



Chemical characterization of lithium based ceramics utilizing charged particle activation and ion beam techniques

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Abstract

Non-destructive methodologies using activation analysis and ion beam analysis techniques were optimized for the chemical characterization of ceramic materials, lithium titanate and lithium niobate, which have application in tritium breeding blanket. The analyses were carried out as a part of chemical quality control exercise. The atomic ratios of Li/Ti, Li/Nb were quantified by charged particle activation analysis using 13 MeV proton beam from variable energy cyclotron facility and particle induced gamma ray emission/Rutherford backscattering spectrometry using 3 MeV/2 MeV proton beam from 3MV tandem accelerator facility. The results of these different analytical methods are in good agreement, which established the applicability of these activation analysis and ion beam techniques for the chemical characterization of these ceramic materials.

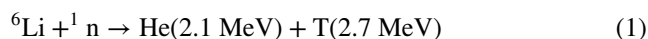
Keywords Lithium titanate · Lithium niobate · CPAA · PIGE · RBS

Introduction

Necessity and problems of tritium production

The fast exhausting stock of energy resources and environmental concerns across the world has seen the attention in energy research shift its direction to alternative and green energy sources in recent years. The possibility of utilizing Nuclear Fusion reaction as a long serving energy source has seen encouraging technological and scientific developments in the past few years. With the continuous research in the fields of sustenance of the plasma core of a fusion reactor and controlling the fusion reaction—the International Thermo-nuclear experimental reactor (ITER) is nearing the reality. The deuterium–tritium (D-T) fusion is the most

feasible (from energy requirement point of view) [1] reaction and is to be used to fuel the ITER. It has the added advantages of its minimal environmental impact and absence of radioactive products, thereby eliminating the allied radioactive waste management processes involved and radiation exposure as well. Deuterium is present in sufficient amount in nature and can be easily extracted. Owing to the extremely low amount of Tritium present in the environment; its production becomes a vital issue for fuelling the fusion reactor. Lithium (Li) undergoes a nuclear reaction with neutrons (Eq. 1) to produce Tritium [2]. As a result, fusion reactors having tritium breeding ratio, (TBR) > 1, using of lithium containing blankets with neutrons multipliers are subjected to extensive study. Lithium containing materials have been subjected to a lot of studies for their properties. High melting lithium containing ceramic materials have attracted much attention in this regard. In addition to this, TBR have been improved using neutron multiplier such as beryllium and lead, in lithium blanket materials [3, 4]. A series of Li containing blanket materials were synthesized and tested for the principle breeding requirements in fusion reactor as tritium breeder.



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Possible target materials for tritium production

The probable Li-based (also enriched in ^6Li) ceramics candidates for application as tritium breeder, are lithium titanate (Li_2TiO_3) [5], lithium-zirconate (Li_2ZrO_3) [6, 7], lithium silicate (Li_2SiO_3) [8], lithium niobate (LiNbO_3) etc. In addition, the lithium titanate-zirconate ($\text{Li}_{2+x}(\text{Ti},\text{Zr})\text{O}_{3+z}$) has been reported recently as a super advanced (SA) tritium breeder material [9]. Some of these tritium breeder materials have also been tested in ITER test blanket module (TBM) and for DEMO reactor [10, 11]. As a result the analytical study of the detailed composition and nature of the probable fuel materials to be used is of major importance. In the proposed fusion reactor, a mixture of (80% Ar and 20% H_2) sweep gas will be used to extract the generated tritium from the breeder blankets. In the sweep gas atmosphere lithium titanate has been found to show mass loss due to Li vaporization and reduction of Ti^{+4} to Ti^{+3} associated with oxygen (O) loss [12, 13]. Such processes render the fuel pellets brittle and also change the thermal diffusivity properties. To prevent the mass loss and conserve the physical and chemical properties of the fuel at elevated temperatures, excess Li addition to the fuel has been suggested [12, 14, 15].

