CHARACTERIZATION OF COMPOSITES OF BERYLLIA AND LITHIUM-TITANATE PRODUCED BY SOL-GEL ROUTE

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Beryllium oxide (BeO) can be used as a constituent for lithium titanate (Li_2TiO_3) based composite material for neutron multiplication and breeding in International Thermonuclear Experimental Reactor (ITER) having tritium breeding ratio (TBR) more than 1.1. In the present investigation, we report the synthesis of various volume ratios of BeO-Li_2TiO_3 composites. The characterization showed that the coefficient of thermal expansion values for the composites are half of that of the Lithium titanate. The novel composites showed improved thermal conductivity compared to Li_2TiO_3. The thermal conductivity at 672 K which is close to the operating temperature is 47 W/mK which is ~17 times that of Li_2TiO_3. So there is a large improvement in thermal conductivity by use of this composite instead of use of Li_2TiO_3 and metallic Be separately. The impedance measurement showed that with the addition of BeO, there is reduction in electrical conductivity which is better to be used in the fusion reactor. The composites are found to have better modulus properties in comparison to Li_2TiO_3. So, from the present investigation, it could be concluded that it is possible to achieve tritium breeding ratio (TBR) more than 1.1 by optimizing the BeO and Li_2TiO_3 ratio with better properties.

INTRODUCTION

Thermal conductivity is an important physical property, which is required in modeling heat transfer through solids and structures. High thermal conductivity of the blanket is desired to maximize the heat extraction efficiency. Lithium titanate (Li_2TiO_3) is one of the best materials for tritium breeding, because of advantages like reasonable lithium atom density, low activation, excellent tritium release characteristics at low temperature, compatibility with structural material [2]. Beryllium is chosen as the neutron multiplier by many countries to increase the neutron population inside the reactor. Also, Beryllium oxide due to its high thermal conductivity, low neutron capture cross section, good strength, low neutron moderation and an appreciable fast neutron multiplication factor due to the (n, 2n) reaction [3] and good compatibility with SS316LN is a good choice for neutron multiplier material. By mixing beryllium oxide with lithium titanate in proper ratio, thermal conductivity of the blanket can be increased without any significant change in tritium generation and neutron multiplication. However, the experimental results or theoretical data on the properties of mixed BeO–Li_2TiO_3 composite breeder–multiplier system are scanty. So, In the present investigation, we present synthesis of single phase BeO–Li_2TiO_3 with volume ratios 80:20; 75:25; 65:35 and 55:45 with the aim of maintaining the tritium breeding ratio more than one and higher thermal conductivity. The volume ratio mentioned above for BeO and Li_2TiO_3 were taken after 3-D Monte Carlo simulation so that the tritium breeding ratio (TBR) should be greater than 1.1. as explained in appendix-1. As the BeO–Li_2TiO_3 compacts which will be used in the fusion reactors as tritium breeding material, it should have sufficient strength to bear the purge gas pressure of Helium at high temperature and thermal stresses generated in the reactors and it should not affect the huge magnets in the fusion reactor. The mechanical properties and electrical impedance properties of the compacts are therefore important. The paper gives the experimental results on the thermal, mechanical and electrical properties of BeO and Li_2TiO_3 composites.

EXPERIMENTAL

Preparation of Li_2TiO_3 by sol-gel reaction route

Li_2TiO_3 prepared by conventional internal gelation process using sol-gel method was used for preparation of BeO–Li_2TiO_3 composites. In the first step, high density Li_2TiO_3 was synthesized from water soluble titanium oxychloride ($TiOCl_2$) and lithium nitrate ($LiNO_3$) employing sol-gel technique [4]. $TiOCl_2$ was obtained from titanium tetrachloride, which is available commercially in high purity at a reasonable cost. The formation of $LiTiO_3$ compounds was confirmed by X-Ray diffraction (XRD) analysis of the product. The sample was chemically analyzed using analytical technique. The trace element analysis of Li_2TiO_3 powder was carried out by D.C. arc carrier distillation method. Table 1 gives the list of major impurities present in the $LiTiO_3$ sample.

Table 1. Major impurities in Li_2TiO_3.

Element	Na	Ca	Mg	Al	Si	Co	Bo	Cu	Mn	Ni	Pb
ppm	120	40	6	50	1500	1	1.2	30	1	1	2

Preparation of beryllium oxide (BeO) powder

The crude beryllium hydroxide of 98 % purity was dissolved in 9 N sulfuric acid to form beryllium sulphate solution. The solution was filtered and the filtrate was mixed with 40 % Sodium hydroxide solution to form a precipitate. On addition of excess sodium hydroxide this precipitate namely beryllate is found dissolved. The beryllate solution was subsequently hydrolyzed and filtered to obtain pure beryllium hydroxide which on calcination at 850°C for 2 hours in resistance-heated furnace yielded BeO powder. The chemical analysis of this beryllia powder was done by D.C. arc carrier distillation method. The analysis is given in the Table 2.

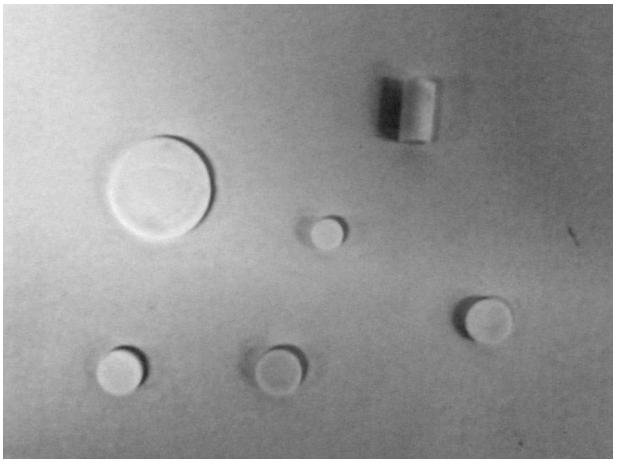


Figure 1. Samples of BeO–Li_2TiO_3.

