*Impact Factor (SJIF): 3.632*

**International Journal of Advance Research in Engineering, Science & Technology**

*e-ISSN: 2393-9877, p-ISSN: 2394-2444*

***Volume 3, Issue 6, June-2016***

# $Li_2TiO_3$ pebble preparation by extrusion spheronization and sintering process for fusion blanket application

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***Abstract***

*India is developing β-$Li_2TiO_3$ Lithium Titanate at Institute for Plasma Research. Lithium titanate will be kept inside the canisters of TBM (Test Blanket Module) in International thermonuclear experimental reactor (ITER) in form of spherical pebble bed. Typical size range of these pebbles would be ~ 1 mm. Lithium titanate ($Li_2TiO_3$) ceramic powder development is currently on-going by the solid state reaction process. Reactant material is calcined at (500$^o$C 600$^o$C 700 $^o$C 800$^o$C 900 $^o$C 1000 $^o$C 1050 $^o$C 1100 $^o$C and 1150$^o$C) at 4 hr, 8hr and 12hr. the optimization of solid state reaction is carried out by XRD(X-ray Diffraction), and $Li_2TiO_3$ Powder has been prepared at 1000$^o$C 4hr.further lithium titanate powder is passed through the process of ceramic extrusion –spheronization-Sintering of ceramics to Prepare spherical Pebble. At every stage of powder and pebble preparation extensive characterization has been carried out. Several physical and mechanical properties are characterized. Sintering parameters of the $Li_2TiO_3$ pebbles are also optimized. Surface morphology study of $Li_2TiO_3$ pebbles are also discussed in this paper.*

## I. INTRODUCTION

The human nature is depending upon electricity for the function of our technical society. We use electricity to light our world, to heat our homes, and to transfer the information, etc. It is expected that after 5 years our energy consumption will be increased by 80%, as fast growth of technology in world. As per the current rate of consumption, we could find ourselves with the shortage of fossil fuels in this century. Unfortunately, our resources cannot sustain even this amount of energy use forever. Nuclear fission accounts approximately 20% of current energy production in world [1]. In nuclear fission power plants, heavy atoms are split into lighter atoms, produce inconvenient amounts of radioactive waste products which are hard to dispose safely. The uranium-235 used in these reactions, is somewhat limited. Solar, wind, hydroelectric and geothermal energy are some other methods of creating electricity, but they cannot generate nearly enough energy to satisfy demands. As per the current rate of electricity consumption, the nuclear fusion process is a key for production of electricity.

Mega engineering project on nuclear fusion doing by the ITER (International thermonuclear experimental reactor), which will be the world's largest magnetic confinement plasma physics experiment which is currently building the world's largest experimental tokomak nuclear_fusion reactor adjacent to the in the south France [2]. The ITER project aims to make the long-awaited transition from experimental studies of plasma physics to full-scale electricity-producing fusion power plants. Nuclear fusion is a reaction in which two lighter nuclei are joined (fused) to create a larger nucleus. The nuclear fusion works based on deuterium (D) and tritium (T) reaction and generate the high energy neutron. This energy is extracted to generate the electricity. The ITER fusion reactor has been designed to generate 500 megawatts of output power while giving 50 megawatts to operate[3]. For effective operation of a typical fusion power plant sufficient amount of deuterium and tritium is need. Deuterium is readily available in sea water with 0.015% [4] however tritium availability in world is in insufficient amount. Tritium can be produced during the fusion reaction through the contact of lithium with neutron. Since fusion reactor operates at elevated temperature, many researchers have already tried to develop suitable lithium compounds.

Among various lithium compounds[5] viz. $Li_2O$, $LiAlO_2$, $Li_2ZrO_3$, $Li_4SiO_4$ and $Li_2TiO_3$, India is focusing on $Li_2TiO_3$ due to its excellent tritium releasing behavior at low temperature [6] and good thermal and chemical stability inside the fusion reactor. Development of $Li_2TiO_3$ powder is currently ongoing at Institute for Plasma research (IPR) with solid state reaction method.