Analytical methods to characterize the breeding blankets

Several methodologies for the synthesis of ceramic breeder materials were developed such as Sol-gel synthesis [16, 17], solid state synthesis [18], solution based methods [8, 18, 19] etc. The ratio of Li/M (M-metal) may vary depending on the use of above methods and this in turn affects the TBR and other properties of the breeding material [20]. Therefore the chemical analysis of the above synthetic ceramic materials for ensuring chemical quality control, is of utmost importance. Unfortunately, the compositional analysis of ceramic materials is quite difficult, especially by employing conventional solution based methods as these materials are difficult to dissolve due to their refractory nature. The conventional analysis methods of chemical constituents for ceramics are long, tedious and sometimes do not enable for both the elements as reported here [21–27]. Analysis using laser ablation ICP-MS [28], LIBS (laser induced breakdown spectroscopy) can be utilized for the solid state analysis, although matrix interference can cause major problems. Though quantification of titanium (Ti) and niobium (Nb) are possible with instrumental neutron activation analysis (INAA), particle induced X-ray emission (PIXE), X-ray fluorescence technique (XRF), they are unsuitable to

be used to quantify Li. In recent times there have been reports of non-destructive and simultaneous analysis of chemical compositions of ceramic samples by NAA, PIGE [29–32]. Non-destructive nuclear techniques such as particle induced gamma ray emission (PIGE) and charged particle activation analysis (CPAA) can however quantify lithium with high precision. PIGE [32] and Proton Rutherford backscattering spectrometry (p-RBS) has been reported to have been successfully employed for simultaneous determination of lithium and titanium in lithium titanate. Charged particle activation analysis (CPAA) is a simultaneous multi-elemental determination technique, similar to NAA. CPAA has widely been used for the determination of several elements in trace and ultra trace levels [33–36]. There are very few reports for the determination of elements in ceramic samples using CPAA [37]. During the present work, lithium titanate and lithium niobate samples were analyzed by CPAA techniques using 13 MeV proton beam from K-130 cyclotron at Variable Energy Cyclotron Centre (VECC), Kolkata, India. The proficiency of CPAA in compositional analysis of technologically important ceramic compounds is presented. The same samples were also analyzed by particle induced gamma ray emission (PIGE) and Proton Rutherford backscattering spectrometry (p-RBS) [38, 39] using 3 MV tandem accelerator machine from National Centre for Compositional Characterisation of Materials (NCCCM), Hyderabad, India for validation purposes.

Experimental

In CPAA, the selection of proton energy depends upon the type of nuclear reaction to be used to produce the desired isotope from the target element. The (p, n) reaction channel is widely used for determination of trace elements by instrumental approach in CPAA due to high cross section, less nuclear interference and low matrix activation. The nuclear reactions used to quantify the elements of interest in this CPAA work were— $^{\text{nat}}\text{Li}(p, n)^7\text{Be}$; $^{\text{nat}}\text{Ti}(p, n)^{48}\text{V}$ and $^{\text{nat}}\text{Nb}(p, n)^{93\text{m}}\text{Mo}$. Available literature shows that the $^{\text{nat}}\text{Li}(p, n)^7\text{Be}$ reaction is having cross section 0.03–0.5 b in the 2.2–13 MeV energy range of proton with $\sigma_{\text{max}} = 0.5$ b at 2.3 MeV [40]. The reaction cross sections for $^{\text{nat}}\text{Ti}(p, n)^{48}\text{V}$ and $^{\text{nat}}\text{Nb}(p, n)^{93}\text{Mo}$ reactions have σ_{max} of 63 mb [40–42] and 30 mb [40, 43–45] at ~11 MeV respectively. There was a significant interference of nitrogen (N) on the determination of Li using proton beam of energy 14 MeV and above [37]. Moreover, the higher reaction channels, e.g., (p, 2n), (p, pn) will start competing with (p, n) reaction channel at 15.6, 11.9 MeV and 9.4, 9.0 MeV respectively for the afore-mentioned ^{48}Ti and ^{93}Nb isotopes [46]. Thus, considering the interferences as mentioned above, a proton

beam of 13 MeV was chosen to carry out the this CPAA work.

