



Development of a reflectron time-of-flight mass spectrometer for non-destructive analysis of isotope ratios in irradiated B₄C pellets—Test measurements on an unirradiated control rod pellet

P. Manoravi, M. Joseph*, N. Sivakumar

Fuel Chemistry Division, Chemistry Group, Indira Gandhi Centre for Atomic Research, Kalpakkam 603102, India

ARTICLE INFO

Article history:

Received 12 March 2008
Received in revised form 12 June 2008
Accepted 13 June 2008
Available online 27 June 2008

Keywords:

Reflectron time-of-flight mass spectrometer
Isotopic ratio in B₄C
Laser-mass spectrometry

ABSTRACT

A laser mass spectrometric facility is developed using a home-built reflectron time-of-flight mass spectrometer (RTOFMS) to analyze the boron isotopic ratio ¹⁰B/¹¹B in the irradiated B₄C pellets of the FBTR control rod. Compared to other mass spectrometry-based methods, the present method is practically non-destructive and makes it relatively easier to handle irradiated (radioactive) B₄C pellets through remote operation. The results with inactive samples indicate that the method yields ¹⁰B percentage values, accurate to within ±1%.

© 2008 Elsevier B.V. All rights reserved.

1. Introduction

Boron carbide (B₄C) is widely used as a control rod material in fast nuclear reactors. The absorption cross-sections of ¹⁰B and ¹¹B for thermal neutrons are 3837 b and 5 mb, respectively, while the values for fast neutrons (corresponding to the neutron energy spectrum seen in a fast reactor), are about 2.7 b for ¹⁰B and a much smaller value (9.7×10^{-5} b) for ¹¹B [1]. The natural abundances of B isotopes are 19.89% of ¹⁰B and 80.11% of ¹¹B [2]. Hence, B₄C pellets enriched in ¹⁰B are used in the fabrication of control rods and the extent of enrichment varies from 60%, for a commercial fast reactor, to as high as 90% for small core test reactors. For the Indian prototype fast breeder reactor (PFBR) [3], it is proposed to use nine control safety rods (CSR) and three diverse safety rods (DSR) and both will have B₄C pellets containing about 65% enriched ¹⁰B. In the case of fast breeder test reactor (FBTR), which has a small core, the extent of ¹⁰B enrichment used is 90% [4]. Generally, the B₄C pellets used in the control rods of fast reactors can be ≈10–40 mm in diameter and up to 50 mm in length. The typical dimensions of control rod pellets used in FBTR are diameter: 38 ± 0.15 mm, length: 40 ± 0.15 mm. The density is 2.25 g/cm³. For the PFBR the B₄C pellets dimensions will be diameter: ~17.4 mm, length: ~20 mm and the density 2.21 g/cm³.

The present laser-mass spectrometric facility is developed with the main objective of measuring the ¹⁰B/¹¹B ratio in the irradiated pellets of FBTR control rods. Being practically a non-destructive method, the pellets which have undergone only very small changes in the ¹⁰B/¹¹B ratio can be reused (after the analysis) for fabrication of new control rods. This is useful since the highly enriched ¹⁰B (90%) is very expensive.

Fabrication of B₄C pellets is an energy intensive process and B₄C is one of the hardest materials known, next to diamond and cubic boron nitride. Any destructive method to analyze isotope ratio of B in B₄C is cumbersome. Hence, a non-destructive method is preferred not only to avoid the difficult dissolution or powdering process, but also because the pellet taken for analysis can be reused after the measurement.

There are nine B₄C pellets stacked in one rod. The ¹⁰B present in B₄C pellets of a control rod is burnt out only to a small extent, as the rod is kept out of active core level for a considerable part of its life in the reactor and the maximum burn-up of ¹⁰B is seen only in the lower part of the control rod pin. Even though the enriched isotope content might be good enough to be used for a longer period of time, these control rods will need to be replaced periodically depending on the cladding stability. During such periodic removal of the control rods, it is necessary to examine whether the pellets from such irradiated pins can be reused or relocated; i.e., one needs to know the value of ¹⁰B/¹¹B ratio in the irradiated pellet. The accuracy needed for such isotope measurements is about ±1%. Though neutron-irradiated B₄C is not expected to be radioactive, some common trace impurities such as Eu present in the B₄C pellet can lead to

* Corresponding author. Tel.: +91 44 2748 0098; fax: +91 44 2748 0065.
E-mail address: mj@igcar.ernet.in (M. Joseph).

Table 1a
Typical chemical analysis of the B₄C pellet used in FBTR control rods [6]

Element	Amount (wt.%)
B	75.3
C	24 ± 0.9
O	0.29
N	0.18
Fe	0.5
Mg	0.14
Ca	0.79
K	0.07

considerable radioactivity due to neutron activation [5]. The results of a typical chemical analysis of the B₄C pellet used in FBTR are given in Table 1a [6] while that of neutron activation analysis are given in Table 1b [6]. Significant amount of Eu and Ta impurities present in the pellet make it important to have a suitable glove box arrangement for the analysis of irradiated B₄C pellets, if sufficient cooling period is not provided. Laser vaporization mass spectrometry (LMS) is a method that is amenable for remote operation and thus very suitable for ¹⁰B/¹¹B ratio measurements on irradiated B₄C pellets.

In LMS, the laser vaporization removes only a few nanograms of the material from the surface [7], without any physical damage to the pellet. For a nanosecond laser pulse, the transiently heated part of surface is typically ≈1 mm in diameter and a few tens or hundreds of Å depth. This paper gives results of such a laser mass spectrometric study on B₄C.

