THERMO-MECHANICAL EXPERIMENTS ON LITHIUM TITANATE PEBBLE BED

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Abstract

Among the various lithium ceramics, Li_2TiO_3 is one which has been received much attention due to its very excellent properties, such as reasonable lithium atom density, low activation, excellent tritium release performance and chemical stability, etc. Lithium Titanate [Li_2TiO_3] pebbles with the diameter of 1mm was widely used for the experiments after successful completion of variety of modeling and experiments. In the present study we have prepared lithium titanate from its high pure raw material of lithium carbonate and titanium dioxide by solid state reaction in the stoichiometric ratio. The reaction temperature has been estimated from the thermo-gravimetric and differential thermal analysis (TG-DTA) and the same scenario has been executed for the bulk production using high temperature furnace. The phase and phase stability at different temperature were analyzed by using powder X-ray diffractometer. The pebble preparation has been carried out from this raw material after once again ground them to fine powder and addition of PVA as a binder for the preparation of green pebbles using extruder-spheronizer technique. The green pebbles were sintered at high temperature to attain desired density for further studies. The details of the Li_2TiO_3 powder and pebble fabrication and their characterizations like XRD, density, porosity, crush load, SEM analysis will be discussed in the paper. The thermo-mechanical experiments such as Young's modulus and Creep of the ceramic breeder pebble bed is of large importance for the calculation of bed-structure interaction which is important for the assessment of the blanket lifetime. The measurement has been carried out for mono-sized pebbles in the range of 0.85 – 1.2 mm with the theoretical density of 85 % using a uniaxial test facility with varying pressure at temperature up to 750 ^oC. The packing fraction of the whole experiment has been approximately around 63% within the vacuum of <100 mTorr. Temperature dependent modulus and bed volume reduction has been observed from the present experiment. The analyses emphasize an increase of strain with continuous cycle and become saturate after certain number of cyclic loading in the stress-strain relation of pebble bed. Creep as a function of temperature, pressure and time is analyzed in the present experiment and observed there is an influence of pressure, the material doesn't get stabilized after 750 ^oC, 2 MPa. The material with varying creep strain doesn't seems to be good for fusion reactor material, so its suggested to be use this material up to 750 °C, 2 MPa.

1. INTRODUCTION

Lithium-containing ceramics such as Li_2O, γ-$LiAlO_2$, Li_4SiO_4, Li_2ZrO_3 and Li_2TiO_3 have been considered as candidates for tritium breeding materials in D–T fusion reactors like DEMO [1-5]. Among those ceramics, Li_2TiO_3 have been received much attention due to its excellent properties such as low activation, excellent tritium release performance and chemical stability, etc [6-9]. In the Indian LLCB TBM (Lead Lithium Ceramic Breeder Test Blanket Module) for testing in ITER, Li_2TiO_3 in the form of pebbles will be used as the tritium breeding material [10]. Pure lithium titanate is prepared in the laboratory from its raw material by solid state reaction. The material prepared is processed well to get fine power for the pebble fabrication. The prepared pebbles are sintered at required temperature to attain properties which is important for the TBM mainly thermal, mechanical and chemical. These pebbles are of ~ 1 mm and arranged in to a box like structure for tritium fuel production in the ceramic breeder blanket called pebble bed. During operation the pebble bed undergoes various thermo mechanical effects due to thermal stresses arise on the thermal expansions of the pebble bed, structural material and pebble bed swelling due to irradiation. As per the conceptual design data of Indian TBM, solid breeder material will experience a maximum temperature in the range of 450 to 950 ^oC and the possible pressure in the range of 2 MPa due to lead lithium and helium purge gas. In order to describe these thermo mechanical behaviour, experiments were carried out in association with theoretical predication with appropriate finite element codes for pebble beds [11]. The generated experimental data will be used as an input data for the finite element code for the prediction of pebble bed characteristics which will be useful for the comparison of experimental and theoretical observation. There are number of experiments performed by different researchers using prototypical model of pebble bed on stress-strain relations of the ceramic breeder pebble beds in the past using oedometric test apparatus [11-14]. In this paper a uniaxial compression test (UCT) have been performed for Lithium titanate pebbles for the determination of uniaxial stress-strain relationship, moduli of deformation

and creep for relevant temperature and pressure range. The pebbles used in the present experiments were of potato shaped and prepared by extrusion technique. The as prepared material was sintered at 1100 ^{o}C to achieve desired density and porosity. Pebbles of ~1 mm dia (0.8-1.2 mm) have been used for the present pebble bed experiment which is basically considered as mono sized pebble. In the present stress-strain experiment we have carried out temperature up to 750 ^{o}C due to the limitation of the test facility and pressure varying from 0 to 6 MPa which is higher than the expected or design limit of pebble bed but in case of creep we have carried out the measurement upto 4 Mpa.

