Thermochimica Acta 500 (2010) 63–68

Contents lists available at ScienceDirect

Thermochimica Acta

journal homepage: www.elsevier.com/locate/tca

Studies on the ignition behaviour of boron powder

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ARTICLE INFO

Article history:
Received 27 August 2009
Received in revised form
11 December 2009
Accepted 16 December 2009
Available online 24 December 2009

Keywords:
Boron
Ignition temperature
Thermogravimetry
Particle size

ABSTRACT

The ignition behaviour of boron powder, prepared through electrowinning process, was studied by using thermogravimetry coupled with simultaneous differential thermal analysis (TG-SDTA). The dependence of the inception of the ignition reaction on the partial pressure of oxygen, particle size of the boron powder and heating rate was investigated. It was observed that all these factors affect the ignition temperature. Boron powder with a mean particle size of about 10 μm was found to be susceptible to ignition in oxygen even at 783 K. In general, the susceptibility to ignition was found to vary inversely with the degree of crystallinity. Presence of carbon was found to retard the oxidation of boron and raise the ignition temperature. These results are useful in safe handling and storage of finely divided boron powder and in the subsequent production of boron carbide from it.

1. Introduction

Elemental boron in the form of boron carbide finds numerous applications in nuclear, abrasive and armor industries. High-density boron carbide pellets containing boron enriched in ^{10}B (65 at.%) will be used in the control rods of the Indian prototype fast breeder reactor (PFBR) [1]. The methodology for the production of high-density boron carbide pellets starting from elemental boron is described elsewhere [2,3]. Submicron powders of elements are generally air sensitive and pyrophoric in nature [2]. Finely divided boron can burn in oxygen at a favourable temperature resulting in an explosive ignition reaction. In fact, boron is used as a combustible in aerospace applications [4].

For efficient fabrication of boron carbide starting from its constituent elements, it is essential to have boron powder with an average particle size of 10 μm [2]. Upon exposure to air/oxygen, this powder would react to form boric oxide layer on the surface [5]. Boric oxide is a highly stable compound for it has large negative Gibbs energy of formation (for example, $-1139.18\,\mathrm{kJ\,mol^{-1}}$ at 500 K). Conversion of boron to boric oxide is a highly exothermic reaction. The high exothermicity of this reaction leads to the ignition of the reaction mixture under favourable conditions. It is essential to identify these conditions in order to establish the optimum conditions for the safe handling and storage of elemental boron. Further such an investigation would also provide valuable inputs to the synthesis of B_4C from the elements.

Many authors have studied the ignition and combustion reaction of boron, as boron based propellants find applications in aerospace industries. The mechanism of ignition of elemental boron can be described as a two stage process. In the initial stage, depending on the partial pressure of oxygen, temperature and particle size a rapid reaction between boron and oxygen occurs leading to the formation of B_2O_3 layer on the surface of the boron particles with large evolution of heat. However, the reaction does not go to completion due to the hindrance for diffusion of oxygen gas by the impervious oxide film. The rate of oxidation is then controlled by the diffusion of oxygen through this film [6]. In the second stage, at higher temperatures the impervious film ruptures and the inner core of boron is also exposed to oxygen gas. At this stage, the oxide film vaporizes and the formation of gaseous species such as BO_2 and B_2O_2 take place. The ignition of these gaseous species results in the complete ignition of boron.

Talley [7] measured the rates of oxygen consumption by boron rods which were electrically heated from 1500 to 2300 K at 1 bar pressure. This author concluded that the rate of burning of boron was controlled by the outward diffusion of boron oxide (B_2O_3) gas across the reaction interface.

Prentice [8] used time-exposure photography of the boron particles with an average size of 177–250 μm, ignited by a xenon flash, in air and in oxygen–argon mixture at 1 bar pressure. This author reported a two stage combustion process. The first, low temperature stage, corresponds to the surface burning process and the second, high temperature stage, to the gas phase combustion.