Preparation of composite

Beryllia powder was mixed with Li_2TiO_3 made by solid-state reaction in ball mill. The mixed powder was pelletized in the die at 3500 kg/cm^2 pressure using a hydraulic press. These pellets were then sintered in resistance heated furnace at 1375°C for 2 h. Some of the samples made are shown in the Figure 1.

X-ray diffraction (XRD) and microscopy study of BeO–Li_2TiO_3 composites

Samples with different volume ratios of BeO to Li_2TiO_3 were characterized by powder X-ray Diffraction technique, recorded in the 2θ angle range 15° - 70°, on ITAL structure X-ray diffractometer using CuK_a radiation. For scanning electron microscopy and energy dispersive X-ray (SEM/EDX) analysis, the samples in the form of disc of ~ 12.7 mm mm diameter and 1.5 mm thickness were taken and coated with silver. For finding the phase distribution titanium and oxygen mapping was done in energy dispersive X-ray analyzer.

Density measurement

The room temperature bulk density was measured by Helium pycnometer. The samples were weighed in micro balance. The sample is taken in a sample holder of known volume and is pressurized. In the next step additional known volume is connected and pressure drop is noted. From the known volumes and pressures recorded, the unknown volume of the sample is determined. Twenty runs were taken for each sample and average of last three runs with minimum requested deviation, is used before evaluating the density.

Heat Capacity Measurement

Heat capacity measurements were carried out using a heat flux type differential scanning calorimeter (Model: Metler DSC-821). The temperature calibration of the calorimeter was carried out in the present study by the phase transition temperature of reference materials (Indium: T_{fus} = 429.748 K; Zinc T_{fus} = 692.6 K). Heat calibration of the calorimeter was carried out from the enthalpies of transition of the reference materials. For the determination of heat capacity, synthetic sapphire was used as the reference material.

Table 2. Major impurities in BeO.

Element	Al	B	Ca	Cd	Cr	Cu	Fe	Li	Mg	Mn	Na	Ni	Si	W	Zn
ppm	< 5	< 0.1	15	< 0.1	< 5	5	13	17	22	10	150	< 5	300	< 5	7

The specific heat was determined using DSC by "Comparison with Sapphire" method. The sample was kept in a platinum pan and heated in DSC from 30 to 700°C at 10°C/min in high purity Ar atmosphere (flow rate was 50 ml/min). The DSC curves of the sample and Sapphire were compared to determine the specific heat of the sample.

Coefficient of thermal expansion (CTE) measurement

The coefficient of thermal expansion (CTE) was measured under argon atmosphere up to the temperature of 1073 K. Due to difficulties in the fabrication of pellets with a dimension of 10 mm long and 5 mm dia. only two samples i.e. 65 BeO–35 Li_2TiO_3 and 75 BeO–25 Li_2TiO_3 compositions could be tested and the average value was used for the thermal diffusivity study for the correction of sample dimension at elevated temperature.