2. EXPERIMENTAL

2.1. Material Preparation

Lithium titanate has been prepared from lithium carbonate and titanium dioxide powder in stoichiometric ratio (1:1). High pure raw materials from Merck and Sigma Aldrich have been used for the present material preparation. Both materials were mixed together by mechanical alloying with the help of iso-propyl alcohol as an agent. High quality ZrO bowls and balls were used for the mixing and milling purpose to avoid contamination in the raw material. The milling/mixing time has been finalized after several trials in different batches followed by solid state reaction. From the trial experiment data for our R&D activity we have carried out 3h ball milling at a speed of 200 rpm which is observed better after X-ray analysis. After ball milling the material were dried well with the help of an oven to remove the iso-propyl alcohol and then transferred the same in to an alumina crucible. For calcination at high temperature based on the thermal analysis data the material were kept on a high temperature programmable furnace. After the completion of solid state reaction the calcined material were removed and carried out X-ray analysis for the phase confirmation and other experimental activities.

2.2. Pebble Preparation and Sintering

The qualified materials after powder X-ray analysis were used for pebble preparation. After solid state reaction the material becomes very hard, so before pebble preparation the material made into fine powder with motor pestle and by ball milling for certain hours to avoid its coarse nature of the powder. This fine powder is added with binder material named PVA (poly vinyl alcohol) to make its dough. Later the dough is inserted into an extruder which will make fine noodles depends on the nozzle used and later transferred the noodles into the spheronizer for pebble making. The pebble size is depending upon the pitch size used in the spheronizer and shapes will completely depend upon the speed of rotation of the speronizer. The green pebble will be very soft and it's having PVA content which has to be removed before further characterization. Sintering has been carried out at different temperature and time for removing PVA and to enhance the density, crush load strength etc.

2.3. Characterization

Thermal analysis was used to find out the weight loss (thermogravimetric analysis, TGA) and energy change (differential thermal analysis, DTA) in the sample with respect to the temperature. The thermal analysis were carried out on the mixed raw material using a combined TG/DSC (Model: STA PT 1600) analyzer at a heating rate of 2° C/min in an inert atmosphere. Powder X-ray diffraction analysis of the prepared ceramic powder was carried out using a Bruker powder X-ray diffractometer with Cu Kα radiation (35 kV, 30 mA), scanning with a step size of 0.02° at ambient temperature for the angular range 10–70° of 2θ, to determine the crystal system and lattice parameters. Grain size is one of the important parameter which can give an idea of quality of the pebbles for tritium breeding is carried out using SEM. Germen made VP Merlin High Resolution Scanning Electron Microscope (HR-SEM) (Model: Carl Zeiss) has been used for the present study. True density of the prepared pebbles were carried out using Gas (Helium) Pycnometer (Model: HumiPyc^{TM}) measures true volume (density) of objects using gas displacement method and employing the ideal gas law, pV = nRT. Porosity of the pebbles was carried out using mercury porosimeter POREMASTER 60. The strength of the material is tested using crush load tester Campbell Electronics Hardness Tester, DHT – 250. Uniaxial compression test and creep has been carried out at UCLA-USA using their test stand (Zwick, Kappa 50 DS) at higher temperature.