Macek and Semple [9] have studied the ignition of boron particles (approx. size 40 μm) at 1 bar pressure and in hot (2200–2900 K) oxidizing atmosphere. They have reported an ignition temperature

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doi:10.1016/j.tca.2009.12.011

of about 1950 K and observed that the burning time of the boron powder was inversely proportional to the mole fraction of oxygen and gas temperature. This implied that both chemical kinetics and oxidizer diffusion play important roles in the combustion of bare boron.

Gurevich et al. [10] studied the ignition and combustion of crystalline and amorphous boron in hot gas streams produced by a plasma generator. Their results showed that the ignition temperature decreases with increase in concentration of oxygen or water vapor while it increases with increase in the particle size.

Mohan and Williams [11] have studied the ignition of boron particles of size of about 100 μm using helium–neon laser. They reported a low temperature burning for crystalline boron and almost explosive burning for amorphous boron. They also have developed an oxide coating model for describing the low temperature combustion and ignition phenomena.

Yeh and Kuo [12] measured ignition delay and combustion times of single boron particles with sizes in the range 2–3 μm. These authors also studied the effect of temperature of the gas, oxygen concentration as well as the effect of magnesium coating on the combustion of boron.

Li et al. [13] used suspensions of boron in a flat flame burner to study the combustion of a single boron particle. These authors observed three types of boron flame plumes and determined the rate constant of this combustion reaction as a function of temperature of the gas. Uda [14] studied the ignition of boron using the shock-tube technique in air at pressures up to 20 bar. These authors observed the fall in ignition temperature viz. from 1900 K at 1 bar pressure to 1400 K at 20 bar. Krier et al. [15] studied the ignition of boron in oxygen, oxygen + 30% H_2O (g), 1–3% SF_6 (g) and 6–12% HF (g) at 8.5–34 bar. Their results indicated that water and SF_6 reduce the ignition delay time for amorphous and crystalline boron particles when compared to pure oxygen but the burning time is unaffected by the additives.

Yuasa and Isoda [16] have reported the critical temperature for self-sustained combustion of boron lumps (size 5–8 mm long and weight 0.09–0.11 g) to be 1580 K and found out the threshold partial pressure of oxygen in O_2–N_2 mixtures to be 0.3. Meinkohn [17] proposed a first stage combustion model and determined the ignition temperature for ignition of boron and also showed that the increase in the pressure and the decrease in the particle size bring down the ignition temperature.

Li and Williams [18,19] proposed that during the ignition, boron dissolves at the B–B_2O_3 (l) interface and diffuses across the liquid layer; at the same time a vitreous complex $(BO)_3$ is formed through the reaction between dissolved boron and B_2O_3 (l). Yeh and Kuo [12] studied the combustion characteristics and measured combustion times of single boron particles. These authors observed that the combustion of fine boron particles occur in two stages. First stage corresponds to the burning of a single boron particle that is covered with a layer of B_2O_3 and a second stage corresponds to the completion of the combustion of the bare boron particle.

The literature on the ignition of boron indicates that extensive studies have been carried out only on the second stage of the ignition process. The work carried out so far on the combustion and ignition of boron concentrated more on second stage of ignition. No study has been carried out to determine “ignition” temperature for the initial stages of ignition. Low temperature ignition and combustion of boron is important for the safe handling and storage of boron. It is important to determine the minimum temperature and partial pressure of oxygen required to initiate the ignition of boron. Further, no studies have so far been conducted on the systematics at low temperature pertaining to the influence of the heating rate, particle size and partial pressure of oxygen on the ignition of boron. In view of the above, a study on the ignition behaviour of elemental boron was undertaken.

The ignition temperature 'T_{ig}' of boron powder is the onset temperature in the differential thermal analysis (DTA) curve when a sudden escalation in the oxidation reaction takes place [20]. This temperature is affected by several factors like partial pressure of oxygen available at the reaction site, the particle size of boron powder and the heating rate [21]. In this study, thermogravimetry (TG) and differential thermal analysis (DTA) methods were used for the determination of the ignition temperature as well as to investigate the effect of partial pressure of oxygen, particle size and heating rate on the ignition temperature.

