

## B<sub>4</sub>C solid target boronization of the MST reversed-field pinch

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A solid rod of hot-pressed boron carbide is being used as the source of boron during boronization of MST. The most striking result of this procedure is the reduction in oxygen contamination of the plasma (OIII radiation, characteristic of oxygen at the edge, falls by about a factor of 3 after boronization.). The radiated power fraction drops to about half its initial value. Particle reflux from the wall is also lowered, making density control simpler. The rod (12.7 mm diameter) is inserted into the edge plasma of normal high-power RFP discharges. B<sub>4</sub>C is ablated from the surface of the rod and deposited in a thin film (a-B/C:H) on the walls and limiters. The energy flux carried by “superthermal” (not “runaway”) electrons at the edge of MST appears to enhance the efficient, nondestructive ablation of the boron carbide rod.

### 1. Introduction and motivation

Many techniques have been used to reduce the interaction (or the detrimental effects of an interaction) between a high temperature magnetically confined plasma and the material boundary at the plasma edge. Substantial work has been done to perfect a method of covering the first walls of plasma confinement devices with a coating of boron and/or carbon. Most techniques employ a glow discharge in a carrier gas containing boron and/or carbon [1–3]. The use of tri-methyl boron as a carrier gas (instead of diborane) has lessened the safety hazard of these procedures [4], but the disadvantage remains that this type of coating must be rejuvenated often, sometimes between every full-power plasma discharge (DIII-D, [5]). Also, it is sometimes difficult to control the ratio of boron, carbon, and hydrogen in the wall coating [4]. The use of tri-methyl boron as a fraction of the fueling gas for full-power tokamak discharges on the Tokamak de Varennes shows promise, but still requires gas handling equipment and continuous monitoring [6].

The attractive features of using a solid target as a source of material for first wall modification became apparent during experiments on MST (Madison Symmetric Torus) in which a carbon (graphite) limiter was inserted to  $r = 0.77a$  ( $a$  is the minor radius of the torus). After several dozen full power discharges in this

configuration, it became apparent that the oxygen impurity level had dropped substantially, presumably because the limiter was acting as a source of carbon which was trapping and/or covering the oxygen on the first wall. Especially appealing was the inherent simplicity of this solid target technique and ease with which coating formation could be controlled (by inserting or withdrawing the target as necessary).

Shortly after the experience with the carbon limiter we became aware of the “solid target boronization” work being done on the Tokamak de Varennes by Hirooka [7–9]. This technique involves the use of a boronized carbon-carbon composite which contains about 40% boron and is shaped into a hemispherical target. Results of experiments in which this target was exposed to full-power tokamak discharges were positive, with less impurity contamination of the plasma and lower loop voltage being recorded.

We have further simplified and advanced the technique of solid target boronization on MST. Our target is simply hot-pressed B<sub>4</sub>C (78% boron by weight) placed in the edge plasma. Impurity contamination of the plasma has been reduced and uncontrolled refueling of the discharge has been eliminated. Our results also provide insight into the mechanism by which boron is ablated from the solid target. We observe that ablation of the boron carbide target is proportional to the energy flux reaching the target and that ablation does

not begin until the surface of the target has absorbed a substantial amount of energy.

## 2. Background

The MST reversed-field pinch (RFP) is a high temperature (electron and ion temperatures  $\leq 500$  eV), high plasma current ( $I_p \leq 600$  kA), moderate pulse length (60 ms), large ( $R = 1.5$  m,  $a = 0.52$  m) plasma confinement device [10,11]. The primary plasma-facing surface is aluminum; a small amount of this surface (about 10% [12]) has been covered with a combination of graphite and ceramic tiles and limiters that protrude out from the wall less than 2.5% of the minor radius. The plasma must be allowed to closely approach the 50 mm thick aluminum conducting wall in order to insure MHD stability (Ho [13,14], Alper [15]). This condition is in direct conflict with the need to limit plasma-wall interaction in order to reduce impurity influx and uncontrollable wall refueling of the plasma discharge. The tiles and limiters have been strategically placed to limit sputtering of the aluminum conducting wall; the major impurities in MST are oxygen and carbon. Helium and/or hydrogen pulsed discharge cleaning (rapid repetition of short, low energy plasma discharges – a variation of the technique known as “Taylor discharge cleaning” [16]) is used to remove impurities from the wall after the vacuum vessel has been opened to the atmosphere. However, even lengthy pulsed discharge cleaning has failed to lower oxygen contamination of RFP plasmas below a few percent [17]. Helium pulsed discharge cleaning between high current RFP discharges will remove hydrogen from the plasma-facing surfaces, but leaves behind an undesirable amount of helium. The obvious next step to the solution of these problems is the modification of the plasma facing surface with a low- $Z$  refractory material. To this end, we have successfully implemented an extremely simple solid target boronization procedure. Results have included a dramatic lowering of the oxygen impurity level in the plasma and improved density control of gas puff fueled hydrogen RFP discharges.