3. RESULTS AND DISCUSSIONS

3.1. Thermal Analysis

The materials mixed together were gone through thermal analysis and shown in Fig.1. From the graph it is observed a gradual slope from the beginning of the TG which is related to the remaining iso-propyl content present in the material even after the material is dried with oven. But a prominent dip has been observed around 500 deg C and it continues upto 680 deg C which is basically shows the material starts decompose and main weight reduction is happening at this point which is due to the phase change. Soon after the phase change there is another weight reduction initiated this may leads to next phase change if we treat the material at high temperature. The sharp endothermic peak around 730 deg C shows that lithium titanate formed at this temperature and it can sustain in this phase unless until the next phase change temperature has been reached. The crystallinity will get increased if we go above 730 deg C and this has been confirmed using powder X-ray diffraction technique. Based on these thermal analysis data we have fixed the solid state reaction temperature should be above 700 deg C and we have tried different set of samples and XRD analysis are provided in the forth coming section.

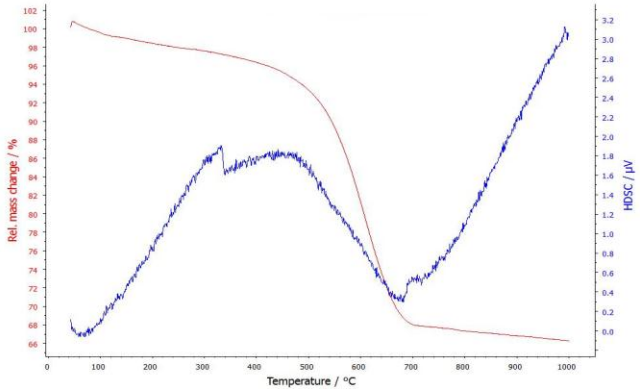

Fig. 1. TG-DSC curve of lithium titanate powder.

3.2. X-ray Analysis

Based on the TG-DSC result of our sample and Phase diagram of Li_2TiO_3, we have started working calcination temperature from 600 to 1100 deg C to find out and fix the calcination temperature and time for present R&D work. The calcination time of 6 h soaking period kept for all experiment for the initial trial and did experiment on different temperature. It is observed that in all temperature Li_2TiO_3 phase has been formed but with certain variation in the phase purity as per the Bruker-TOPAS software. The temperature range 600 to 900 deg C have a lot of unreacted material but it is in the decreasing order towards temperature increases, 1000 and 1100 deg C have mostly matching with all reported peak positions of Li_2TiO_3 in the PDF 00-033-0831. We have fixed 1000 deg C for our powder preparation because 1100 deg C is bit close to next phase formation as per the phase diagram of Li_2TiO_3. Fig. 2. Shows the powder xrd spectra of the sample prepared at 1000 deg C and by using software's we have calculated the lattice parameters of the material which is tabulated in Table. 1. Later the material has gone through isotopic abundance study as well. From the isotopic abundance study $^6Li/^7Li$ atom ratio is observed as 0.0824 ± 0.0003 and atom % of ^6Li is 7.61 ± 0.03 which is better than the commercially available product which we have purchased and tested with our sample.

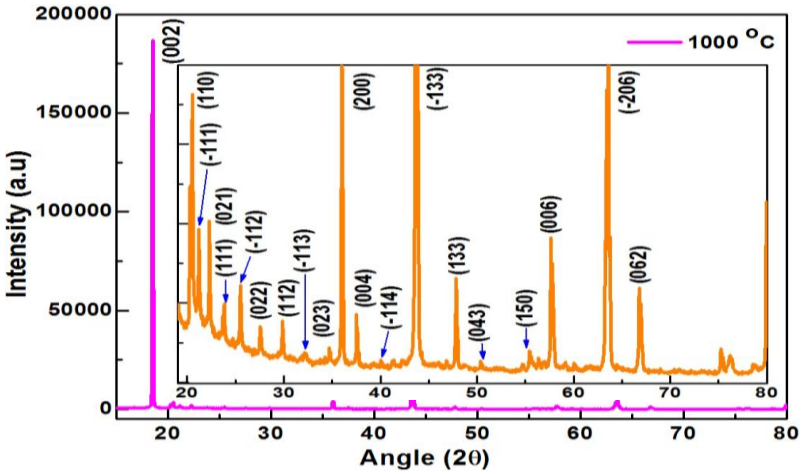

Fig. 1. Powder XRD pattern of lithium titanate prepared at 1000 deg C.