2. Experimental facility

We have developed in house a laser–RTOF mass spectrometry system. The experimental layout is shown in Fig. 1 and the schematic of the RTOFMS is shown in Fig. 2. The details of various components used in this facility including vacuum system, remote sample loading and unloading, glove box facility and the data collection system are discussed below.

The whole system, other than the detector chamber is pumped by a turbo molecular pump (TMP, 450 L/s, Electrorava, model ETP450G) and the detector chamber is pumped additionally by another TMP of 80 l/s capacity (M/s Electrorava, model ETP80G). Both these TMPs are backed by suitable two stages, direct drive mechanical rotary pumps of 30 m³/h (model 2033SD Alcatel) and 10 m³/h (model 2010SD Alcatel) capacity, respectively. The typical vacuum attained during experiment is ~3 × 10⁻⁷ mbar.

The sample chamber has many ports and view ports. The sample position is remotely seen using microcameras kept at two of the view ports of this chamber. One of the view ports is used to allow the laser beam to fall on the sample and a collar kept just above this chamber over the drift tube is used for positioning the focusing lens. This chamber also holds both the deflection plates; a feedthrough attached to this chamber is used to apply the voltages to these deflection plates and to the sample (through the sample holder).

All grids were made by laser welding of 90% transmission nickel mesh to 2-mm thick SS rings. Efforts were made to keep the mesh

Table 1b
Results of neutron activation analysis showing activation products of the impurities present in FBTR B₄C pellet [6]

Radionuclide	Specific activity (nCi)
¹⁵² Eu	62.8 ± 2
¹⁵⁴ Eu	9.0 ± 1.2
¹⁸² Ta	21.2 ± 1.7
⁶⁰ Co	0.6 ± 0.2
¹³⁷ Cs	0.54 ± 0.2

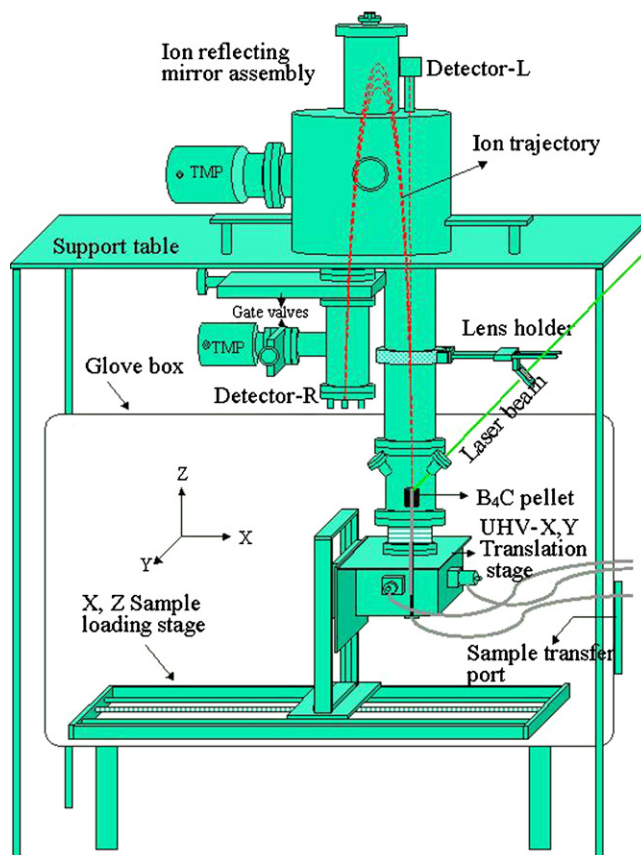


Fig. 1. Home-built reflectron time-of-flight mass spectrometer for the analysis of ¹⁰B/¹¹B ratio in FBTR control rod B₄C pellet.

flat. The grids used immediately after the sample are of 100 mm o.d. and 25 mm effective aperture. For the reflector region, the grids o.d. and aperture area are 125 mm and 75 mm, respectively. We have used nine guard rings (SS circular disks) of 2 mm thick, 125 mm o.d. and 75 mm i.d. The regions between the grids are separated by PTFE spacers. The resistor chain attached to the guard rings provides a homogeneous electric field for the reflector.

Fig. 3(a) shows the photograph of the sample loading mechanism used in set-up. The sample holding cup sits on the 10 mm diameter SS rod projecting out in the sample holding bellow assembly mounted on the X, Y translation stage (UHV compatible) for scanning the sample. This movement is remote controlled by two DC motors. This sample translation stage along with the bellow assembly is in turn held on a X, Z translation stage placed at the bottom of the glove box and its motion is controlled by two AC motors, that are remotely controlled. The sample (B₄C pellet of FBTR control rod) is placed in the sample cup made up of stainless steel, which in turn sits in a PTFE cup. Fig. 3(b) shows the sample as loaded in the bellow assembly.

The laser used in this study is a pulsed Nd-YAG laser (Model Continuum NY-60-10) having pulse width of 8 ns. For the present experiments, the 532 nm beam is used because of the ease of beam alignment. The pulse energy used was very low such that the power densities for the focussed beam are in the range of 10⁶ to 10⁷ W/cm². A quartz lens with a focal length of 50 cm was used for focusing the laser beam. Laser power density falling on the target surface was varied by adjusting the distance between the focusing lens and target.