## 3. Experimental results

### 3.1. Half-disk boronization

Initial solid target boronization experiments (MST had never been boronized by any means prior to this experiment) were done using a 6 mm thick disk of

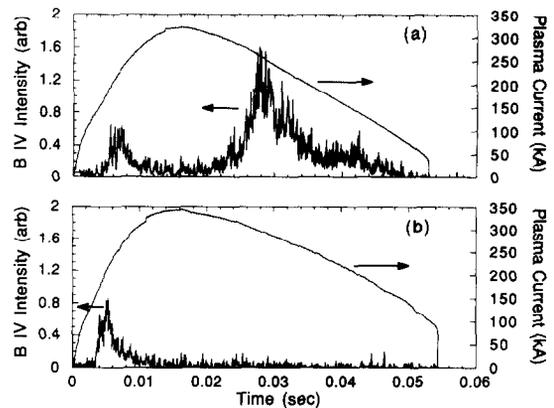


Fig. 1. BIV (282.17 nm) radiation recorded (a) during half-disk boronization and (b) after half-disk boronization. Note that during boronization the largest influx of boron occurs after peak plasma current, presumably because the B<sub>4</sub>C target must be heated before ablation begins.

hot-pressed B<sub>4</sub>C (boron carbide) purchased from Cerac, Inc. of Milwaukee, Wisconsin. The disk was cut along its 125 mm diameter and mounted in a graphite holder. About 50 helium fueled 310 kA RFP discharges were taken with the half-disk inserted into the edge plasma. Clear evidence of the influx of boron into the plasma is represented by the appearance of a large amount of 282.17 nm BIV line radiation during the equilibrium portion of the discharge (fig. 1a). The BIV line was observed at a location toroidally opposite the insertion location of the boron carbide, indicating that boron was transported throughout the plasma. Fig. 1b shows the behavior of the BIV line immediately after the half-disk was retracted. Note that this line “burns out” before the plasma current and electron temperature have peaked. This implies that the boron that enters the bulk plasma from the wall during startup is probably fully stripped and no longer radiates during the equilibrium portion of the discharge.

The results shown in fig. 1 also illustrate the observation that boron does not begin to ablate from the target and move into the plasma until the surface of the target has absorbed a substantial amount of energy. We consistently do not see spectroscopic evidence of boron ablation until well into the discharge, usually 10 ms after the peak plasma current. This result indicates that efficient ablation of a solid target does not begin until the surface has been heated to some level.

As a day-to-day indication of the impurity content of MST plasmas, we routinely monitor the 464.74 nm C III and 375.99 nm O III lines. These ionization states

Table 1  
MST operational parameters immediately before and immediately after boronization with half-disk B<sub>4</sub>C target