TABLE 1. Powder XRD data of lithium titanate powder prepared at 1000 deg C

Temperature	2θ	Intensity	hkl	Lattice Parameter	Phase Purity (TOPAS)
1000 °C	18.5401	186756	002	a = 5.0659 Å	Li_2TiO_3 = 99.70 %
	20.2751	2431	020	b = 8.7877 Å	TiO = 0.00 %
	20.5230	3634	110	c = 9.7375 Å	TiO_2 = 0.00%
	21.1990	1936	-111	α = 90 °	Li_2O = 0.14 %
	22.2806	2035	021	β = 100.160 °	SiO_2 = 0.16 %
	25.5029	1227	-112	γ = 90 °	
	29.8518	780	112	Vol = 426.70 $Å^3$	
	35.9808	4303	200, -131		
	43.6422	11110	-133		
	47.8559	1319	133, -204		
	57.6353	1825	-135, 006		
	63.4264	3692	312		
	63.5841	4509	-206		
	66.7839	1195	-333, 062		

3.3. SEM Analysis

The pebbles prepared by extrusion and spheronization has been sintered at different temperature for good density so that it can sustain good amount of load and even at higher temperature. But at the same time it should have porous nature as well otherwise the tritium will not be able to taken out from the pebble so that the density and porosity should be at the optimum range so that one can extract maximum tritium from the pebble. Here in this present study we have carried out SEM analysis of the pebble treated at different temperature and different sintering time. The grain size from the images was measured by using the software ImageJ Fiji win-64. An average of 30 grains from each picture is used for the grain size measurement the detailed data is tabulated in Table 2.

TABLE 2. Grain size measurement of pebbles sintered at different temperature and time

Sr. No.	Sintering Time (h)	Average Grain Size (μm) of pebbles Sintered at		
		900°C	1000°C	1100°C
1	1	1.41	1.22	8.95
2	5	1.76	1.43	10.16
3	10	2.04	3.36	21.20
4	15	2.08	3.94	24.29
5	20	2.18	4.71	-

From the analysis it is observed that the grain size of the pebbles increases with time and temperature for all sintering temperatures. At 1100 deg C it is drastically increases when the sintering times goes up which is basically occurs due to material getting highly crystalline and this is observed in X-ray analysis as well. Fig. 3 shows the micrograph of pebble sintered at 1000 deg C in different time.

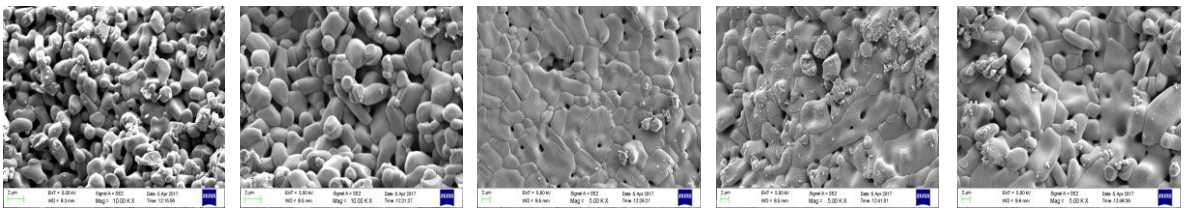

Fig. 3. SEM image of Li_2TiO_3 Pebbles Sintered at 1000°C for (a) 1 Hour, (b) 5 Hours, (c) 10 Hours, (d) 15 Hours and (e) 20 Hours.

3.4. Density Measurement

The green pebbles sintered at 1000°C for different time intervals were carried out its hardness under room temperature with certain load. From the SEM image, it was observed that the powder sintered at 900°C was containing very less interparticle bonding and large number of pores and pebbles sintered at 1100°C was containing very high interparticle bonding with very less amount of pores. Therefore the sintering temperature

was set as 1000 deg C with different time for density measurement. The density measurement was carried out in different trials and the data is tabulated in table 3. It was observed that the density of the pebbles increased with increase in sintering time. From the results the graph of density (gm/cm^3) v/s sintering time (h) was plotted to see the sintering behavior as shown in Fig. 4. In this graph the plot of the density was observed increasing when sintering time increases. This is because of the interparticle bonding increases at higher temperature with increasing time which is also observed in SEM analysis. As the interparticle bonding increases, the grain boundary increases with decreases the number of pores. This will cause the denser the material with less number of pores.