The detection of ions is done using two types of detectors. First one is a simple secondary electron (SEM) multiplier (Model

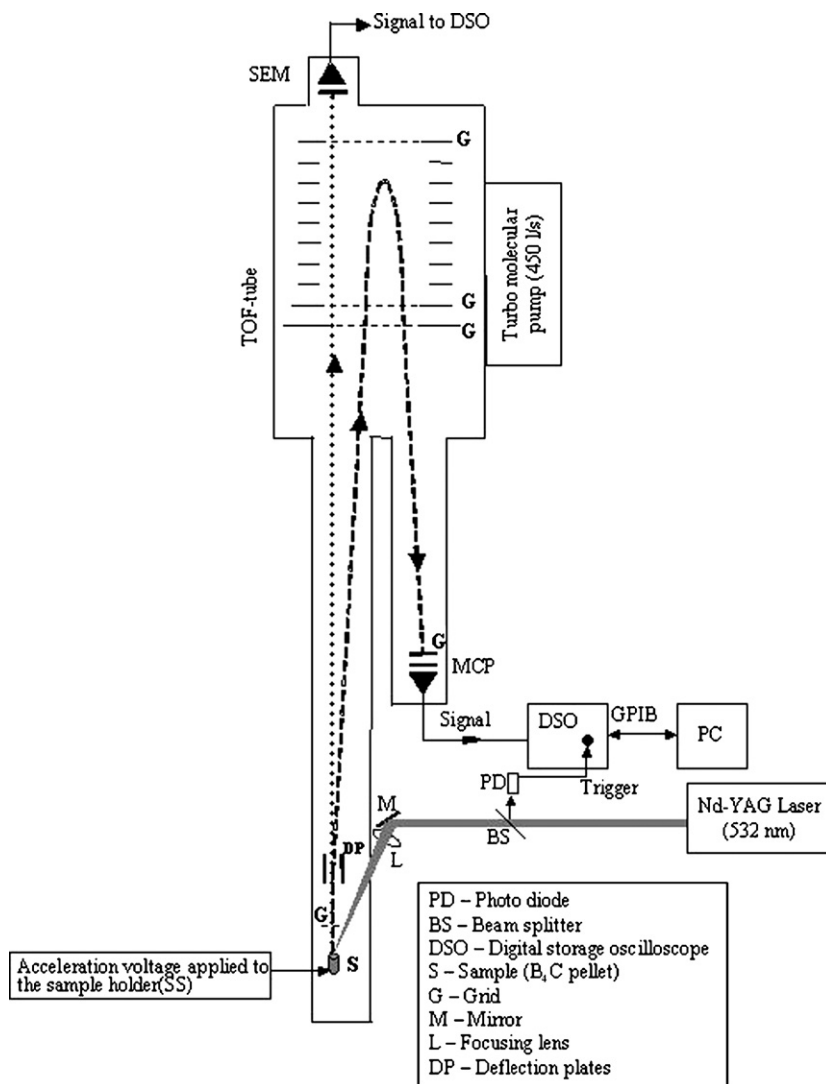


Fig. 2. Schematic of the homemade reflectron time-of-flight mass spectrometer (RTOFMS).

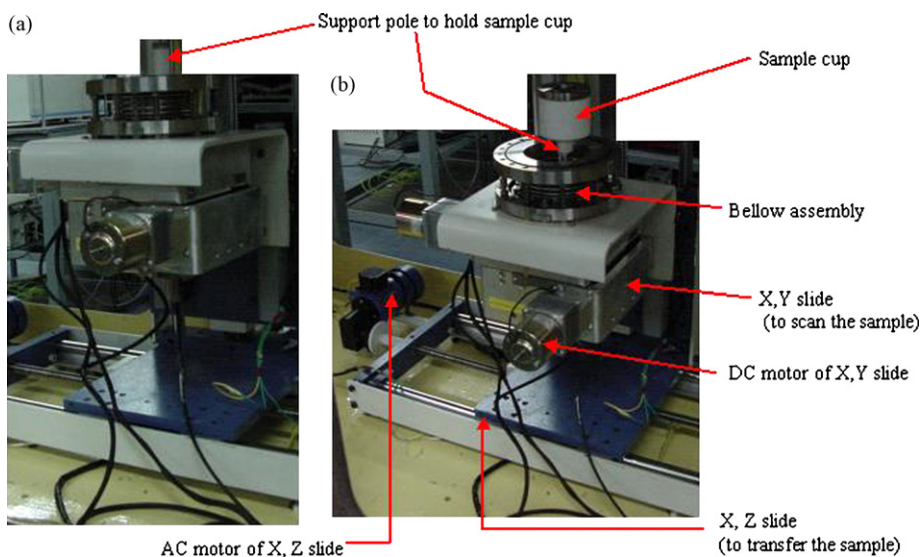


Fig. 3. The sample loading arrangement (including the X, Z stage for sample transport; and X, Y stage for sample scanning). (a) Without sample holder and (b) along with the sample holder.

Table 2
The ratios of ¹⁰B/¹¹B measured in terms of area under the peak and the enrichment calculated for a typical experiment using enriched B₄C pellet (FBTR sample)

S. No.	Area of ¹⁰ B peak (A ₁₀) (a.u.)	Area of ¹¹ B peak (A ₁₁) (a.u.)	Ratio of (A ₁₀ /A ₁₁)	% of ¹⁰ B in B ₄ C
1	4.397E-05	4.366E-06	10.07	90.97
2	2.299E-04	2.105E-05	10.92	91.61
3	1.644E-04	1.506E-05	10.91	91.61
4	1.425E-04	1.274E-05	11.19	91.80
5	1.026E-04	9.534E-06	10.76	91.50
6	9.654E-05	8.882E-06	10.87	91.57
7	9.394E-05	8.584E-06	10.94	91.63
8	8.546E-05	8.255E-06	10.35	91.19
9	7.685E-05	7.115E-06	10.80	91.53
10	7.609E-05	7.344E-06	10.36	91.20
11	9.730E-03	8.940E-04	10.88	91.59
12	9.570E-03	9.011E-04	10.62	91.39
13	9.060E-03	8.774E-04	10.33	91.17
14	7.740E-03	7.490E-04	10.33	91.18
15	6.590E-03	6.437E-04	10.24	91.10
16	6.250E-03	6.002E-04	10.41	91.24
17	5.420E-03	5.129E-04	10.57	91.36
18	5.670E-03	5.303E-04	10.69	91.45
19	5.700E-03	5.307E-04	10.74	91.48
20	5.520E-03	5.018E-04	11.00	91.67
21	4.910E-03	4.687E-04	10.48	91.29
22	4.480E-03	4.316E-04	10.38	91.21
23	4.590E-03	4.378E-04	10.48	91.29
24	4.450E-03	4.379E-04	10.16	91.04
25	4.220E-03	4.056E-04	10.41	91.23
26	3.720E-03	3.623E-04	10.27	91.13
27	3.680E-03	3.555E-04	10.35	91.19
28	3.270E-03	3.268E-04	10.01	90.91
29	3.430E-03	3.280E-04	10.46	91.27
30	2.655E-03	2.646E-04	10.04	90.94
Mean			10.5	91.3
S.D.			0.3	0.2
% R.S.D.			2.9	0.2
95% Confidence level			10.5 ± 0.6	91.3 ± 0.5