The lithium titanate and the lithium niobate samples were prepared at R&D laboratories of Bhabha Atomic Research Centre (BARC), Mumbai [47]. The minimum amount of sample required to make pellets of samples (in quadruplicate) and standards were calculated from the ranges of 13 MeV proton beam in the above two matrices respectively. For the Li/M ratio analysis of these samples, a number of comparators were used as standards, e.g., (1) titanium oxide (TiO_2 , Sigma Aldrich) and lithium titanate, (Li_2TiO_3 , Sigma Aldrich) for the lithium titanate sample, (2) niobium pentoxide, (Nb_2O_5 , Sigma Aldrich) and lithium niobate, (LiNbO_3 , Sigma Aldrich) for the lithium niobate sample. The ranges of 13 MeV proton beam in stoichiometric TiO_2 , Li_2TiO_3 , Nb_2O_5 , and LiNbO_3 are 637.7, 1570.0, 683.7, 651.9 μm , respectively computed from the range-energy table of SRIM—2008 [48]. The above comparators (~300 mg) and samples in pebbles form were first crushed into powder and then ground thoroughly in a mortar-pestle for 6 h to obtain a fine powder. Finally pellets of 10 mm dia. and 1.5–2.5 mm thickness were prepared using automatic hydraulic press. The pellets of samples and comparators were irradiated with 13 MeV proton beam using ~450–800 nA beam current for 15–30 min at VECC, Kolkata (Fig. 1).

The detailed experimental setup of irradiation was described in our earlier work [49]. The radioactivity measurements were carried out offline with a high resolution

γ -ray spectrometer using high purity germanium (HPGe) detector (relative efficiency: 40%) coupled to PC based multi channel analyzer. Details of the nuclear parameters used in the CPAA work are summarized in Table 1 [50, 51].

For PIGE and RBS analysis pellets were prepared by thoroughly mixing the respective samples and/or standards with graphite in a 3:1 ratio (w/w) in a mortar-pestle for 12 h. Polyvinyl alcohol was then added to the mixtures, mixed well and the mixtures were dried under an IR lamp for 5–10 min. These mixtures were then pelletized using a manual pelletizer. The pellets so prepared were of 20 mm diameter. For lithium titanate samples analysis, standard Li_2TiO_3 , Sigma Aldrich was used as comparator and for lithium niobate samples standard LiNbO_3 , Sigma Aldrich was used as comparator. The targets in the form of pellets were irradiated with 3 MeV and 2 MeV proton beams for PIGE and proton beam RBS measurements respectively. The samples were put in the scattering chamber using a stainless steel (SS) ladder arrangement. A detailed discussion of the arrangement can be found in literature [38, 39, 52]. The vacuum inside the sample chamber was better than 5×10^{-6} torr. A standard gold (Au) foil was used for the channel vs. energy calibration of the detector in the RBS experiment. The radioactivity measurements were carried out with a high resolution γ -spectrometer using high purity germanium (HPGe) detector (relative efficiency: 40%) coupled to PC based multi channel analyzer placed at 180° with respect to the beam direction for the PIGE work. The

Fig. 1 Typical snapshot of **a** lithium titanate and **b** lithium niobate sample

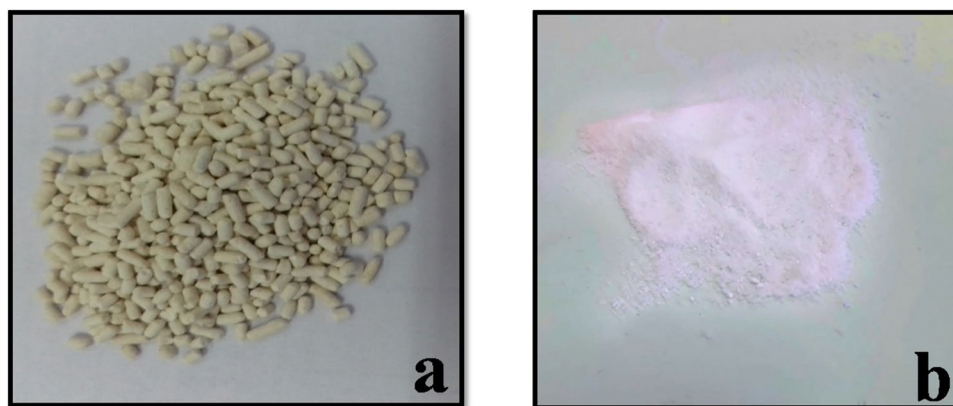


Table 1 Relevant radioisotope products, their nuclear properties and reaction energetics for the CPAA work