Observation	Before boronization	After boronization
Plasma current (kA)	310	327
Loop voltage (V)	17.2	16.3
Ohmic input power (MW)	5.33	5.33
Radiated power fraction (%)	30	17
Carbon III (arb.)	1.00	0.436
Oxygen III (arb.)	1.00	0.353
Surface barrier diode (arb.)	1.00	0.467

are primarily in the edge plasma during the equilibrium portion of the discharge, thus their intensity is a good indicator of impurity influx and recycling at the edge. The intensity of these lines was substantially lower following the boronization session described above (table 1). Also notable is the fact that soft X-ray emission (as monitored by a surface barrier diode with a 7.6  $\mu\text{m}$  Be filter) was substantially reduced. This strengthened the supposition that such emission was primarily from oxygen impurities in the plasma. The radiated power fraction of the ohmic input fell from 30 to 17%. (Total radiated power was measured with an absolutely calibrated bolometer of the type used on the ZT-40 reversed-field pinch [18].) Note, however, that the loop voltage fell only slightly; this observation will be discussed in more detail in the next section. A difficulty with this experiment (later corrected – see the experiment described below) is the fact that neither electron density or temperature measurements were available. Even though the helium fueling rate was kept constant before and after boronization, it is likely that wall refueling (from impurity influx and recycling) fell. Post-boronization discharges exhibited the characteristics (such as early termination) of low density RFP discharges. Thus, part of the drop in impurity radiation may have simply been due to an overall drop in density. It is clear, however, that impurity content and edge recycling were lowered by this boronization procedure.

Auger analysis of a 100 mm<sup>2</sup> aluminum surface placed flush with the aluminum wall of MST at a location toroidally opposite the half-disk of boron carbide revealed the presence of a 10 to 15 nm layer of boron, carbon, and oxygen. After a short period of argon sputtering to remove the atmospheric contamination layer, the atomic concentrations of B, C and O were found to be in a ratio of 1:2:2. (Control samples

exposed prior to boronization exhibited coatings containing similar amounts of carbon and oxygen but no boron.) The ratio of boron to carbon is remarkably large given that the amount of graphite in the tiles and limiters is much larger than the amount of boron carbide exposed to the edge plasma. This ratio compares favorably with that achieved by gaseous boronization [2,4] and was the first indication that hot-pressed boron carbide could be effectively sputtered and transported by the MST plasma. Also, although we have no direct evidence, it is likely that the boron transported by the plasma is not evenly deposited on the walls and limiters. Work done on the Tokamak de Varennes suggests that boron will be preferentially deposited on areas facing the ion drift direction [6,9]. This may be beneficial if these areas and limiters are those which sustain substantial interaction with the edge plasma.

The only practical difficulty with the above experiment was the thermal stress cracking and destruction of the half-disk of boron carbide during its 50-shot exposure to the MST plasma. Our initial attempt to overcome this problem was to place an identical half-disk in the 4 ms, 100 kA pulsed discharge cleaning (PDC) plasmas that are run every night on MST. Positive results such as lowered impurity content and less wall fueling were achieved in subsequent RFP discharges, but the effects were not dramatic. Auger analysis of aluminum surface samples placed flush with the wall failed to reveal any coating of boron. It is possible that the efficacy of this method could be improved by positively biasing the boron carbide target placed in the PDC plasma. A bias of several hundred volts might enhance heat flux to the target and result in more effective boronization of the walls and limiters. However, we have taken a different direction and directly solved the thermal stress cracking problem by modifying the shape and structure of the boron carbide target placed in the RFP plasma.

### 3.2. Rod boronization

The boron carbide target that has proven resistant to thermal stress cracking is in the shape of a cylindrical rod 12.7 mm in diameter. It was vacuum hot-pressed by Cerac, Inc., from 99.5% pure 4.5  $\mu\text{m}$  B<sub>4</sub>C particles. Apparent density was held to 70% of that theoretically achievable. This was done to lower the probability that stress cracks would propagate and fracture the bulk material. (We have not seen large fractures develop in the rod.) Lowering the density of hot-pressed B<sub>4</sub>C does increase the apparent porosity of the material, but

problems have been avoided by baking the rod and allowing it to outgas in vacuum for several days before exposure to plasma.

The B<sub>4</sub>C rod holder benefited from the careful design shown in fig. 2. The boron nitride collar prevents damage to the holder and ablation of the base of the B<sub>4</sub>C rod. The rod itself is held in the holder by a pin, a scheme which allows the rod to thermally expand and contract without encountering mechanical stress. Note that the rod is in electrical contact with a graphite holder and can be electrically biased (which we have not done – all boronizing has been done with the rod at the same potential as the aluminum wall). The insertion of the rod is precisely controlled by a stepping motor actuator. The rod itself can be withdrawn to a viewing window while still under high vacuum, a feature that allows regular inspection of the ablated surface.