Detector used is SEM.

14880, ETP) with an active area 10 mm × 25 mm, rise time of about 2.0 ns, recovery time <5 ns and a typical gain of 1 × 10⁶ at 2.8 kV. The bare SEM purchased was mounted on a 6-in. o.d. CF flange and the required electrical connections for high voltage and signal cabling were done at home. Operating voltage of this SEM is usually maintained in the -2.4 to -3.0 kV range. The second one is a dual microchannel plate (MCP) detector (Proxitronic) in a chevron configuration, with 25 mm diameter active area and a maximum gain of 10⁷ at 1000 V per plate, with <150 ps rise time. This detec-

tor is generally used with -300 V on the bottom plate and -2100 V on the upper plate and the typical gain is about 10⁶. The signal from the SEM or MCP is fed to a preamplifier (Philips Scientific Model 6950) and then to a fast digital storage oscilloscope (LeCroy, Model 9350AM) which in turn is interfaced with a PC through a GPIB (general purpose interface bus) for data collection [8].

Two types of B₄C pellets are used in this study. Both had densities higher than 95% of theoretical density. Sample 1 corresponds to B₄C pellets (40 mm diameter and 40 mm long), with natural isotopic

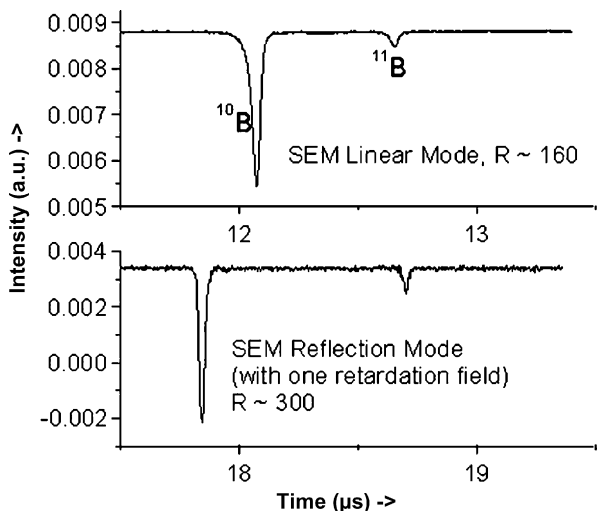


Fig. 4. The typical boron isotopes mass spectrum obtained with linear, Reflectron modes (1000 sweeps).

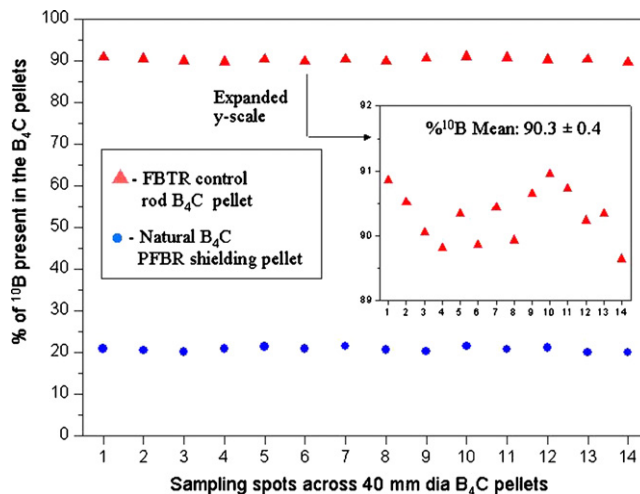


Fig. 5. Typical values obtained for ¹⁰B across diameter of B₄C pellets used (of FBTR control rod and natural composition used in PFBR).

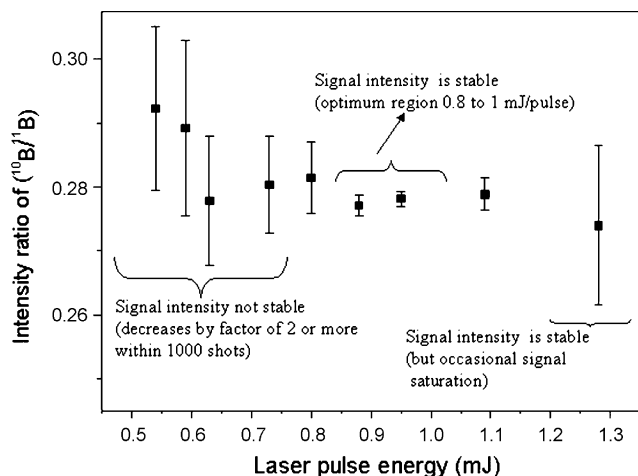


Fig. 6. Variation of isotope ratio (of natural B₄C) as measured as a function of laser pulse energy.