Element	Target isotope (abundance, %)	Nuclear reaction; Eth, Threshold energy, MeV; Ec, Coulomb barrier, MeV	Product isotope	Half-life	γ -ray (keV) (intensity, %)
Li	^7Li (92.5)	(<i>p, n</i>); $E_{\text{th}} = 1.88$; $E_{\text{c}} = 0.98$	^7Be	53.4 d	477.6 (10.4)*
Ti	^{48}Ti (73.7)	(<i>p, n</i>); $E_{\text{th}} = 4.89$; $E_{\text{c}} = 4.55$ MeV	^{48}V	15.97 d	983.5 (99.9)*, 1312.1 (98.2), 944 (7.9)
Nb	^{93}Nb (100)	(<i>p, n</i>); $E_{\text{th}} = 4.41$; $E_{\text{c}} = 7.11$ MeV	$^{93\text{m}}\text{Mo}$	6.9 h	1477.1 (99.1)*, 684.7 (99.9), 263.0(57.4)

* γ -rays of isotopes used for this analysis

measurements of the backscattered particles were carried out using a silicon surface barrier detector placed at 165° with respect to the beam direction.

Result and discussion

The offline gamma ray spectra of representative proton irradiated lithium titanate and lithium niobate samples from CPAA are shown in Fig. 2. The gamma ray spectrum of lithium niobate is quite clean as Nb is mono-isotopic. Only long-lived radioactive isotope ^7Be (53.4 d) is produced from lithium and all other isotopes produced are short lived (< 20 min). In the case of lithium titanate sample, titanium has several natural stable isotopes and out of all these ^{48}Ti only produces the desired radioactive isotope ^{48}V via (p, n) reaction. The proton energy of 13 MeV was selected such that, the other isotopes of Ti, with abundance in the range 5.3–8.0% do not produce any significant long lived gamma emitting products. Therefore the matrix activation was kept at a minimum in both samples of lithium titanate and lithium niobate and after few days the gamma spectra contained only gamma lines from radioactive isotopes of our interest. Thus the instrumental method of quantification of major elements Li, Ti and Nb in their respective compounds by CPAA technique is relatively simple and fast. The quantitative estimation has been done by the comparator method [49] due to its simplicity and convenience with respect to other techniques.

In this work, CPAA results were compared with the simple, rapid and non-destructive PIGE approach using 3 MeV protons for the determination of atomic composition of lithium titanate. The determination of Li is based on the detection of 478 keV γ -rays while that of Ti on the detection of 981 keV γ -rays from $^7\text{Li}(p, p'\gamma)^7\text{Li}$ and $^{48}\text{Ti}(p, p'\gamma)^{48}\text{Ti}$ reactions respectively. Proton irradiated gamma-ray spectrum of lithium titanate using 3 MeV proton beam in PIGE method, is shown in Fig. 3. The determination of oxygen

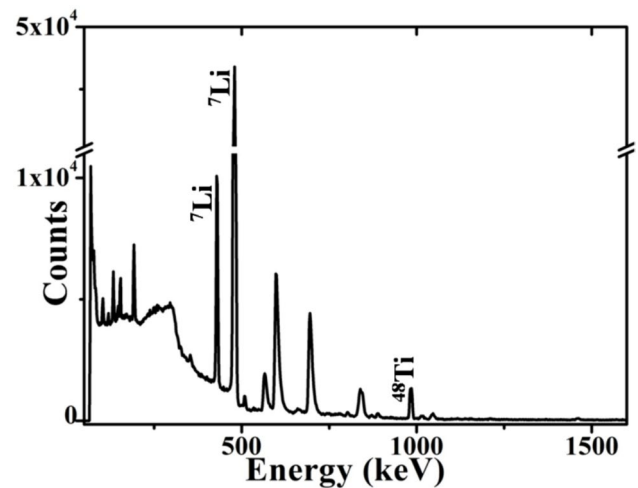


Fig. 3 Proton induced gamma-ray spectrum of thick lithium titanate sample using 3 MeV proton beam in PIGE method