2. Experiment

2.1. Chemicals

Electrodeposited boron in the form of chips was obtained from the Boron Chemistry Laboratory, Chemistry Group, Indira Gandhi Centre for Atomic Research, India. Reference boron sample (purity 99.999%, particle size <32 μm) was procured from M/s. Aldrich Chemical Company, Inc., USA. The calibration standards for DTA viz. In, Sn, Al and Au were procured form M/s. Mettler Toledo (I) Pvt. Ltd., Mumbai.

2.2. Sample preparation and chemical assay

Elemental boron was produced through electrowinning from potassium fluoroborate dissolved in a mixture of molten potassium fluoride and potassium chloride. The electrodeposited boron was ground in a planetary ball mill (Model: Pulverisette 5, M/s. Glen Creston Ltd., Germany) with tungsten carbide lined hardened steel bowls and tungsten carbide balls (diameter: 20 mm), at 150 rpm for an hour. The boron powder thus obtained was purified by leaching it with boiling water and then with 11.3N hydrochloric acid. The details of this purification procedure are described elsewhere [22]. The purified boron powder was graded into different size fractions using a sieve shaker with stack that comprised 32, 45, 56, 71, 90, 125, 160, 180, 250 and 350 μm stainless steel sieves (M/s. Firtsch GmbH, Germany). These size fractions were collected separately, stored under argon and used in the ignition experiments.

Boron powder was characterized for the boron assay and impurities. Boron assay was carried out by volumetric method. The residual carbon present in the boron powder samples was determined by heating these samples in a stream of oxygen and measuring the carbon dioxide generated using an IR detector. The silicon present in the boron was analyzed by a spectrophotometric method. The other metallic impurities (iron and nickel) were analyzed using atomic absorption spectroscopy.

2.3. Thermogravimetry and differential thermal analysis

The thermal analysis experiments were carried out using a thermal analysis system (TG-SDTA 851^e Mettler Toledo, Switzerland). This equipment has a weight sensitivity of 0.1 μg and a temperature sensitivity of 0.01 K. A platinum crucible of 70 μl capacity was used as the sample container. The temperature was measured using a Pt–Pt-10% Rh thermocouple in firm contact with the sample holder on which the Pt sample container was placed. The temperature calibration was carried out by measuring the melting points of pure metals such as In, Sn, Al and Au. The inner muffle of the furnace was constantly purged with O_2 gas, the flow of which was controlled by using a mass flow controller (AFC 80MD, Qualiflow, France). A constant gas flow of 1.2 $m^3\ h^{-1}$ and a predetermined heating rate were used in these experiments. A three step temperature programme was used for this study. In the first step the sample was heated from 303 to 393 K at a heating rate of 10 K min^{-1}. In the second step, the sample was heated at 393 K for about 15 min in order to remove

Table 1
Details of the heating profiles employed in the TG-DTA experiments.

Segment	Start temperature/K	End temperature/K	Heating rate/K min^{-1}
Step 1	303	393	10
Step 2	393	393	15 min (isothermal)
Step 3	393	1073	5, 10, 15, 20, 25

Table 2
Chemical assay of electrodeposited boron powder.

S. no.	Element	Concentration/wt%
1	Boron	96
2	Carbon	1.4
3	Nickel + iron	0.98
4	Silicon	1.13

Table 3
Ignition temperatures obtained by heating the sample in a stream of oxygen gas.

Particle size	Ignition temperature/K				
	5[a]	10[a]	15[a]	20[a]	25[a]
10–32 μm	783.24	801.22	814.73	823.74	826.98
32–45 μm	784.87	801.29	815.48	824.08	827.43
45–56 μm	785.09	802.18	817.21	822.81	828.03
56–71 μm	785.37	804.6	819.32	824.76	828.2
71–90 μm	785.34	805.01	819.86	825.86	829.23
90–125 μm	786.27	806.15	820.82	826.99	834.56
125–160 μm	791.13	807.1	824.26	827.89	834.98
160–180 μm	792.24	808.23	824.88	828.64	836.45
180–250 μm	797.58	811.62	826.21	830.79	839.83
250–350 μm	800.78	816.41	829.53	832.7	840.54

[a] Heating rate/K min^{-1}

the moisture adsorbed on to the fine powder. The first two steps were common to all the experiments while the third step varied between the experiments. Various heating rates viz. 5, 10, 15, 20 and 25 K min^{-1} were used in order to heat the sample from 393 to 1073 K. The details of the heating profiles are given in Table 1.