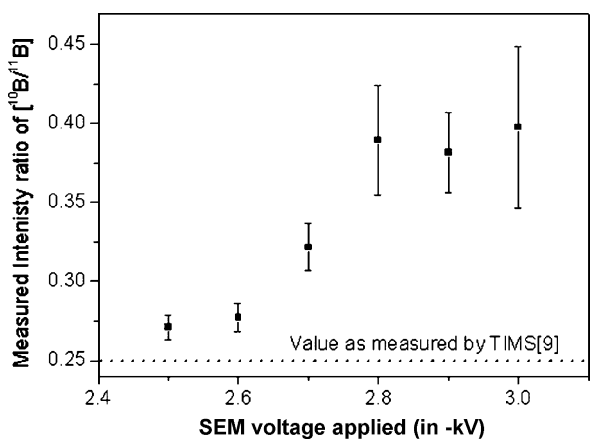


Fig. 7. Variation of intensity ratio of boron in natural B₄C pellet as a function of SEM voltage (in linear TOF mode). The signal appears to saturate for voltages beyond 2.8 kV.

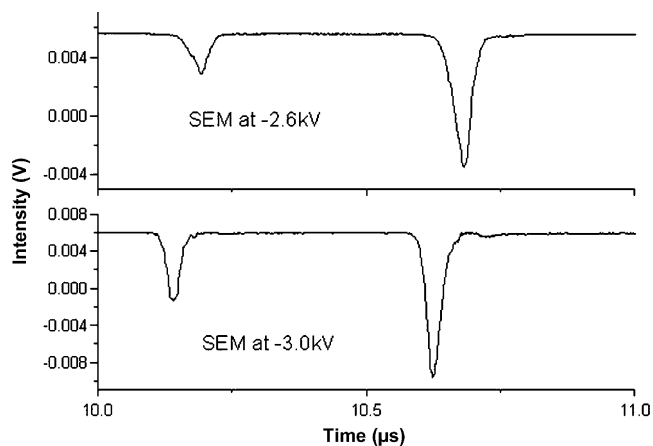


Fig. 8. A typical mass spectrum obtained at –2.6 and –3.0 kV of SEM, keeping all other experimental conditions identical. At –3.0 kV, square shaped peaks were visible for some laser shots, due to detector saturation. Though such square peaks are not clearly visible here due to signal averaging, the spectrum could lead to wrong isotopic ratios.

Table 3

Typical values of isotopic ratios ¹⁰B/¹¹B obtained for natural B₄C and FBTR control rod composition using MCP and SEM along with the TIMS values

% 10-B in the sample [TIMS value]	% 10-B value obtained using SEM in linear mode in LMS	% 10-B value obtained using MCP in reflectron mode in LMS
19.897 ± 0.142	21.9 ± 0.4	20.9 ± 0.7
90.653 ± 0.07	91.3 ± 0.2	90.3 ± 0.7

composition of ~20% in ¹⁰B, used as reflector in PFBR and FBTR core and sample 2 corresponds to pellet enriched to ~90% in ¹⁰B (39 mm diameter and 40 mm long) used in the control rod in our Fast breeder test reactor (FBTR) [4]. These pellets were loaded as such, without any additional preparations. In order to demonstrate the applicability of this method for analyzing irregular-shaped B₄C solid pieces, a broken piece of B₄C (natural B) of irregular shape is also analyzed.

It is known that thermal ionization mass spectrometric (TIMS) method provides a high degree of accuracy and precision for the determination of B isotope composition. Hence, for the sake of comparison, samples used in LMS studies were subjected to TIMS measurements as well, using a VG Micromass 30BK mass spectrometer available in our laboratory. However, TIMS cannot be adopted for such non-destructive analysis, that too for such a relatively big pellet.

3. Results

The typical boron isotopes mass spectra obtained for the FBTR control rod pellet samples with both TOF configurations (linear and reflectron modes) are shown in Fig. 4. The resolution of the TOFMS is defined as $m/\Delta m = T/2\Delta T$ [9], and in our instrument we obtained a resolution of ~150 and 300 (Fig. 4) for the linear and the reflection modes, respectively. Similar results were obtained with the MCP as

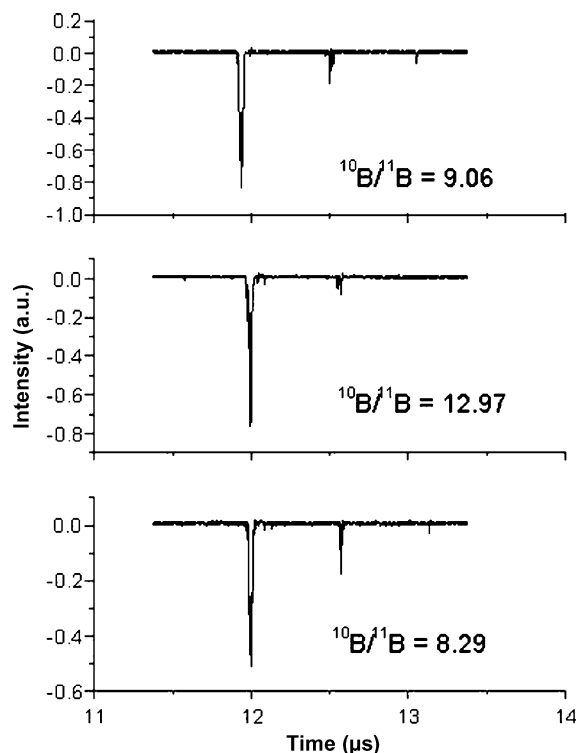


Fig. 9. Mass spectrum of boron isotopes obtained for B₄C FBTR pellet for different single shots, to show the variation in the observed intensity over shot to shot.

detector. The resolution obtained can be improved further by using high voltage power supplies having stabilized output voltage, low level jitter in the laser beam (good pulse to pulse energy stability) and by adjusting the laser power density, etc. All these aspects are being pursued. The $^{10}\text{B}/^{11}\text{B}$ ratio is calculated by measuring the area under the peaks, and from this ratio the enrichment value is deduced. Typical values of percentage of ^{10}B obtained for enriched B_4C pellet (FBTR sample) using SEM detector is shown in Table 2. Similar measurements are done using MCP detector and also for the natural B_4C pellets (used in PFBR). No definite advantage is realized in using one type of detector over the other in this present study.