$Li_2TiO_3$ ceramic will be kept inside the ITER in form of packed spherical bed. Size of these pebbles will be ~ 1.00 mm. out of various conventional methods like Solid State Method [7], Solution Combustion reaction[8] and Sol gel method[9] we have adopted solid state reaction method for powder preparation and extrusion spheronization sintering process[10] for pebble preparation.

## II. EXPERIMENT PROCEDURE

Solid state reaction involves the mixing of solid state reactants. Since these reactants do not react at room temperature, the increase in the temperature is essential. This increase in the temperature provides the sufficient activation energy to initiate the reaction. To prepare a homogeneous mixture of the raw material sufficient mixing is required. $Li_2CO_3$ (lithium carbonate, ACS reagent ≥99.0%, Sigma Alderich) and $TiO_2$(Titanium dioxide,ACS regent,99-100%,Sigma Aldrich) are taken in a stoichiometric ratio(1:1 molar).

$$Li_2CO_3 + TiO_2 \rightarrow Li_2TiO_3 + CO_2$$

To achieve micron range of particle size wet grinding is preferred over dry grinding. Therefore isopropyl alcohol is added to make a slurry of the reactants. Reactants are further grinded & mixed inside the ball mill (Fritzsch GmbH, Zirconia bowl with zirconium oxides balls). Ball to Powder ratio kept 1.6 with operational speed of ball mill 350 rpm. Grinding and mixing is performed for 3 hr. after the grinding process, the reactants are recovered from Zirconia bowl by using isopropyl alcohol. The excess alcohol is further recovered by the distillation method. The dried material is further calcined inside the electrically heated high temperature muffle furnace (Nabertherm GmbH). Several experiments are performed to pin point the calcination temperature and time[11]. Experiments are performed on 500°C to 1150°C with 4 h, 8h & 12 h calcination time. After calcinations, obtained product is analyzed for its phase purity. It is observed that the obtained product is hard in nature (obtained in form of solid chunk) with coarse particle size which is not suitable for the pebble preparation. Pebble preparation needs fine particle size; therefore this material is again grinded with high energy ball mill. The flow chart of the process is described below.

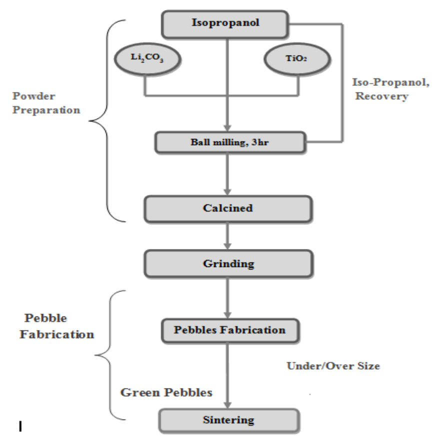



**Figure1: Flow chart of Pebble Fabrication**

### Pebbles Preparation by Extrusion-Spherionization-sintering Process

Pebble preparation needs fine particle size; therefore this material is again grinded with high energy ball mill. To prepare a dove of $Li_2TiO_3$, this powder is mixed with poly vinyl alcohol solution in various compositions. Through a hopper this dove is fed to single roller extruded which consist 1 mm sieve mesh at the end. Due to the force applied by the roller, Li2TiO3 is extruded through the sieve and converted into 1 mm extrudes of variable length. To convert L:D(Leangth:Diameter) ratio 1, these extrudes were fed into the rotating disk (chequered plate,3.25). This rotating disk cuts the extrudes of variable length to 1 mm to form L:D ratio 1. The obtained cylindrical extrudes of 1x1 mm (D x L) follows the rope way motion inside the chamber due to the centrifugal force and forms a spherical shape. The pebble fabrication parameters are optimized for ~1.00 mm pebble preparation.

## III.RESULTS AND DISCUSSION

### Powder Preparation

Below figure is the representation of 4hr ,8hr, 12hr calcination time with temperature. It is observed that with increase in the reaction temperature conversion % is decrease. In other words with increase in reaction temperature weight loss is observed. The observed weight loss is compared with theoretical values and found equal.