was not suitable in this energy range of proton, because thick target yields of 495 and 871 keV gamma-rays from $^{16}\text{O}(p, \gamma)^{17}\text{F}$ and $^{17}\text{O}(p, p'\gamma)^{17}\text{O}$ respectively, are low and require more than 6.1 MeV of proton resulting in 6129 keV γ -rays from $^{16}\text{O}(p, p'\gamma)^{16}\text{O}$ [31]. The atomic ratio of Li-to-Ti from the γ -rays spectrum was determined from the area under the respective peaks using stoichiometric lithium titanate as comparator. The atomic compositions ($C_{\text{Li}}:C_{\text{M}}$, M-Ti), along with their uncertainties of the sample lithium titanate thus determined are given in Table 2.

The determination of Nb could not be possible in the same way using 3 MeV protons by PIGE method as the yield of Nb is undetected/non-sensitive in this energy domain. The energy of the proton beam could not be increased further because of the limitations of the tandem accelerator itself. Therefore, proton Rutherford backscattering spectrometry (p-RBS) was employed for the determination of the atomic ratio of Li-to-Nb in lithium niobate through the $^7\text{Li}(p, p')^7\text{Li}$

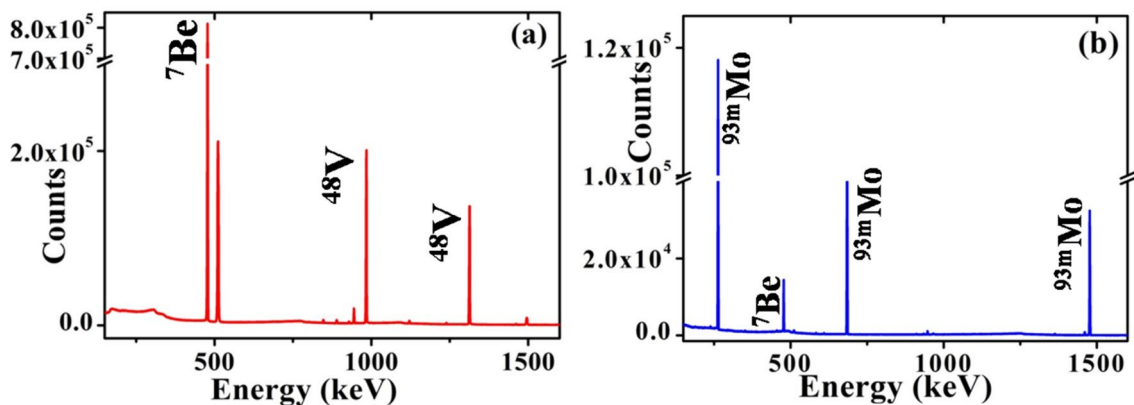
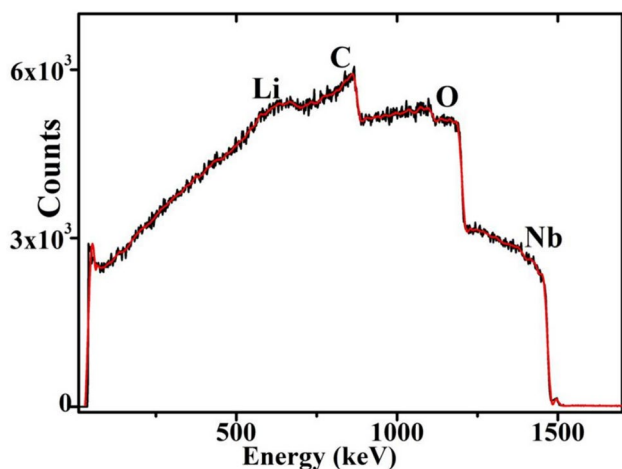