To study the effect of partial pressure of oxygen (p_{O_2}) on the ignition temperature (T_{ig}), the ignition temperature of the boron powder was determined by varying p_{O_2} over the sample. To vary p_{O_2}, pure oxygen gas was mixed with ultra high purity nitrogen gas. The flow of oxygen and nitrogen was precisely controlled by mass flow controllers. In a typical TG experiment, other experimental parameters such as particle size of the boron powder and heating rate were kept constant. "p_{O_2}" in the gas mixture (oxygen and nitrogen) was determined by using an in-house developed electrochemical oxygen meter. This oxygen meter utilizes calcia stabilized zirconia as solid electrolyte and air as the reference electrode. The reliability of the oxygen meter was checked against pure oxygen gas as the test electrode (gas).

3. Results and discussion

Chemical assay of the elemental boron is given in Table 2. The amount of metallic impurities present in boron is low enough not to affect the ignition and combustion behaviour of boron.

A typical TG curve along with the DTA curve obtained for the ignition of elemental boron is shown in Fig. 1. A sharp gain in the weight of the sample due to oxidation of the boron powder is reflected in the TG curve. A temperature lag between the sample and the reference temperature for a short duration is a clear indication of the ignition reaction of the boron. Appearance of the sharp exothermic peak in the DTA curve also confirms that the ignition and combustion of boron are exothermic processes. The heat of formation of B_2O_3 is approx. −1255 kJ mol^{-1} [4].

It was observed that the total weight gained ($[(w_f - w_i)/w_i] \times 100$ where w_f: final weight of the sample, w_i: initial weight of the sample) during the oxidation process is less than the theoretical weight gain obtained by considering the complete oxidation of boron to boric oxide, indicating that this reaction does not proceed to completion even after prolonged heating. This could be due to the formation of a glassy layer of boron oxide on the surface of the boron particles that could slow down further diffusion of oxygen across the reaction interface. This was further substantiated by an X-ray diffraction study of the reaction products [23].

3.1. Dependence of ignition temperature on mean particle size

The variation of the ignition temperature, T_{ig}, of boron in oxygen with the average particle size and with heating rate is given in Table 3 and shown in Fig. 2. The average particle size represents the arithmetic mean of the extreme values of the particles in a typical size range, e.g. for those particles falling in the size range 10–32 μm, the average size is taken as 21 μm. It is observed that the T_{ig} increases with the size of the particles for a given heating rate. It is evident from Fig. 2 that the ignition temperature varies linearly with the particle size of the boron powder. The linear fit parame-

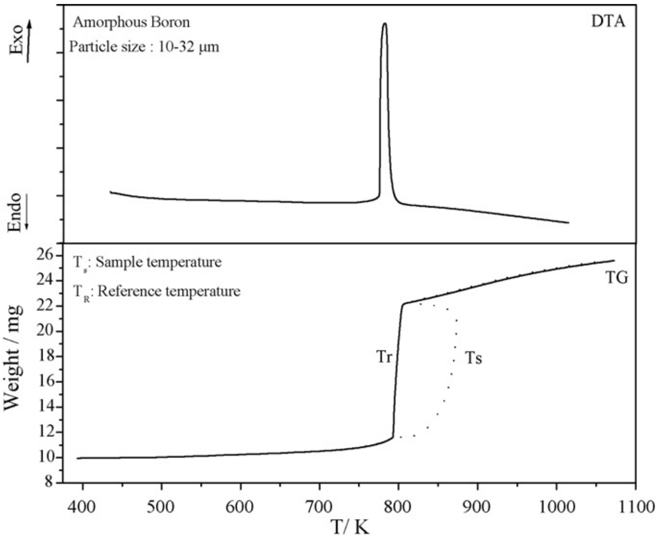

Fig. 1. Thermogram and DTA curve for the ignition of boron.