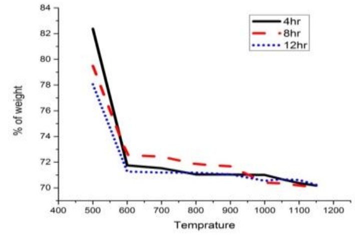
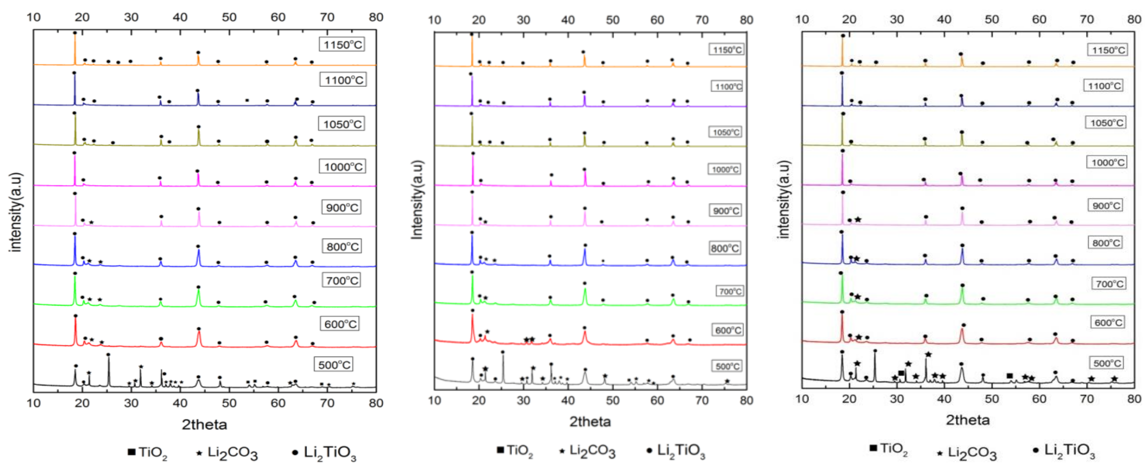



**Figure 2: graph of 4,8 and 12 hrs of $Li_2TiO_3$ Powder**

Prepared material is characterized for its phase purity by X-ray diffractrometer (Bruker D8 Advanced Vantec). The obtained data is compared with JCPDS card (Joint committee for powder diffraction standards) $Li_2TiO_3$–00-033-08331, $Li_2CO_3^-$ 87-0728, $TiO_2^-$ 89-6975. Three different graphs with calcination temperature 4-12 h with respect to temperature are shown below. From fig. 3 it is observed that at 500°C, several peaks of unreacted $Li_2CO_3$ and $TiO_2$ are observed. These unreacted peaks disappear only after 900°C calcination temperature. It is also found that the monoclinic crystal structure is obtained. However increase in calcination temperature leads to coarse particle size and also material formed in form of hard chunks. Therefor 1000°C and 4 h calcination is considered as process parameters for further $Li_2TiO_3$ preparation.




**(a) 4hr** **(b) 8hr** **(c) 12hr**

**Figure3: X-ray diffraction at different temperature with 4, 8 and 12hr**

## Surface morphology of $Li_2TiO_3$ Pebbles

$Li_2TiO_3$ pebbles are characterized with scanning electron microscopy. Surface morphology is studied is carried out with this characterization. Pebbles sintered with three different time 5 h, 15 h , 25 h are given below. With SEM image analysis it is observed that grain size of pebbles sintered at 1100°C for 5 h is up to 3 micron. As the sintering time increased to 15 h the grain growth appears and the grain size increases up to 10 micron. Sintering up to 25 h leads the grain size up to 20 micron. As per figure 4, with increase in the sintering time the grain growth is significant it's also observed that the visible surface porosity is decreasing with increase in sintering time.