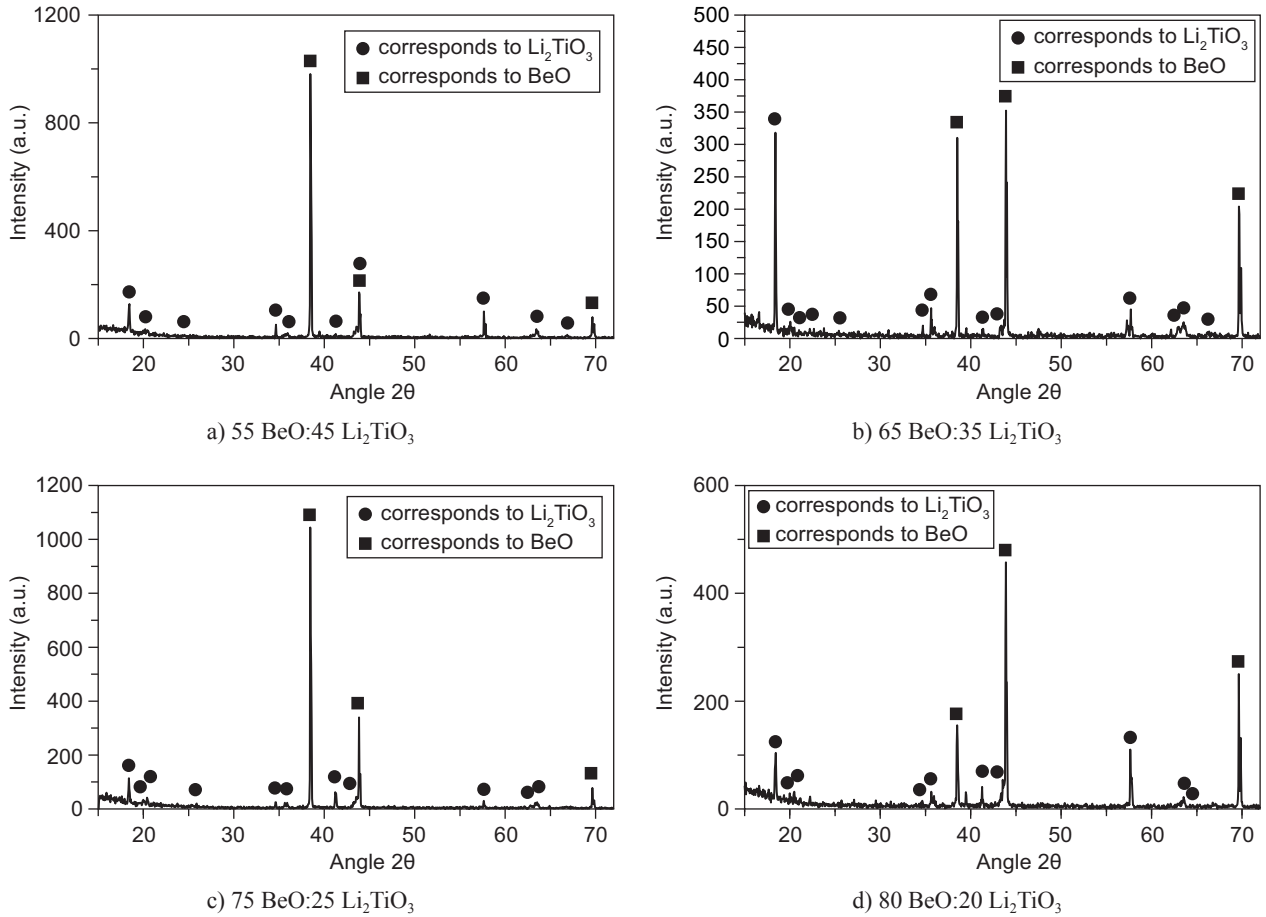
Thermal diffusivity measurement

The thermal diffusivity was measured by Laser flash technique in the temperature range of 320 K to 1221 K. For the thermal diffusivity measurement, the samples pellets were made in the form of circular discs with 10 mm dia. and 2 mm thickness. The top and bottom surface of the discs were coated with a thin layer of graphite. The error in the measured values of thermal diffusivity was within ± 3 %. For measuring thermal conductivity, the coefficient of thermal expansion was determined and a value of 10×10^{-6}/°C was used.

The thermal conductivity 'κ' for the samples were derived from the density, specific heat and thermal diffusivity values of the composite using the standard relationship $\kappa = a \cdot r \cdot Cp$, where α is the thermal diffusivity (W/cm·K), r is the bulk density (gm/cc) and Cp is the specific heat capacity (J/g·K).

Electrical impedance measurement

The electrical properties of composites were investigated by impedance spectroscopic technique. Impedance measurements were performed in a Solatron AC Frequency response Analyzer (Model 1260) in the frequency range from 10 MHz to 1 Hz. The sintered pellets was uniformly coated with a thin layer of platinum paste and annealed for 4 h in air at 673 K to remove organic binders. The platinum paste ensured proper electrical contact with the platinum electrode. The electrical measurements were carried out in the temperature range from 573 K to 873 K at an interval of 25 K. The temperature was controlled by a microprocessor.



a) 55 BeO:45 Li_2TiO_3 b) 65 BeO:35 Li_2TiO_3

c) 75 BeO:25 Li_2TiO_3 d) 80 BeO:20 Li_2TiO_3

Figure 2. XRD scans for a) 55 BeO:45 Li_2TiO_3 sample, b) 65 BeO:35 Li_2TiO_3, c) 75 BeO:25 Li_2TiO_3, d) 80 BeO:20 Li_2TiO_3

The sample temperature was measured by a K-type thermocouple placed very close to the sample with an accuracy of ± 1 K. At each temperature the sample was equilibrated for 20 min before recording the spectra. The diameter and thickness of the sintered pellets were measured.

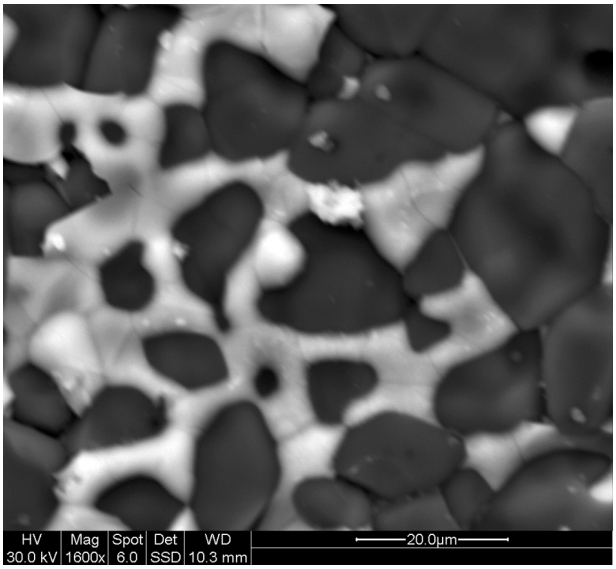
Mechanical testing

In order to assess the mechanical properties, cubic pellets of 12.5 mm length were prepared and tested in TIRA UTM machine at a strain rate of 10^{-4}. As the samples contain toxic BeO, special precautions were taken during testing. The modulus values were determined by ultrasonic testing using longitudinal and transverse velocity in the sample.