One of the objectives for the present study is to measure the radial distribution of $^{10}\text{B}/^{11}\text{B}$ ratio in the irradiated B_4C pellets. In order to do this, measurement of the ratio is done by scanning the sample and the data is collected at space intervals of ~ 1 mm and the focused laser spot on the sample is of diameter of about $400\ \mu\text{m}$. The typical values obtained for percentage of ^{10}B across the diameter of the B_4C pellet is shown in Fig. 5. As can be seen in this figure, there is a slight variation of the isotopic ratio distribution across the pellet, even though the actual variation is within the experimental uncertainty. Similar experiments were also carried out with the natural B_4C pellet.

As discussed before, the ratio of $^{10}\text{B}/^{11}\text{B}$ is found to deviate from the actual value at very high laser power density. A high laser power density gives rise to a large density of ions and also a large spread in the kinetic energy of these ions. The mass resolution is degraded due to the energy spread and due to space charge effects. A large number of ions can cause the MCP to have a nonlinear response, as well as reduce the expected linear electron multiplier gain in SEM. Consequently, the actual intensity of the signal for both the isotopes could be different from those expected for the relative abundances in the sample. On the other hand, it is necessary to produce enough

number of ions so as to have a good S/N ratio and thereby to reduce uncertainty in the area under the peak. Hence, it is very important to optimize the laser intensity. Results of such a measurement of the intensity ratio for different laser pulse energies are shown in Fig. 6. As can be seen from this figure, the optimum laser pulse energy is about $0.8\text{--}1$ mJ/pulse.

The measured isotopic ratio was observed to depend on the voltage applied to the SEM or MCP. Increasing the voltage applied to the upper plate of MCP from -1900 to -2100 V changes the signal intensity ratio from 0.256 to 0.277 (TIMS value: 0.248) for natural B_4C and from 8.6 to 9.1 for the composition used in the FBTR control rod (TIMS value: 9.696) [10]. Considerable signal to noise (S/N) ratio improvement occurs at the operating voltage of -2100 V. In the same manner for a change in the voltage applied to the SEM from -2.5 to -3.0 kV, the $^{10}\text{B}/^{11}\text{B}$ ratio changes from 0.271 to 0.397 for a natural B_4C pellet as shown in Fig. 7, with all other experimental conditions remaining identical. Such an increase can occur if higher intensity isotope signal saturates at the higher SEM voltages. Typical mass spectra obtained at -2.5 and -3.0 kV are shown in Fig. 8. Even though the signal saturation is observed for several laser shots, often it was not obviously visible in the averaged spectrum and hence results obtained in such cases can be misleading. So it is essential to observe the spectrum during data acquisition itself and avert such saturation. In other words, the linearity of the detector is to be ascertained during the experiment. For optimized conditions, the SEM can be operated between -2.6 and -2.8 kV.

Typical values of isotopic ratios of $^{10}\text{B}/^{11}\text{B}$ obtained for natural B_4C and FBTR control rod composition using the MCP and the SEM are shown in Table 3, along with the TIMS values. As can be seen from this table, the scatter in the values is marginally less with SEM, even though the deviation from the TIMS values is more. Hence, in regular analysis of this ratio, one needs to correct for the deviation.