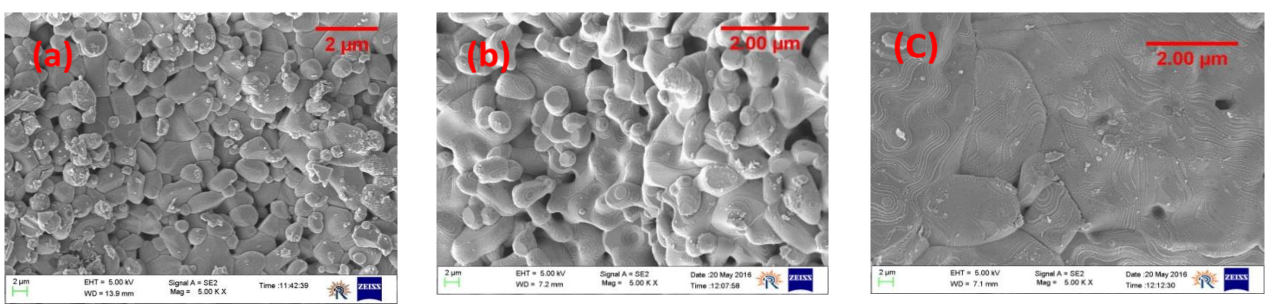



**Figure 4 : Grain size of Li2TiO3 pebbles (a) 1100°C, 5 h sintered (b) 1100°C, 15 h sintered (c) 1100°C, 25 h sintered**

## Mechanical Properties

Crush strength of sintered spherical pebbles are measured with crush load tester. It is observed that with increase in the sintering time there is significant increase in the crush strength of the $Li_2TiO_3$ pebbles. Crush strength of the pebbles sintered at 1100°C for 25 h is comparable with 1100°C 15 h.

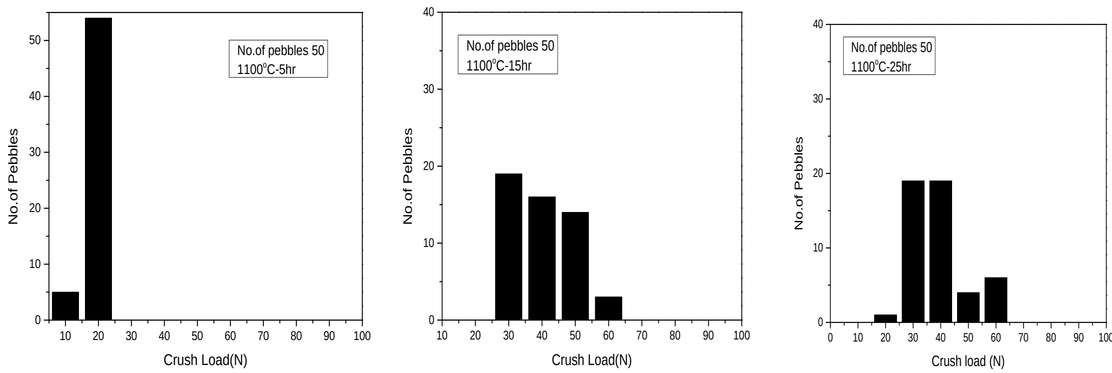



**Figure 5 : (a)1100°C-5hr (b)1100°C-15h (c)1100°C-25hr**

## IV.CONCLUSION

$Li_2TiO_3$ Powder Prepared by solid state reaction process and Experiments performed from 500-1150$^o$C with different heating rate of 4,8 and 12hr. obtained product is characterized for its phase purity with XRD analysis and it is observed that the powder is prepared on a calcination temperature > 900$^o$C, however powder calcined >1050°C possess hard nature. Due to its hard nature it is difficult to prepare pebbles therefore the calcination temperature for powder preparation is 1000$^o$C- 4hr is optimised parameter. $Li_2TiO_3$ Pebbles fabrication from $Li_2TiO_3$ powder by extrusion Spherionization & sintering process is performed. Physical and mechanical characterization of $Li_2TiO_3$ powder and pebbles are carried out. From microscopic analysis it is observed that fabricated pebbles are in the range of (0.85-1.18mm Diameter). Fabrication parameters are optimized with the desired properties. Other physical properties like surface area, sphereocity, thermal conductivity, thermal diffusivity & specific heat is also planned. Thermo-physical and thermo-mechanical properties are also in future scope**.**

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