TABLE 3. Average density of sintered pebbles by helium pycnometer

Sr. No.	Sintering Temp. (°C)	Sintering Time (Hr)	Density (gm/cm^3) Trial 1	Trial 2	Trial 3	Average Density (gm/cm^3)
1.	1000	1	3.2554	3.1608	3.0489	3.1550
2.	1000	5	3.2602	3.3561	2.9061	3.1741
3.	1000	10	3.2828	3.1522	3.2487	3.2279
4.	1000	15	3.2970	3.3156	3.3015	3.3047
5.	1000	20	3.2740	3.3846	3.3876	3.3487

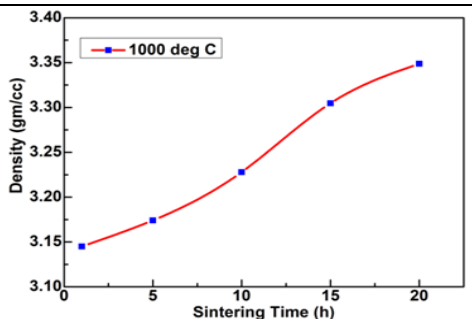

Fig. 4. Density Vs sintering time.

3.5. Porosity by Mercury Porosimeter

From the SEM analysis it's observed that the grain size is getting increased with high temperature sintering as on increasing the time. This leads to decrease the porosity of the material. As the material needs sufficient porosity for tritium breeding which is the main task of this particular material, so we have used 1000 deg C sintered material with different time for the present porosity studies. From the data tabulated in table 4. its observed that the porosity is getting decreased as the time increases which is expected based on the SEM and density studies. Both density and porosity are interrelated as the porosity decreases density increases and the grain boundaries plays a major role over here. These are all related to the temperature and time period of the sintering. So sintering time and temperature is important factor for getting best quality pebbles for better performance mainly for tritium production.

TABLE 4. Percentage porosity of sintered pebbles by mercury porosimeter

Sr. No.	Sintering Temp. (°C)	Sintering Time (h)	Bulk Density (gm/cm^3)	Apparent Density (gm/cm^3)	%Porosity
1.	1000	1	2.76	3.35	19.53
2.	1000	5	2.80	3.18	18.36
3.	1000	10	2.89	3.16	15.74
4.	1000	15	3.12	3.46	9.030
5.	1000	20	3.12	3.45	9.030

3.6. Crush Load

Sintering leads material becomes harder with time so that material will be able to sustain different loads based on the heat treatment with different temperature and time. In the present study we have performed the crush load at room temperature for the pebbles sintered at 1000 deg C in different time. The data is tabulated in Table 5. From the data it is observed that the pebbles getting harder and harder as we go on sintering for longer period. In each set minimum 50 samples of size 1.00 ± 0.15 mm has been used for the analysis. The crush load

strength is initially increasing and get gradually stabilized. But in all set of experiment it is observed that the pebbles having smaller in dimension provide very less crush strength. As we are unable to maintain the standard 1mm and it is potato shaped as well rather than the round shape there will be a minor variation can occur. But the average trend shows very good crush strength of the sample which is better than the required 20 N.

TABLE 5. Average crush load strength of sintered pebbles

Sr. No.	Sintering		Maximum	Minimum	Mean
	Temp. (°C)	Time (h)			
1	1000	1	85.7	16.6	41.2
2	1000	5	100.3	17.6	52.9
3	1000	10	110.6	10.2	52.4
4	1000	15	104.3	22.3	47.2
5	1000	20	89.1	16.7	47.2

3.7. Uniaxial Compression Tests

Table 6. contains the characteristic data of Li_2TiO_3 pebbles used for the present pebble bed experiment. The desired packing fraction for the present experiment has been achieved by using an external vibrator as well as tapping at the walls of the sample holder, during experiment a pre-load of 100 N has been applied and calculated the exact packing fraction based on the piston levels. Pebbles with uniform size and shape were in the category of mono-dispersed pebble bed and in our case we have used certain size distribution which is around 0.8 mm to 1.2 mm and will be considered ~ 1 mm pebble of uniform size which is in the category of mono-sized pebble bed. As per the literature the present pebble bed distribution of 0.8 mm to 1.2 mm can achieve around ~65% for the face centred cubic array [11]. In the present study we have achieved ~ 63% of initial packing fraction in our all experiments as like the earlier report [15].