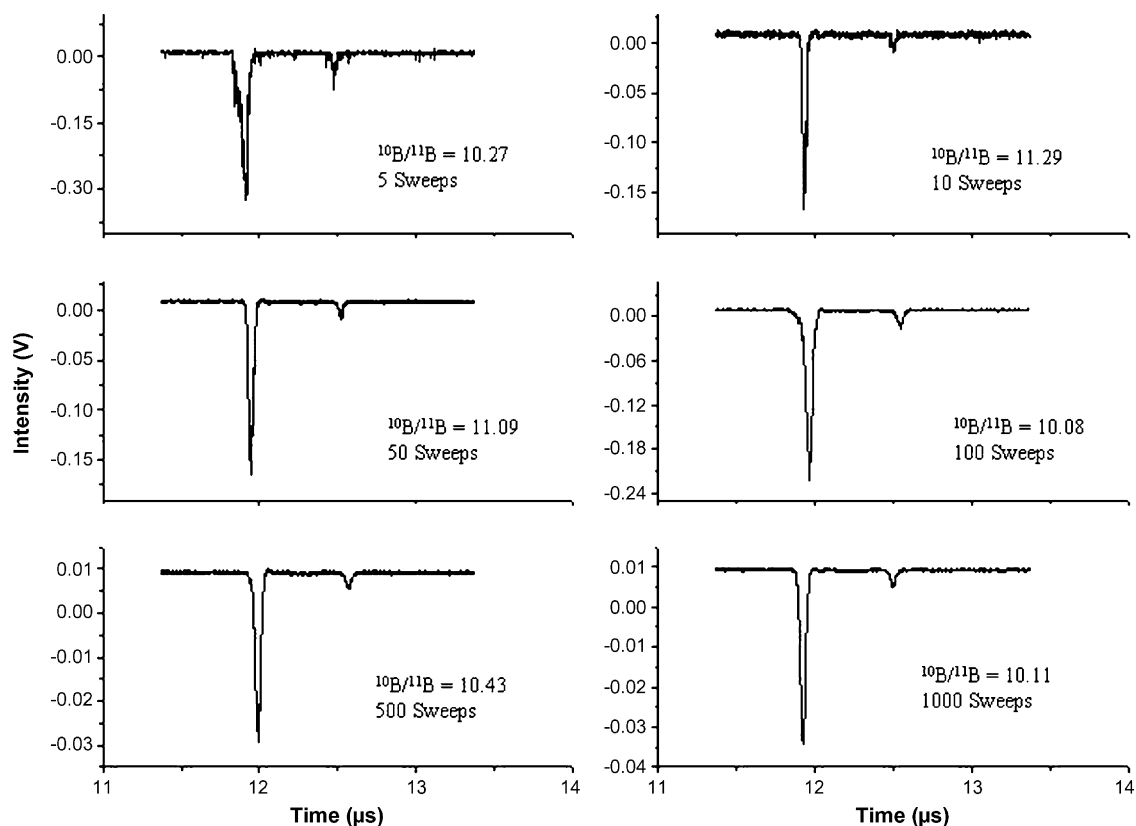


Fig. 10. Mass spectrum of boron isotopes obtained for FBTR pellet averaged over different number of laser shots, namely, 1, 5, 10, 100, 500, 1000 laser shots.

It is important to note that the signal averaging is a must to get reasonably good signal to noise ratio in the acquired mass spectra. This is done using the digital storage oscilloscope to sum average 1000 traces (for 1000 laser shots). If the signal averaging is not done, then the spectrum is very noisy and the scatter in the data is very high. For example, the mass spectrum obtained for the FBTR pellet is shown in Fig. 9 for three single shots; the $^{10}\text{B}/^{11}\text{B}$ ratio of varies from 9.06 to 8.29. The mass spectra obtained averaging for a different number of laser shots (1, 5, 10, 100, 500, 1000) are shown in Fig. 10. As can be seen from this figure, in order to get reasonably good signal-to-noise ratio, at least 500 sweeps are required. The oscilloscope used in the present study can average a maximum of 1000 sweeps and all the data reported in this article are averaged for 1000 sweeps.

As indicated earlier, the main objective of developing this facility is to determine the boron isotope ratio in the irradiated B_4C

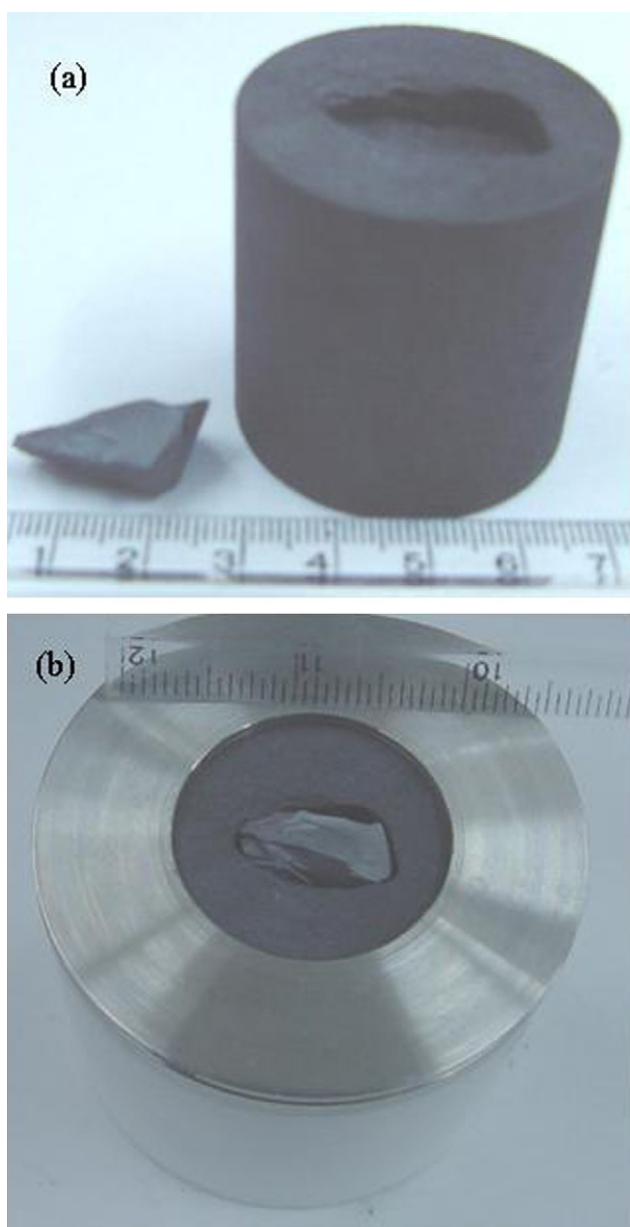


Fig. 11. (a) Broken B_4C pellet having an irregular shape and a graphite rod with cavity to hold it and (b) B_4C sample loaded in the graphite holder in the sample cup.