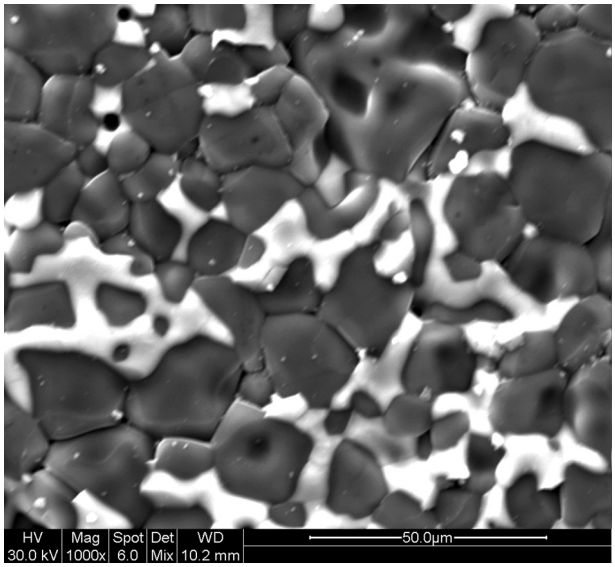
RESULTS AND DISCUSSION

X-ray diffraction (XRD) and microscopy study

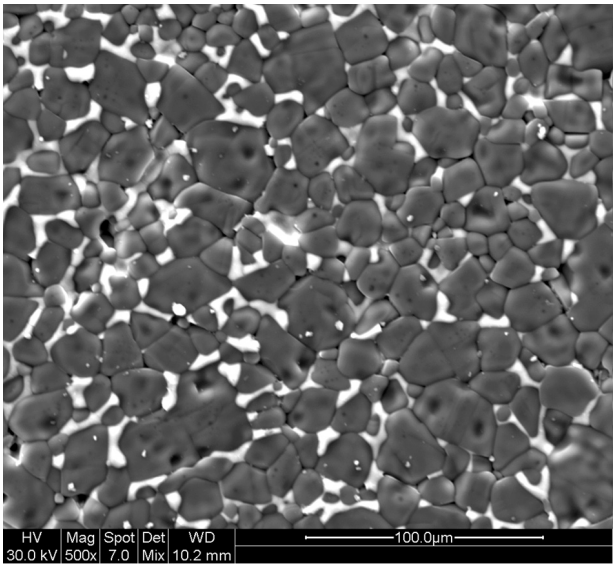
Figure 2 give the XRD patterns of BeO and Li_2TiO_3 composites volume ratios of 80:20; 75:25; 65:35 and 55:45. From the XRD plots it could be observed that all the samples are biphasic mixtures of hexagonal BeO and Monoclinic Li_2TiO_3 phases. Further it was observed that in these samples the most intense XRD lines corresponds to hexagonal BeO phase which has higher volume fraction compared to the Li_2TiO_3. Figure 3 gives the SEM images of 55 BeO:45 Li_2TiO_3, 65 BeO:35 Li_2TiO_3, 75 BeO:25 Li_2TiO_3 and 80 BeO:20 Li_2TiO_3 samples. In all these compositions two homogenously distributed distinct phases (black and white) were observed. The



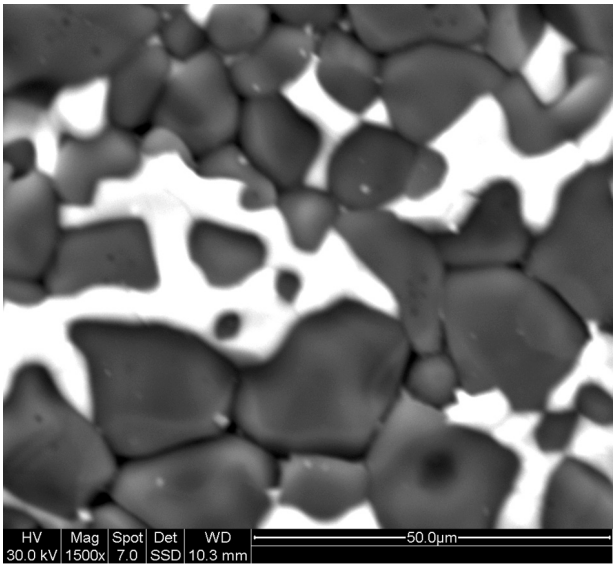
a) 55 BeO:45 Li_2TiO_3



b) 65 BeO:35 Li_2TiO_3



c) 75 BeO:25 Li_2TiO_3



d) 80 BeO:20 Li_2TiO_3

Figure 3. SEM images for a) 55 BeO:45 Li_2TiO_3 sample, b) 65 BeO:35 Li_2TiO_3, c) 75 BeO:25 Li_2TiO_3, d) 80 BeO:20 Li_2TiO_3.

EDX elemental mapping of sample indicates that the white phase corresponds to Li_2TiO_3 while the black phase corresponds to BeO which is depicted in the Figure 4.

Density measurement

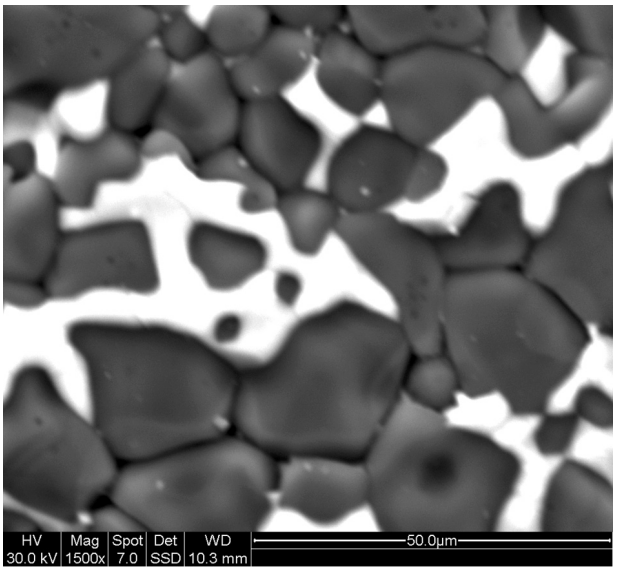
The measured density and the porosity of the different compositions are given in Table 3. The density of BeO–Li_2TiO_3 composites are found to decrease with decrease in volume fraction of Li_2TiO_3, which is can be explained by the lower theoretical density of BeO (i.e. 3020 kg/m^3) compared to Li_2TiO_3(i.e 3430 kg/m^3). The maximum density of the sintered pellet was observed with BeO–Li_2TiO_3 volume ratio 55:45. The measured densities of the samples showed that there is around 2.5 - 5.5 % of porosities present in the sample.

