.4%
 - Modulus @ 100% → 400 GPa
 - Modulus @ 91% → < 300 GPa

➤ 0.5 mm thick beams

- 245.2 +/- 1.4 GPa
- 249.6 +/- 3.12 GPa
- 253.2 +/- 3.7 GPa

➤ 1.0 mm beams: probably too stiff for good measurements

- Generally in the 230 – 250 GPa range

Grade	Al ₂ O ₃ /Porosity %	Young's modulus GPa
A1	≥ 99.6 / 0 - 2	410 - 380
A2	≥ 99.8 / < 1	405 - 380
A3	≥ 99.5 / < 1	400 - 398
A4	≥ 99.6 / 3 - 6	380 - 340
A5	≥ 99.0 / 1 - 5	380 - 340
A6	96.5-99.0 / 1 - 5	375 - 340
A7	94.5-96.5 / 1 - 5	370 - 300
A8	86.0-94.5 / 2 - 5	330 - 260
A9	80.0-86.0 / 3 - 6	330 - 260

*VTT manufacturing, 1996, ISBN 951-38-4987-2

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Figure 7. Modulus measurements on 0.5 mm wafers of Alumina Data on the table are from the literature.

Measurements of Elastic modulus for Alumina were done on the wafers produced by Tape casting method. Thickness of wafer was 0.5 mm. The thicker wafers measuring 1.0 mm were too thick for the measurements. The results are shown in the Fig 7. Three values are listed as 245.2, 249.6 and 253.2 yielding an average value of 249 GPa.

Towards the end of the program, we were planning to make measurements of yield strength, UTS and elongation of the BST and Alumina. Samples of these materials could be easily made into a dog bone tensile samples using tape casting method. However, due to lack of time we were not able to complete those measurements. It is recommended that when and if such measurements are required, the tape casting method will be most suited for producing the samples.

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Results and discussion

Elastic modulus measurements were made using three point configuration testing method was used. Samples of BST were made using regular powder pressing, sintering, pelletizing and sawing method (pelletizing method) as well as a Tape Casting technique. Samples of Alumina were made by Tape casting method.

For BST, a value of Elastic modulus of between 99 to 114 GPa was obtained for the ARL method and 161 GPa for the Tape casting BST material. This is result of wafers of BST made by Tape casting having less porosity that the other method. We also noted that of the variation between the measured values for the Tape Cast samples was very little as compared those from the pelletizing method. This is due to the fact that the wafers of Tape Cast method were found to be more uniform in thickness that those made by the other method.

For the Alumina, an average value of elastic modulus of 249 GPa was measured. This value is lower than reported in the literature and seems to be related to the relative density. The density of pure Alumina should be 3.96 gms/cc. However our coupon was tested to have a density of about 3.62 gms/cc indicating high value of porosity resulting in a relative density of 91.4 %. According the chart on fig. 7 the values obtained at this relative density correlates well with what is reported in the literature (see table in fig 7) . The high value of porosity may be an indication of insufficient sintering process, i.e. either less time or low sintering temperature.

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