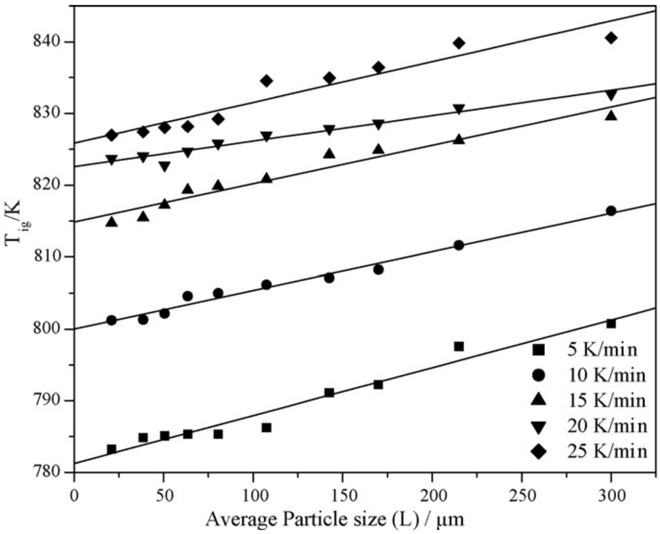

Fig. 2. Variation of the ignition temperature with the particle size of boron powder.

Table 4
Linear fit parameter for the variation of ignition temperature with particle size.

T_{ig} (K) = $A + BL$ (μm)				
Heating rate/K min^{-1}	A (intercept)	B (slope) (K/μm)	R (regression)	SD (standard deviation)
5	781.2	0.06	0.98	1.2
10	800.0	0.05	0.98	0.7
15	814.8	0.05	0.97	1.1
20	822.5	0.03	0.98	0.7
25	825.8	0.05	0.96	1.6

ters are shown in Table 4. The reactivity of the powder depends on the size of the constituent particles. The powder with finer particles provides greater surface area and is thus more reactive. Boron powder of lower size fraction reacts faster and gets ignited at a relatively lower temperature. The powder having particles with a size of 32 μm or lower could be handled safely without the risk of ignition up to 783 K in air. These observations testify the conclusion that the oxidation of the boron powder at room temperature is a kinetically hindered process.

3.2. Effect of partial pressure of oxygen on the ignition temperature

The variation of ignition temperature and the percent gain in weight with partial pressure of oxygen "p_{O_2}" for particle size 10–32 μm of boron is given in Table 5 and plotted in Fig. 3. It is seen from Table 5 that a minimum partial pressure of oxygen is required for the ignition reaction to take place as no exothermic peak was observed in the DTA curve below $p_{O_2} = 2 \times 10^3$ Pa. Below this p_{O_2}, the oxidation of boron powder occurs to a small extent which is seen from the percent gain in weight. This shows that the oxygen pressure was not sufficient to cause the ignition of boron though it could oxidize the sample. As the partial pressure of the oxygen gas increases the ignition of the boron gets facilitated. It is seen from Fig. 4 that T_{ig} falls exponentially with the increase in p_{O_2} and at higher p_{O_2} ($p_{O_2} > 4 \times 10^4$ Pa) the T_{ig} becomes constant. The extent of oxidation reaction as inferred by the gain in weight shows that as the partial pressure of oxygen increases the extent of oxidation also increases. At higher p_{O_2} ($p_{O_2} > 2 \times 10^4$ Pa) the gain in weight due to oxidation does not change much with the increase in p_{O_2}.

Table 5
Ignition temperature T_{ig}/K obtained by varying the partial pressure of oxygen for boron powder (particle size < 32 μm).

p_{O_2}/Pa	T_{ig}/K	Weight gain/%
12	[a]	11.3
15	[a]	14.5
30	[a]	20.5
2×10^3	[a]	23.4
4×10^3	807.9	75.8
6×10^3	806.5	116.0
8×10^3	805.5	131.5
1×10^4	802.6	135.7
2×10^4	800.5	141.3
3×10^4	799.3	144.4
4×10^4	798.7	151.5
5×10^4	798.9	152.1

[a] No exothermic peak observed in the thermogram.