TABLE 6. Characteristic of the pebble used for the pebble bed experiment

No.	Material	Size (mm)	Density (g/cc)	Porosity (%)	Average crush load (N)
1.	Li_2TiO_3	0.8 – 1.2	2.92	~15	38.6 at RT 36.4 at (640 ^oC)

The stress-strain behaviour of Li_2TiO_3 pebble bed at high temperature and pressure has been shown in Fig. 5, in which it is clearly visible that the axial strain value represents the volume reduction of pebble bed once the cycle (loading and unloading) is completed. One can observe the maximum reduction is happening with in the first few cycles and the first pressure increase and decrease curve emphasize plastic deformation of the material which mainly because of the irreversible nature of the material and leads to elastic deformation in the forthcoming cycles. Fig. 6, clearly depicts the bed volume reduction based on continuous cycles in different pressure and temperature of lithium titanate at a rate of 1 MPa/min. This clears the pattern of gap formation may occur during the blanket operation. But the gap formation may get increased once the rate of loading has been increased even after reaching saturation at a particular loading pattern.

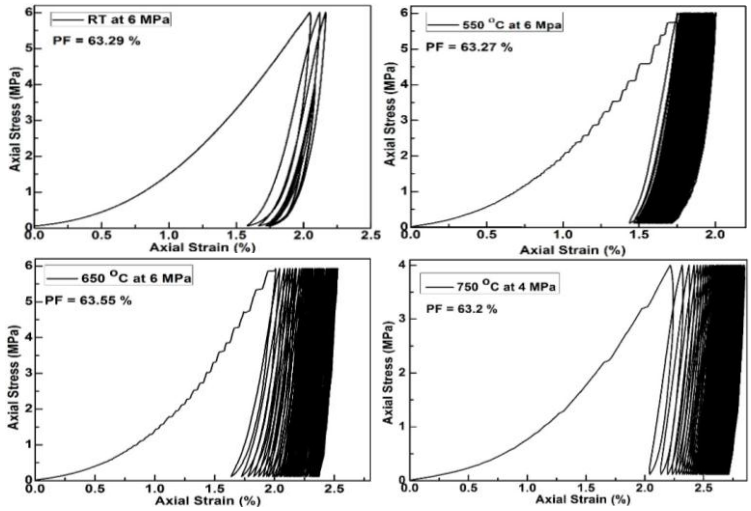

Fig. 5. Axial stress-strain loop of Li_2TiO_3 pebbles
at different temperature and pressure

At temperature below 650 ^{o}C creep doesn't have any role in material like lithium titanate and helps the bed to saturate very easily in few cycles and it's visible in Fig. 6. There is only a minor increment in the strain rate that has been observed in 500 ^{o}C bed at 6 MPa when it compared with first cycle which is of 1.5 % and leads to a further change of ~0.25% reduction in the bed volume at the 50th cycle of the experiment. This leads the pebble bed to saturate in a way that it had stopped further deformation at the 50th cycle and make stable its own properties during reactor operation. But at 650 & 750 ^{o}C there is much more reduction in the volume which is near to ~0.75 % of the pebble bed while comparing with the first cycles, here the higher pressure also causes more creep deformation in higher temperature and will take much more cycle to saturate the bed.

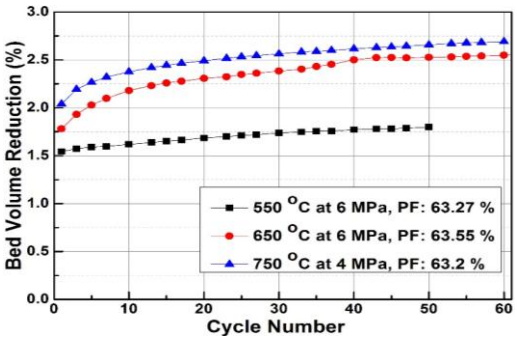

Fig. 6. Bed volume reduction of Li_2TiO_3 pebble bed at different temperature and pressure