Fig. 2 Gamma ray spectra of a representative 13 MeV proton irradiated a lithium titanate and b lithium niobate sample

Table 2 The atomic compositions of ($C_{Li}:C_M$, M-Ti, Nb) in lithium titanate and lithium niobate ceramics samples (uncertainties associated with ratio measurements are given in parenthesis) by CPAA, PIGE and RBS

Sample			$(C_{Li}:C_M)$		
			CPAA	PIGE	RBS
Lithium titanate	Batch 1	Value	2.11(± 0.02):1.01(± 0.01)	2.10(± 0.03):1.02(± 0.02)	–
	Batch 2	Value	2.28(± 0.01):1.02(± 0.01)	2.30:1.03	–
Lithium niobate		Value	1.00(± 0.01):1.01(± 0.02)	–	1.02(± 0.02):1.00(± 0.01)

**Fig. 4** 2 MeV proton backscattered spectrum of a thick lithium niobate sample. The graph in red is the fitted curve

and $^{93}\text{Nb}(p, p')^{93}\text{Nb}$ nuclear scattering reactions. Figure 4 shows a typical p-RBS spectrum of lithium niobate sample (red line represents the fitted curve). It consists of prominent steps of Nb and Li and a rather weak step of oxygen (O). The signal of Nb is largely due to Rutherford backscattering while the signals of Li and O arise from the scattering reactions of $^6\text{Li}(p, p)^6\text{Li}$, $^7\text{Li}(p, p)^7\text{Li}$ and $^{16}\text{O}(p, p)^{16}\text{O}$, respectively. The spectrum also consists of an overlapping step of C which is the signal from $^{12}\text{C}(p, p)^{12}\text{C}$ reaction. The atomic ratio of Li-to-Nb from the backscattered spectra was determined by taking the step heights of Li and Nb into consideration. The atomic compositions ($C_{Li}:C_M$, M-Nb), along with their uncertainties of the sample lithium niobate thus determined are given in Table 2.

The Li:Ti ratio of lithium titanate sample was found to be in the range of 2.11 ± 0.02 (Batch# 1) to 2.28 ± 0.01 (Batch#2) using CPAA technique, whereas the same using PIGE technique was found to be in the range of 2.10 ± 0.03 (Batch# 1) to 2.30 ± 0.02 (Batch#2). The above results are found to be in good agreement with the expected atomic

ratios. The Li:Nb ratio was found to be $1.00(\pm 0.01)$ and $1.02(\pm 0.02)$ by CPAA and RBS techniques, respectively. There are reports which suggest that the tritium breeding lithium titanates may have Li:Ti in the range of 1.8–2.2 [9, 12, 20], 2.15 [9], 2.2 [12] to be successfully employed in ITER. Again the excess of Li present in these samples, especially in a neutron environment is important considering good tritium release behaviour, mechanical and chemical stability of the pebbles under the reactor operating conditions. The probable mechanism of Li loss, other than the nuclear reaction that produces tritium, may be in the form of LiOH (g) at elevated temperatures.

Conclusion

For the first time, the CPAA method using 13 MeV proton beam has been successfully applied for the determination of atomic ratio of metals in lithium titanate and lithium niobate ceramics. The analyses of the same ceramic samples were carried out using PIGE and p-RBS methodology to validate our established CPAA method. Thus the capability of CPAA in quantifying the relative stoichiometry of Li:M (M-metal) in refractory materials is established. Although we could not determine oxygen in this work due to absence of suitable offline gamma-rays, this could easily be done in an online measurement as discussed in the result and discussion section. Being a simple, free from blank and non-destructive method, similar to other nuclear analytical methods like INAA, PIGE, etc., CPAA can be used for compositional characterization of ceramic tritium breeders in its instrumental approach. These results may be useful in synthesising the targeted tritium breeding blanket materials and other designing related to a shielded facility for tritium release studies.

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Data availability Data will be made available on request to the corresponding author.

Declarations

Conflict of interest It is declared that, there is no known conflict of interest regarding the work reported in this manuscript.

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