3.3. Effect of heating rate on ignition temperature

The variation in ignition temperature with heating rate for various size fractions of elemental boron is shown in Fig. 4. It is observed that the T_{ig} increases with heating rate. Further, the ignition temperature does not increase significantly with the rate of heating when the value of the latter exceeds 20 K min^{-1}. At lower heating rates, the temperature gradient across the sample would be less

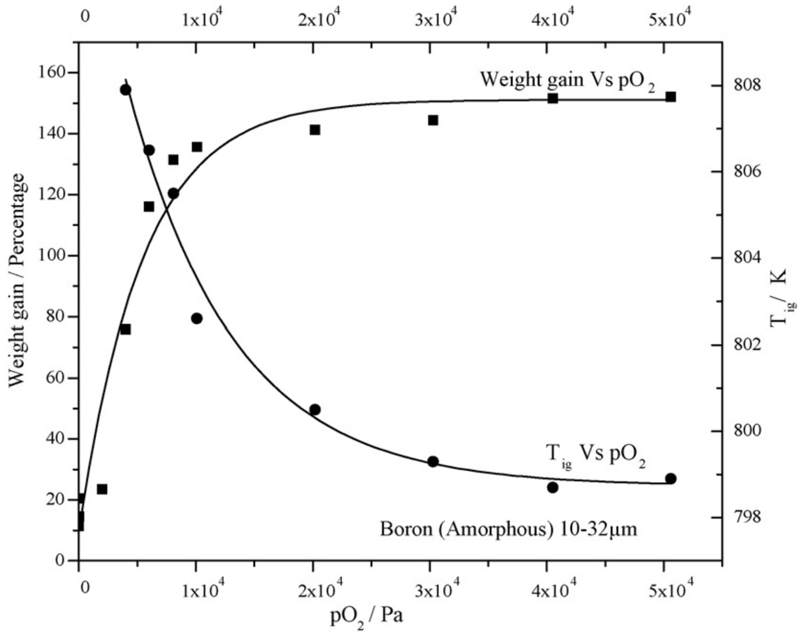

Fig. 3. Plot of partial pressure of oxygen with gain in weight during oxidation and ignition temperature of the boron.

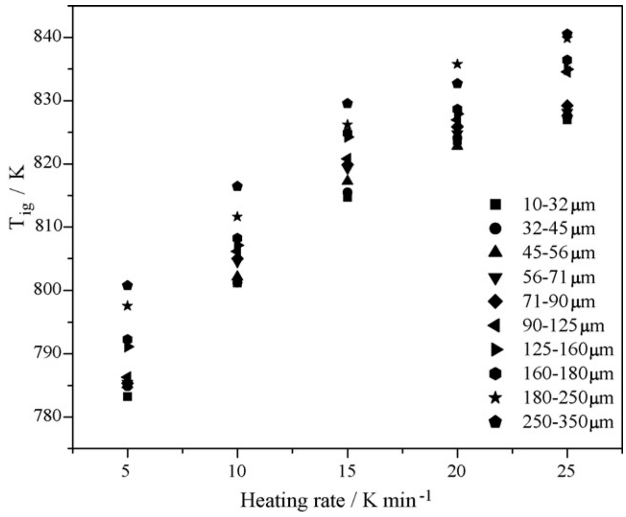

Fig. 4. Variation of ignition temperature with the heating rate.

and the difference between the furnace temperature and sample temperature will be low, facilitating a thermal equilibrium between the sample and the furnace. During this process the sample sees the particular temperature for a longer interval of time which results in uniform heating of the bulk sample. Thus, the measured values of the onset temperature would be closer to the minimum ignition temperatures observed for a given size fraction. It is observed from the DTA curves that the peak height varies with the heating rate. Sharper and taller peaks were observed for higher heating rates (>10 K) whereas shorter and wider peaks were observed for lower heating rates. No exothermic effect was observed in DTA curve for the ignition of elemental boron powder when the ensuing rate of heating was less than 5 K min^{-1}.