The B<sub>4</sub>C rod has typically been inserted to a depth of  $r = 0.9a$ . This is slightly past the edge plasma scrape-off layer. Ablation of boron carbide is confined largely to the end of the rod and is highly asymmetric. After approximately 100 RFP discharges, the planar surface at the end of the rod is no longer perpendicular to the cylindrical axis, but is ablated away to a surface that forms a 35–45° angle to the axis. This surface faces the flow of “superthermal electrons” in the edge of MST and is strong evidence that this electron population is primarily responsible for the efficient ablation of the boron carbide rod. We have set up a monochromator to directly view the rod and the plasma in the immediate vicinity and have found that bursts of B III (449.77 nm) line radiation occur coincident with the sawtooth events that generate toroidal magnetic flux and correlate with bursts of “superthermal electrons.”

The phenomena of an edge population of “superthermal electrons” has been studied extensively in MST

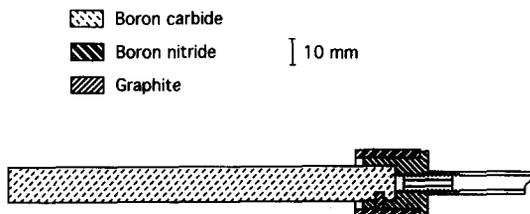


Fig. 2. The B<sub>4</sub>C rod holder. Not shown in this drawing is the thermocouple probe placed into the base of the B<sub>4</sub>C rod. This probe allows the rod temperature to be monitored following a shot.

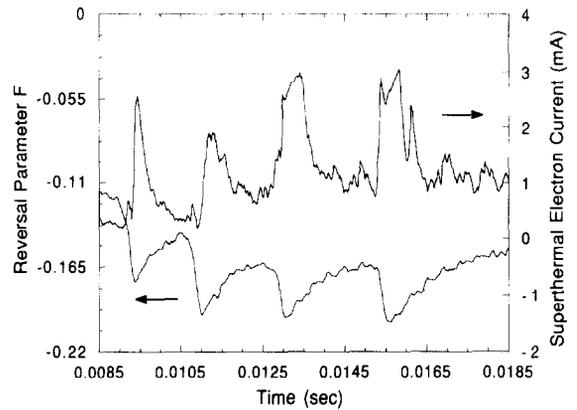


Fig. 3. The bursts in superthermal edge electron current occur coincident with the sawtooth events that generate toroidal magnetic flux. (The reversal parameter  $F$  is the ratio of edge to average toroidal magnetic field.)

[19,20] and other reversed-field pinches [21–23]. These “superthermal electrons” are not similar to the high energy “runaway electrons” seen in tokamaks. In brief, this population flows along the field lines at the edge of MST and is characterized by a parallel temperature similar to that of the bulk electrons at the core of the plasma. The density of these electrons is 5–20% of the bulk electrons in the edge plasma, the parallel temperature about three times that of the bulk edge electrons. These “superthermal electrons” carry much of the current that flows in the edge region of an RFP. (See fig. 3 for an example of a measurement of this current [20].) Initial measurements of the heat flux carried by this electron population have been done with a LiTaO<sub>3</sub> pyro-electric bolometer [24]. The “superthermal electron” heat flux during a sawtooth event is at least 5 W/mm<sup>2</sup> at an insertion depth of 0.95 $a$ , cf. fig. 4. It is also clear from the same measurements that this heat flux has a strong positive gradient with insertion depth; at 0.9 $a$  the heat flux is probably several times larger. This gradient is in fact an advantage, as it is possible to precisely control the ablation rate of the boron carbide rod by adjustments of the insertion depth. (The ablation rate is strongly correlated with the intensity of the B III line as seen by the viewing monochromator.)