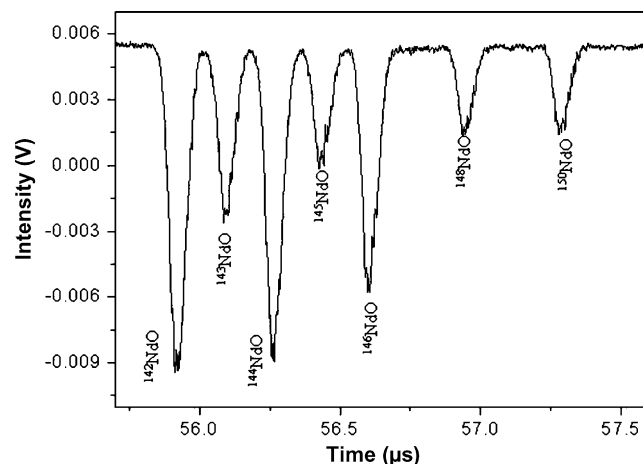


Fig. 12. Typical mass spectrum of Nd isotopes obtained from a sample of $\text{Nd}(\text{NO}_3)_3$ in tri-butyl phosphate and methanol matrix (1000 sweeps).

pellets non-destructively so that, if possible, the pellets can be used again in the control rods. It may become necessary to measure such isotopic ratios on irregular shapes of broken B_4C pellets. One such sample was taken whose shape is shown in the photograph (Fig. 11(a)). Unlike regular pellets, these kinds of samples cannot be loaded directly; they need to be placed inside a graphite mount as shown in Fig. 11(b) (photograph) and loaded. The spread in the values obtained for this sample were similar to those shown in Table 2.

The utility of this mass spectrometer for the analysis of other samples such as rare earths in solution was tested with Nd nitrate solutions. The sample was prepared as indicated in the literature [11]. A typical mass spectrum obtained is shown in Fig. 12 and the isotopic ratios obtained for this Nd sample of natural abundance is shown in Table 4. As can be seen from the figure, complete base line separation is not obtained. The incomplete resolution (of ions of two different masses reaching the detector at the same time) can lead to error in the measured abundances. This effect could be noticed in the figure, where the first peak (^{142}Nd) is $\approx 1\%$ less than that given by the natural abundance, whereas for all other peaks which are at unit mass separated, this difference is less. The problem of contribution from larger flight time part of the ionic species of the earlier mass peak to the next larger mass peak could be minimized if one resorts to (i) accelerate the ions to very large potential (about 10 kV) or (ii) use gracing incidence of laser beam on the sample surface, so that the kinetic energy spread of the ions is made less. The latter was employed by us in another RTOFMS and a complete base line separation could be observed for almost same acceleration (1000 V) [11]. Due to design constraints in handling radioactive samples, such gracing angle incidence of the laser beam is not easy to adopt in the present

Table 4

Comparison of isotopic abundance of natural Nd as measured using the present LMS with literature values [2]

Isotopes Nd	Natural abundance [2]	Value obtained
142	27.2 (5)	26.2 ± 0.4
143	12.2 (2)	12.6 ± 0.4
144	23.8 (3)	23.6 ± 0.3
145	8.3 (1)	8.8 ± 0.2
146	17.2 (3)	17.4 ± 0.3
148	5.7 (1)	5.9 ± 0.2
150	5.6 (2)	5.6 ± 0.3

experimental facility. Such an option is being explored in our next design.

4. Conclusions

An experimental facility has been built for the analysis of the isotopic ratio of $^{10}\text{B}/^{11}\text{B}$ in irradiated B_4C pellets of the FBTR control rod and demonstrated satisfactorily, using inactive samples (^{10}B percentage values were obtained within $\pm 1\%$ of the actual). Further modifications/additions to the system are under way to be able to handle the radioactive samples.

Acknowledgements

The authors are grateful to Dr. P.R. Vasudeva Rao, Director, Chemistry Group and Dr. T.G. Srinivasan, Head, Fuel Chemistry Division, for their encouragement, Dr. R. Viswanathan, Head, Mass Spectrometric Studies Section, for useful discussions, Mr. R. Balasubramanian for providing the TIMS data and Mr. R. Parthasarathy for technical support.

References

- [1] A.G. Rafi Ahmed, Sujoy Sen, V. Gopialakrishnan, "Cross-sections of some control-rod materials for PFBR" Report No. RPD/NDS/103, Indira Gandhi Centre for Atomic Research, January 2003.
- [2] Hand Book of Chemistry and Physics, 73rd ed., CRC Press, 1992–1993, p. 11.
- [3] S.C. Chetal, V. Balasubramanian, P. Chellapandi, P. Mohanakrishnan, P. Puthiyavinayagam, C.P. Pillai, S. Raghupathy, T.K. Shanmugham, C. Sivathanu Pillai, Nucl. Eng. Des. 236 (2006) 852.
- [4] P.V. Ramalingam, M.A.K. Iyer, R. Veerasamy, S.K. Gupta, L. Soosainathan, V. Rajan Babu, International Atomic Energy Agency, Vienna, IAEA-TECDOC-884, 1996, 60.
- [5] V.D. Risovany, A.V. Zakharov, E.P. Klochkov, A.G. Osipenko, N.S. Kosulin, G.I. Mikhailichenko, Proceeding of a Technical Committee meeting on 'Absorber Materials, Control Rods and Designs of Shutdown Systems for Advanced Liquid Metal Fast Reactors' IAEA-TECDOC-884, 1996, 219.
- [6] S. Anthonysamy, Chemistry Group, IGCAR, private communication.
- [7] G.L. Klunder, P.M. Grant, B.D. Andresen, R.E. Russo, Anal. Chem. 70 (2004) 1249.
- [8] M. Joseph, N. Sivakumar, IGC Report-169, 1995.
- [9] R.J. Cotter, Time-of-Flight Mass Spectrometry, Instrumentation and Applications in Biological Research, ACS, Washington, DC, 1997.
- [10] M. Joseph, N. Sivakumar, P. Manoravi, R. Balasubramanian, Rapid Commun. Mass Spectrom. 18 (2004) 231.
- [11] M. Joseph, N. Sivakumar, P. Manoravi, R. Balasubramanian, Int. J. Mass Spectrom. 253 (2006) 98.