3.8. Temperature Dependent Nonlinear Modulus of Pebble Bed

The isotropic non-linear elasticity, defined by Eq. (1), is used to describe pebble bed elastic behaviour (16-19). The Young's modulus is a function of two stress invariants, I_1 and J_2, and temperature, T. The material parameters of A_e(T) and s can be identified by experimental data. The required experimental data has been obtained from the uniaxial compaction pebble bed experiment. Pebble bed modulus has been calculated from the unloading path of the first cycle of the stress-strain relation graph at elevated temperature and pressure. For the case of uniaxial compaction test, the modulus equation can be reformed as the followings (Eqs. 2-3).

$$E = A_e[(1+\nu)J_2 + \frac{1-\nu}{3}I_1^2]^{s/2} + E_0 \quad (1)$$

$$E = E_0 + A_e\left[\frac{2(1-\nu)}{(1+\nu)(1-2\nu)}\right]^{-s/2}(\sigma_y)^s \quad (2)$$

$$\sigma_y = \frac{1-\nu}{(1+\nu)(1-2\nu)}E\varepsilon_y \quad (3)$$

$$E = \frac{(1+\nu)(1-2\nu)}{(1-\nu)}.\frac{d\sigma y}{d\varepsilon y}$$

Where ν = 0.25 for the example case of Li_2TiO_3 pebble bed.

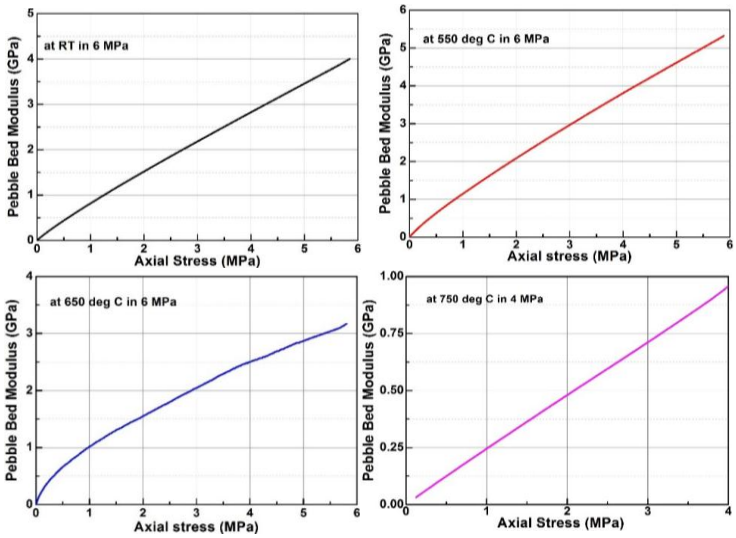

Fig. 7. Pebble bed modulus of Li_2TiO_3 pebbles.

The equation resolve the Young's modulus of pebble bed is shown in Fig. 7. Temperature rise reduced the bed modulus at higher temperatures during the experiment at 550 to 750 ^{o}C. However, pebbles will be more packed under higher temperature and the same peak load, which will increase the bed modulus. Based on the data, apparently temperature plays a key role for the modulus and the packing status of pebble bed.

3.9. Creep test

Creep test has been performed in the same experimental setup where stress-strain measurement has been carried out and shown in Fig. 8. At temperature 650 deg C in 4 MPa and 750 deg C in 2 MPa the material gradually set after few hours of experiment and observed a stabilized graph with minimum creep strain percentage. The stabilization shows the material will not enter into further deformation and the properties of the pebble bed will not be altered further. But at 750 deg C in 4 MPa shows the material is not getting stabilized even after 6-8 hours, which is completely away from the other two data plots. The material with varying creep strain doesn't seems to be good for fusion reactor material, so its suggested to be use this material up to 750 °C, 2 MPa.

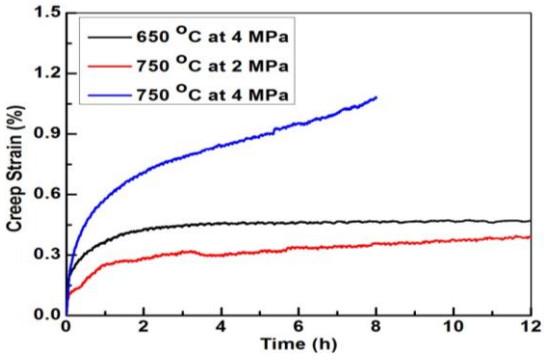

Fig. 8. Creep strain vs time of Li_2TiO_3 pebble bed.

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