When the sample is heated in a stream of oxygen, the difference in the ignition temperature corresponding to the highest heating rate (25 K min^{-1}) and the lowest heating rate (5 K min^{-1}) is approximately 43 K. This difference remains nearly the same for all the size fractions from 10 to 350 μm. Therefore, it could be concluded that ignition temperature is influenced by the change in heating rate for fixed sized particles present in the powder. The difference in ignition temperature for the different size fractions arise due to the temperature gradients prevailing across the sample and this temperature gradient is higher for higher heating rates.

Table 6
Ignition temperature for boron samples having different concentration of carbon.

S. no.	Quantity of carbon added/wt%	Ignition temperature/K
1	10	787.5
2	20	796.7
3	30	803.8
4	40	809.8
5	50	816.2
6	60	819.3
7	70	819.4
8	80	808.2
9	90	806.9

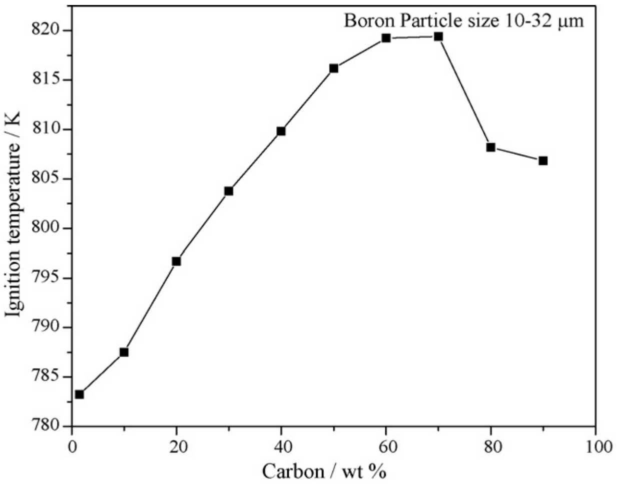

Fig. 5. Effect of carbon impurities on the ignition temperature.

3.4. *Effect of carbon impurity on the ignition temperature*

Carbon is a significant impurity in the elemental boron obtained through high temperature molten salt electrowinning process. The carbon impurity is introduced in boron due to the erosion of the crucible which is used as a container for the molten salt and due to attack of corrosive gases such as Cl_2 and F_2 on the graphite surface. In order to study the effect of carbon impurity present in boron sample on the ignition temperature, samples of boron powder having different quantities of carbon residue (carbon black, purity > 99.999) were prepared by ball milling carbon and boron powders. The experimental parameters such as the particle size (<32 μm) and heating rate (10 K min^{-1}) were kept constant and only the quantity of the carbon residue was varied. The results obtained are given in Table 6. The ignition temperature is plotted against carbon residue present in boron powder as shown in Fig. 5. It is inferred from these data that the ignition temperature increases with the concentration of the carbon in boron sample up to 50 wt% and remains almost the same as the concentration of the carbon increases further up to 70 wt%. A fall in the ignition temperature was observed when the concentration of the carbon is increased beyond 70 wt%. It was observed from the DTA curve (Fig. 6), for boron powders containing up to 70 wt% carbon, a single exothermic peak was observed. Beyond this carbon concentration two relatively small exothermic peaks were observed. This observation leads to the conclusion that at this concentration of carbon (carbon > 70 wt%) the individual ignition of the boron and combustion of carbon takes place. Here the first peak belongs to ignition of boron and second one to combustion of carbon. As the concentration of carbon increases the combustion reaction of carbon dominates the ignition reaction of boron.

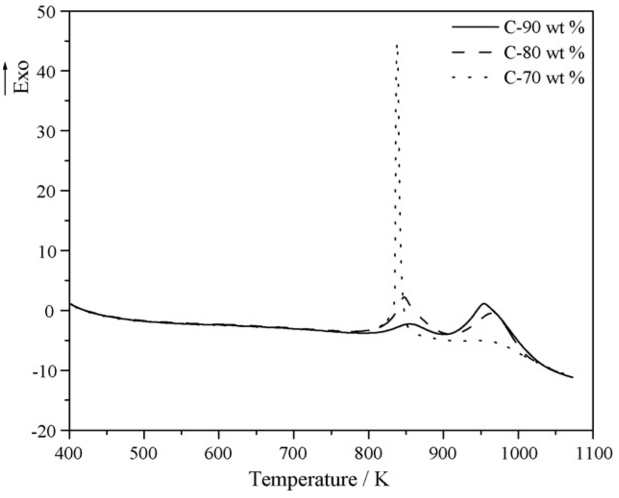

Fig. 6. DTA curve for the ignition of boron and carbon mixture.