Specific heat

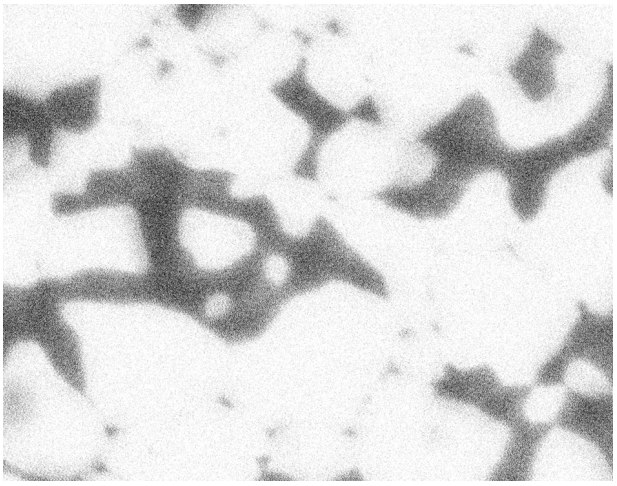
The specific heat measured by DSC for all the four compositions are shown in the Figure 5. In the same graph, the specific heat of Li_2TiO_3 measured by DSC is plotted. Also, the specific heat data of BeO taken from the literature is also shown [5, 6]. It is seen that the maximum specific heat is observed for the BeO–Li_2TiO_3 pellet of 80:20 volume ratio and it reduces with increase in Li_2TiO_3 content.

Co-efficient of thermal expansion (CTE) studies

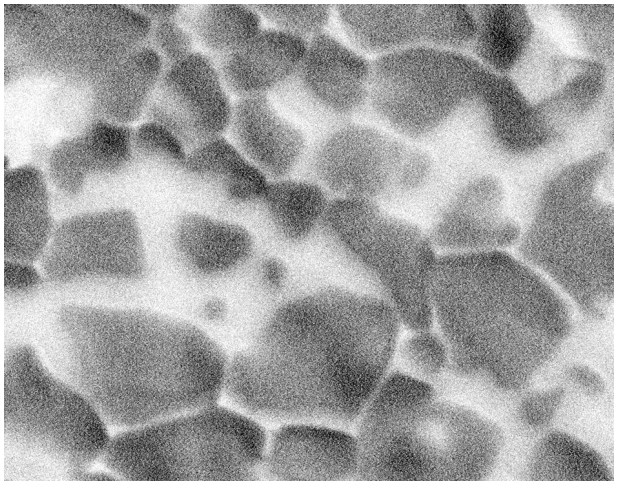
CTE was measured under argon atmosphere up to the temperature of 1073 K. As there was difficulty in the fabrication of the samples of dimension of 10 mm long and 5 mm diameter samples, only two samples of 65 BeO–35 Li_2TiO_3 and 75 BeO–25 Li_2TiO_3 compositions could be tested. The change in length vs temperature plot is given in the Figure 6. The average CTE value is ~ (11 - 11.5) $\times 10^{-6}$/K which is far better than the Li_2TiO_3 which is ~ 18×10^{-6}/K.



a) 80 BeO:20 Li_2TiO_3



b) titanium mapping



c) oxygen mapping

Figure 4. BSE image for 80 BeO:20 Li_2TiO_3 composition a), titanium mapping b) and oxygen mapping c).

Table 3. Density values for the different BeO–Li_2TiO_3 compositions.

Sample No.	Sample	Weight of the sample (kg) $\times 10^{-3}$	Volume of the sample (m^3) $\times 10^{-6}$	Density (kg/m^3)	Porosity (%)
1	BeO–Li_2TiO_3 (55:45)	0.74625	0.2391	3120	2.5
2	BeO–Li_2TiO_3 (65:35)	0.50760	0.1653	3070	2.85
3	BeO–Li_2TiO_3 (75:25)	0.78526	0.264	2970	4.8
4	BeO–Li_2TiO_3 (80:20)	0.72064	0.2459	2930	5.48

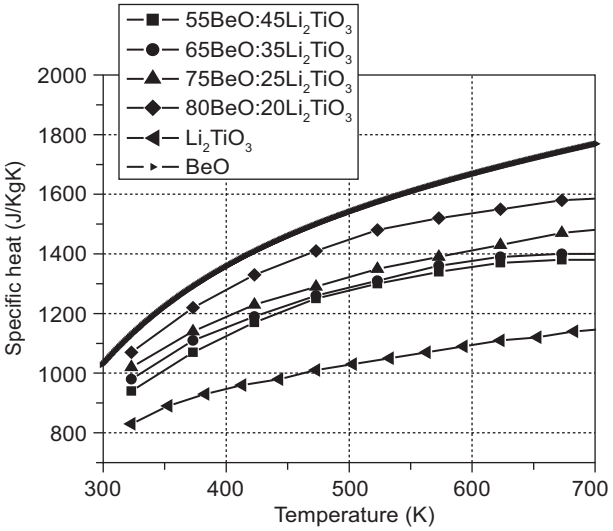


Figure 5. Specific heat variation of all the four samples with temperature.

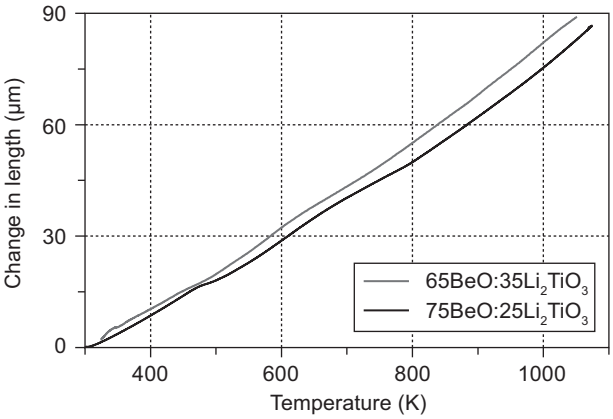


Figure 6. Change in length vs temperature plot for two compositions.