Note, however, the flux of energy to the boron carbide rod is key to efficient ablation, not the presence of “superthermal electrons.” Fig. 4b illustrates the fact that boron is being ablated away from the rod even between the bursts of heat flux carried by the “superthermal electrons”. This implies that this tech-

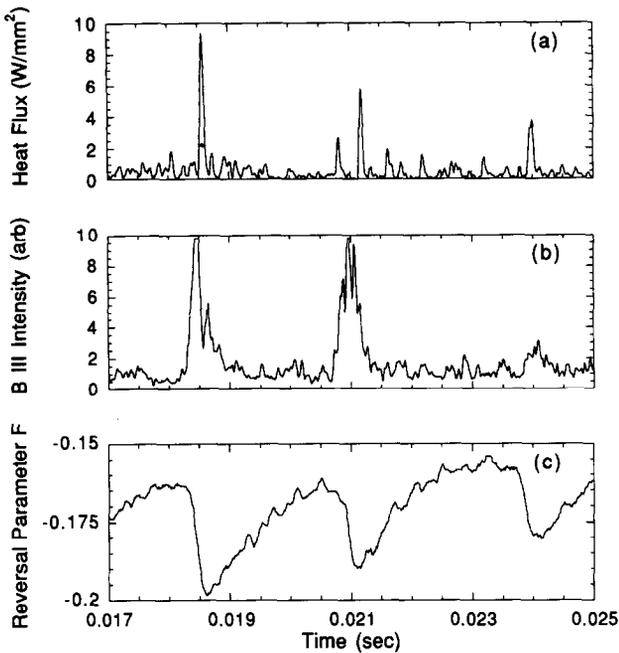


Fig. 4. An illustration of the coincidence of (a) bursts of heat flux due to superthermal edge electrons (measured with a pyroelectric bolometer), (b) bursts of B III (449.77 nm) radiation, and (c) sawtooth events that generate toroidal magnetic flux. The heat flux scale in (a) may be underestimated by a small amount; the sensitivity of the bolometer fell as it was boronized.

nique of solid target boronization will work in any plasma that can provide a heat flux to a target.

Auger analysis of a 100 mm<sup>2</sup> aluminum surface sample placed flush with the wall during  $B_4C$  rod boronization indicates the presence of boron and/or boron oxide in the surface coating. Some of the samples were intentionally exposed to several hundred RFP shots and several nights of PDC discharges. The thick coating that formed was opaque (rather than the refractive blue or yellow seen on thinner coatings). This thick coating was insulating, making Auger analysis difficult because of charge buildup on the surface. This thick coating was also significantly harder than the aluminum substrate and resistant to scratching. These characteristics lead us to believe that these coatings are amorphous (of the form a-B/C:H) and similar to those described by Winter [1,2].

Most of the  $B_4C$  rod boronization experiments have been done in 350 kA hydrogen RFP discharges, although the technique seems to work well in both higher and lower current discharges. The carbon and oxygen impurity contamination of the plasma is much

lower when MST has been extensively boronized, cf. table 2. (“Extensive boronization” means that the  $B_4C$  rod had been inserted to  $r = 0.9a$  for 500 RFP discharges – no further drop in impurity contamination was recorded after this point.) Note that even though carbon is being ablated from the rod into the plasma, the carbon contamination of the plasma drops as a result of boronization. As suggested by Winter [2], this is probably a result of the lowered oxygen contamination of the plasma. (Oxygen is probably the agent which most strongly sputters material off the graphite tiles and limiters in MST. These tiles and limiters are in the scrape-off layer and do not appear to receive significant heat flux from the “superthermal electrons.”) We have also been able to study the changes that occur in the soft X-ray spectrum as a result of boronization. As is shown in table 2, the intensities of the  $OK\alpha$  and  $AlK\alpha$  lines are lower (The magnitude of the drop in  $OK\alpha$  is suspect due to fitting uncertainties in the analysis of the Si(Li) detector soft X-ray spectra, but a substantial decrease is unquestionable.). There is little doubt that oxygen is gettered and held in the a-B/C:H coating.