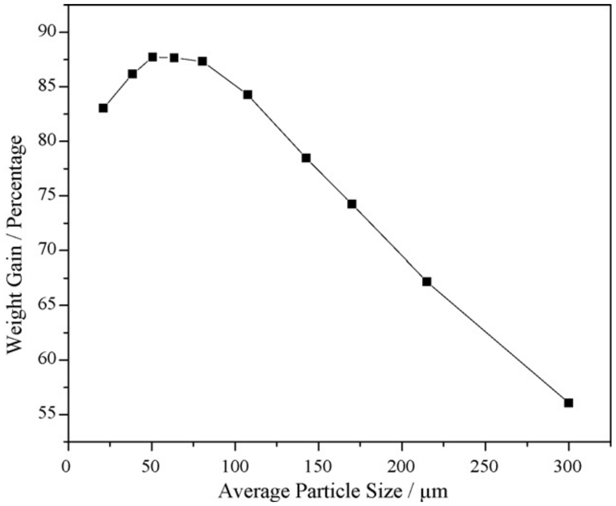

Fig. 7. Variation of the gain in weight with the particle size of boron powder.

Table 7
Ignition temperatures obtained by heating crystalline boron in a stream of oxygen.

	Heating rate/K min^{-1}				
	5	10	15	20	25
T_{ig}/K	798.8	800.0	801.4	802.5	804.9
Weight gain/%	70.0	64.0	61.5	57.7	55.4

3.5. *Effect of particle size on the total weight gain during ignition*

The variation in weight gain with the particle size is shown in Fig. 7. The weight gain is almost the same for the particles in the size range from 45 to 90 µm, while for those having size greater than 90 µm, the weight gain falls with increase in particle size. The weight gain in sample is due to the oxidation of boron and its conversion in to boric oxide. Weight gain is the indication of the extent of the reaction and it decreases as the size of the particles in the boron sample increases. Finer particles provide more surface area and react with oxygen at a higher rate compared to the coarser particles. It is important to note that the gain in weight increases with particle size for the particles in the range of 10–45 µm. The same trend is followed for all heating rates viz. 5, 10, 15, 20 and 25 K min^{-1}.

3.6. *Ignition temperature for crystalline boron powder*

The ignition temperatures for the crystalline boron powder having particles in the size range of 10–32 µm with weight gain during the ignition reaction are given in Table 7. The ignition temperature is higher for the crystalline boron compared to amorphous boron, which indicates that crystalline boron is more resistant to ignition compared to amorphous boron. The extent of oxidation reaction is also lower for the crystalline boron as reflected from the weight gain data. A comparison of the T_{ig} of crystalline boron with the T_{ig} of amorphous boron shows that the ignition temperature of crystalline boron powder does not vary much with the heating rate. This may be due to better packing of the crystalline powder in the sample container. The difference in the ignition temperature obtained at lowest heating rate (5 K min^{-1}) and the highest heating rate for amorphous boron is 43 K where as this difference is only 6 K for crystalline boron.

4. Conclusions

The ignition temperature of electrodeposited elemental boron was determined. It was observed that the ignition temperature was influenced by p_{O_2}, the heating rate and the particle size of the elemental boron powder. The oxidation of boron becomes significant at temperature (783 K) if the particle size of the boron powder is less than 32 µm. The presence of carbon retards the ignition of boron. Even in the presence of oxygen, boron is not completely oxidized due to the formation of a glassy layer of boric oxide at the reaction interface. The glassy layer acts as a barrier for further diffusion of oxygen across the interface. Compared to amorphous boron, crystalline boron is more resistant to oxidation.

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