Thermal diffusivity

The measured thermal diffusivity (α) values are shown in the Figure 7. In the same graph, the experimental thermal diffusivity of Li_2TiO_3 and literature thermal diffusivity data of BeO are also plotted [5]. The thermal diffusivity data for Li_2TiO_3 is in very good match with the reported data [7]. From the graph, it is observed that the thermal diffusivity is the maximum for BeO and it shows a reducing trend with increase in Li_2TiO_3 content.

Thermal conductivity

The calculated thermal conductivity values are shown in the Figure 8. The experimental thermal conductivity of Li_2TiO_3 and literature thermal diffusivity data of BeO are also plotted [5]. It is observed from the figure that with increase in the temperature, thermal conductivity reduces and it is the lowest for the BeO–Li_2TiO_3 pellet of 55:45 volume ratios. The novel BeO–Li_2TiO_3 mixed ceramic material prepared by sol-gel route showed much improved thermal conductivity compared to Li_2TiO_3. In

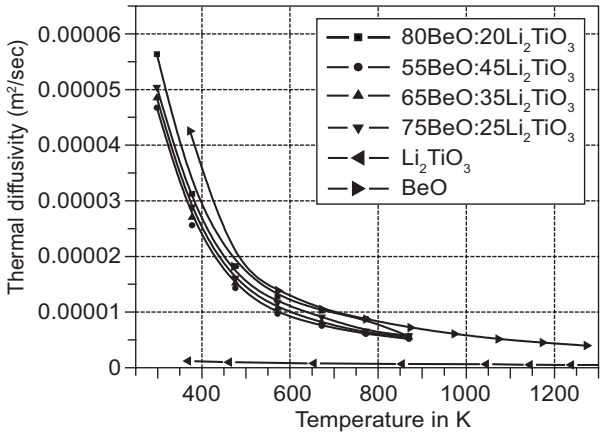


Figure 7. Thermal diffusivity values for the samples.

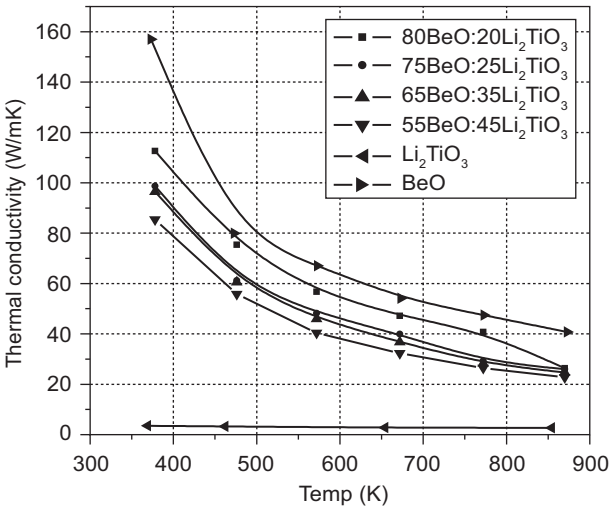


Figure 8. Thermal conductivity values for the samples.

general, the thermal conductivity of Li_2TiO_3 is close to 2.4 W/m·K [7, 8]. So by the use of novel BeO–Li_2TiO_3 there is a large benefit of better thermal conductivity keeping tritium breeding ratio > 1.1.

Many models are there for predicting the thermal conductivity of composite systems [9]. Among the models, the Russel theoretical model [10] with a power factor of (1/4) and geometric mean models [11] with empirical constant of 1.4 predict a very good match for this type of composite. The results of the measured thermal conductivity and the calculated thermal conductivity are presented in the table 4 for a particular temperature of 572 K.

The formulation of Russel model is as follows:

$$k_e = \frac{k_{BeO}\,[\Phi^{1/4} + (k_{BeO}/k_{Li_2TiO_3}) \cdot (1 - \Phi^{1/4}]}{\Phi^{1/4} - \Phi + (k_{BeO}/k_{Li_2TiO_3}) \cdot (1 + \Phi - \Phi^{1/4})}$$

where, k_e is the effective thermal conductivity, k_{BeO} is the thermal conductivity of BeO, $k_{Li_2TiO_3}$ is the thermal conductivity of Li_2TiO_3 and Φ is the volume fraction of Li_2TiO_3 in the compact.

The formulation of geometric mean model is as follows:

$$k_e = k_{BeO}^{(1-\Phi)} \cdot k_{Li_2TiO_3}^{\Phi} \cdot 1.4$$

Electrical Property Measurements

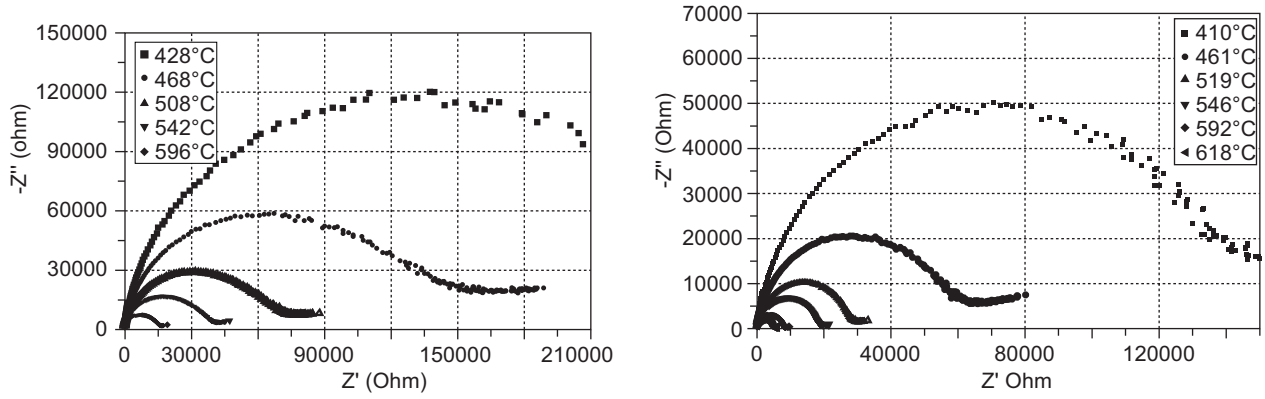
The electrical conductivity of the composites with $BeO:Li_2TiO_3$ volume ratios of 80:20 and 55:45 were determined by impedance spectroscopec measurements. Figure 9a, b gives the typical impedance plots (Nyquist plots) for 55 BeO:45 Li_2TiO_3, and 80 BeO:20 Li_2TiO_3 samples recorded at different temperatures. For each sample, single semicircles were obtained for the sample at different temperatures. The magnitude of the semicircles was found to decrease progressively with the increase in temperature. The intersection of the arc with the real axis gives the resistance R_{dc} for the sample which can be related to its conductivity σ_{dc} by:

$$\sigma_{dc} = (1/R_{dc}) \cdot L/S$$

where L is the thickness and S is the area of cross section of the pellet. Figure 10a and b shows the plot of log σ versus reciprocal temperature that is the temperature dependence of the bulk dc conductivity for the 55 BeO:45 Li_2TiO_3 and 80 BeO:20 Li_2TiO_3 samples. The activation energy of 55 BeO:45 Li_2TiO_3 and 80 BeO:20

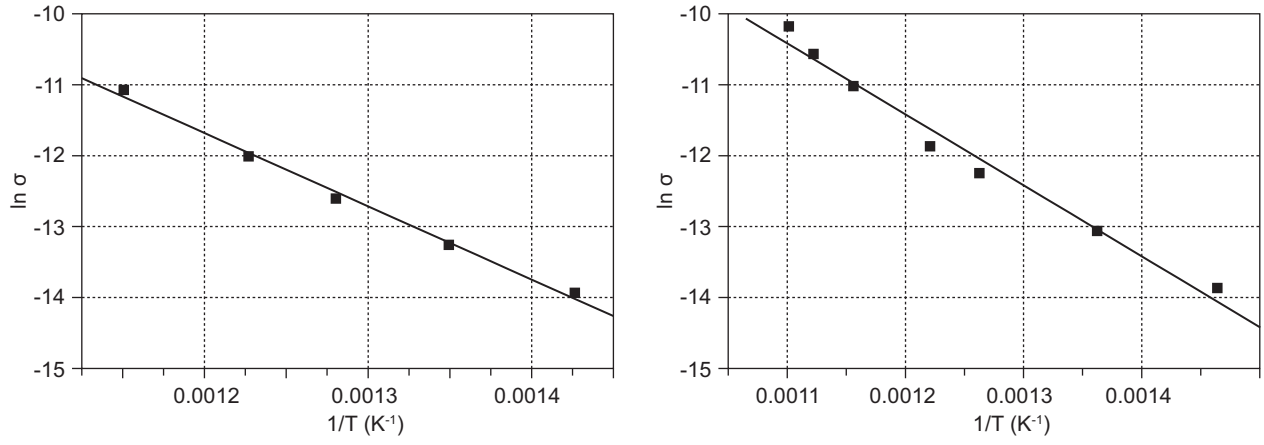
Table 4. Calculated and measured thermal conductivity for the four samples at 572 K.

Volume ratio of $BeO:Li_2TiO_3$	Thermal conductivity (W/mK)		Measured thermal conductivity (W/mK)	Calculated thermal conductivity (W/mK)	
	of BeO	of Li_2TiO_3		by geometric mean model	by Russell model
80:20	68	2.99	56	51	44
75:25	68	2.99	48	44	39
65:35	68	2.99	45	32	30
55:45	68	2.99	40	23	23



a) 55 BeO:45 Li_2TiO_3 b) 80 BeO:20 Li_2TiO_3

Figure 9. Nyquist plots for a) 55 BeO:45 Li_2TiO_3 composition, b) 80 BeO:20 Li_2TiO_3 composition.



a) 55 BeO:45 Li_2TiO_3 b) 80 BeO:20 Li_2TiO_3

Figure 10. log σ versus reciprocal temperature for a) 55 BeO:45 Li_2TiO_3 composition, b) 80 BeO:20 Li_2TiO_3 composition.

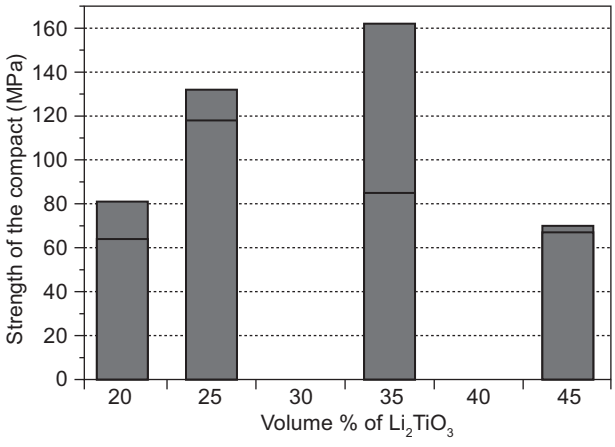
Li_2TiO_3 samples deduced from the linear part of the plot of log σ versus reciprocal temperature. The activation energy was found to be 0.88 and 0.855 eV for 55 and 80 BeO containing samples respectively.

Mechanical property evaluation

In order to assess the mechanical properties, cubic pellets of 12.5 mm length were prepared and tested in TIRA UTM machine at a strain rate of 10^{-4}. As the samples contain BeO, so special precautions were taken during testing, as shown in the Figure 11a. The modulus values were determined by ultrasonic testing using longitudinal and transverse velocity in the sample. Though only two samples from each of the compositions could be tested, but still the encouraging result is that it gives a boost that the strength values can be achieved far better than the Li_2TiO_3 as depicted in the Figure 11b. The strength of sintered Li_2TiO_3 pebbles is ~ 100 MPa [12].