Once MST has been boronized, it is possible to maintain the coating by slightly retracting the  $B_4C$  rod to  $r = 0.94a$  and allowing a small amount of boron carbide to be ablated into the plasma during every RFP discharge. Once formed and maintained, the a-B/C:H coating improves the electron density control and reproducibility that can be achieved with simple hydrogen gas puff fueling. Edge recycling and refueling

Table 2

MST operational parameters during non-boronized conditions and during well-boronized conditions (no further decreases in the impurity levels with further boronization). Solid target boronization was done with the 12.7 mm  $B_4C$  rod. Central electron temperature was not available when this set of non-boronized data was taken

Observation	Non-boronized	Well-boronized
Plasma current (kA)	312	338
Loop voltage (V)	19.4	17.6
Ohmic input power (MW)	6.05	5.95
Radiated power fraction (%)	48	30
Carbon III (arb.)	1.00	0.478
Oxygen III (arb.)	1.00	0.383
Oxygen $K\alpha$ (arb.)	1.00	0.038
Aluminum $K\alpha$ (arb.)	1.00	0.582
Chord-averaged electron density (/m <sup>3</sup> )	$1.5 \times 10^{19}$	$1.5 \times 10^{19}$
Electron temperature (eV)	120 @ $r/a$ = 0.5	140 @ $r/a$ = 0.0

appears to be dramatically lower, as the gas puff fueling rate must be increased by a factor of 2 to 3 over that needed to achieve similar electron densities prior to boronization. The ease with which MST can be reproducibly operated near the RFP density limit [25] has also been improved – previous attempts to operate near this limit were made difficult by the tendency of the walls and limiters to load with hydrogen and then uncontrollably release it during a discharge.

The improvements due to boronization of MST cited above are similar to those seen in most boronized tokamaks. However, there is one distinct difference. We have not observed any distinct improvement in the discharge parameters related to energy confinement. In particular, the loop voltage recorded before and after boronization is almost identical in MST. This is in direct contrast to the experience in tokamaks, where, for instance, in the Tokamak de Varennes, the loop voltage dropped by a factor of 40% following solid target boronization [9]. This is probably due to the fact that the loop voltage in reversed-field pinches is anomalously high [26]. Typically, the loop voltage in MST is four times that expected from an application of Spitzer resistivity to model current profiles. The supposition that this resistivity enhancement is not due to a large value of  $Z_{\text{eff}}$  is supported by our boronization experience; analysis of Si(Li) spectra indicate that  $Z_{\text{eff}}$  is 65% lower following boronization. (We are in the process of constructing an array of absolutely calibrated bremsstrahlung detectors to accurately measure the  $Z_{\text{eff}}$  profile.) It is likely that any reduction in Spitzer resistivity due to a drop in impurity content of the plasma was small compared to that portion of the loop voltage determined by non-Spitzer (anomalous) factors. We have also measured the product  $n_{e0}T_{e0}$  by Thomson scattering following boronization and found no change; thus we conclude that boronization has not improved the energy confinement time of MST.

#### 4. Conclusion

Results from MST have demonstrated that boronization can be accomplished simply, safely, and effectively with a solid target of hot-pressed B<sub>4</sub>C. High purity hot-pressed boron carbide is readily available; we suggest that the apparent density of the solid target be lowered to about 70% to improve resistance to stress cracking. An advantage of boronization targets made of pure B<sub>4</sub>C is that the ratio of boron to carbon (78% by weight) is higher than boron containing amorphous C–C composites. Solid target boronization has

also been easy to implement on MST, as we have not had to deal with the gas handling systems and safety equipment associated with diborane or tri-methyl boron.

The efficacy of B<sub>4</sub>C solid target boronization has been demonstrated in MST. Results of our boronization experiments have paralleled those obtained in other boronized fusion research devices. Impurity contamination of the plasma (especially by oxygen) has been reduced and uncontrolled refueling of the discharge by the particles on the wall has been eliminated. Particularly interesting is the observation that the heat flux carried by the edge “superthermal electron” population (characteristic of reversed-field pinches) enhances the efficient ablation of the boron carbide target. This mechanism makes it easy to control the ablation rate by varying the insertion depth of the target. Boronization can be done quickly with deep insertion (where the heat flux to the target is large), then the target retracted to the point where ablation is just large enough to maintain the wall coating. Our experience indicates that this technique of solid target boronization should work well in any plasma in which there is a moderate flux of energy (about 10 W/mm<sup>2</sup>) to a B<sub>4</sub>C target placed within the plasma.

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