The modulus of elasticity was determined for 55 BeO–45 Li_2TiO_3 composition in which the longitudinal velocity and the transverse velocity was found to be 9132 m/sec and 5378 m/sec respectively. So for this density, the elastic modulus comes out to be 220 Gpa which is far better than Li_2TiO_3 which is close to 100 GPa.

a)



b)

Figure 11. Compression test set-up a) and graph of compressive strength b) for different compositions (two samples each at all compositions).

CONCLUSIONS

BeO and Li_2TiO_3 composites with volume ratios of 80:20; 75:25; 65:35 and 55:45 with 2.5 - 5.5 % of porosities have been synthesized and characterized by XRD, SEM-EDX. XRD analyses reveals that the BeO and Li_2TiO_3 retained their identities separately and do not form any ternary compound and the phases are uniformly distributed in the sample. The coefficient of thermal expansion values for the composites are in the range of ~ 10 $\times 10^{-6}$ K^{-1}, which is half of that of the Lithium titanate. The novel BeO–Li_2TiO_3 mixed ceramic material prepared by sol-gel route showed improved thermal conductivity compared to Li_2TiO_3. The thermal conductivity for 80 BeO–20 Li_2TiO_3 is 113 W/mK at 378 K which reduces with increase in temperature. The thermal conductivity at 672 K which is close to the operating temperature is 47 W/mK. At the same temperature the thermal conductivity of Li_2TiO_3 is 2.8 W/mK. So there is a large improvement in thermal conductivity by use of this novel mixed oxide of BeO and Li_2TiO_3 instead of using Li_2TiO_3 and metallic Be separately. The activation energy for conductance increases with addition of Beryllium-oxide. The impedance measurement showed that with the addition of BeO, there is reduction in electrical conductivity which is better to be used in the fusion reactor. The mechanical strength of BeO–Li_2TiO_3 composites are found to better than that of pure Li_2TiO_3. The composites are found to have better modulus properties in comparison to Li_2TiO_3.

From the present investigation, it could be concluded that it is possible to achieve tritium breeding ratio (TBR) more than 1.1 by optimizing the BeO and Li_2TiO_3 ratio with better properties. Further studies on neutron irradiation are necessary to assess the TBR values and fix the appropriate composition of BeO and Li_2TiO_3.

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APPENDIX-1

A 3-D Monte Carlo neutronic calculation has been carried out to estimate the Tritium Breeding Ratio (TBR) for Indian DEMO. A neutronic model was constructed in which BeO–Li_2TiO_3 was taken as the breeder and multiplier material and EUROFER as the reference structural material. The main parameters for the IN DEMO machine are given in the Table A.1.

Table A-1. Main Indian DEMO parameters.

Fusion Power	3300 MW
Plasma major/minor radius	7.7/2.6 m
Breeder/Multiplier material	BeO–Li_2TiO_3 compound with 15 % porosity
Li-6 enrichment	40 %
Structural material	LAFMS
Volume percentages of Structural and Breeder/Multiplier material	30 % and 70 %

Modeling analysis was performed for a range of volumetric fractions of BeO and Li_2TiO_3 in the breeder mixture compound to estimate the tritium-breeding ratio. In general, for the Indian DEMO blanket the required TBR is aimed > 1.1 for tritium self-sufficiency. The modeling estimation will help us to determine the volumetric composition of the mixture in which the TBR is the highest. The first estimation is based on 40 % enrichment of Li-6 and 30 % of structural material used in the breeding blanket. The parametric study has been carried out to calculate the TBR for volumetric fractions ranging from 5 - 80 % of Li_2TiO_3 and the corresponding 95 - 20 % of BeO. The Figure A-1 shows TBR as a function of the Li_2TiO_3 volume fraction for the full range at 85 % and 100 % sintered density of BeO–Li_2TiO_3. It is clear from the Figure A.1, that initially the TBR rises with the increase in Li_2TiO_3 volume percentage up to 35 %, later gradually decreases for higher percentage of Li_2TiO_3. The initial increase is due to the increase in Li content in the compound. The combination of 35 % Li_2TiO_3 and 65 % BeO in the mixture of BeO–Li_2TiO_3 provides the maximum TBR of 1.135. As the Li_2TiO_3 volume ratio further increases, for higher volume ratio of Li_2TiO_3, the Beryllium content reduces and therefore the neutron multiplication reduces which has an impact on TBR. So, from this neutronic analysis, the compositions for the experiment have been taken.

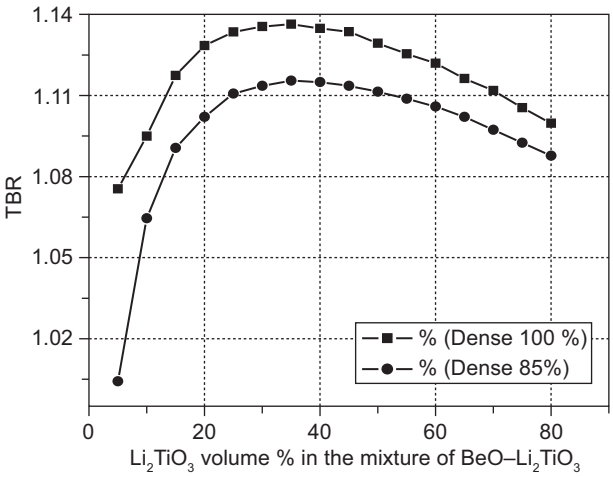

Figure A-1. TBR as a function of the Li_2TiO_3 volume fraction for 85 % and 100 % sintered density of BeO–Li_2TiO_3.