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STTR FY2003.1 Proposal No: F033-0354 Contract No. F49620-03-C-0090 May 30, 2004 The Use of Boron Nitride for Improved Cold-Cathode

Electron Field Emission Technology – Final Report

### 1. INTRODUCTION

Low-power Hall thrusters potentially offer important advantages for certain military applications. An important factor impacting efficiency is that the state-of-the-art techniques for electron generation used in propellant neutralization (such as hollow cathodes operating on the same propellant as the thruster) do not scale down in mass, power, and propellant consumption as readily as the miniaturized thrusters themselves. The work under this contract evaluated a possible solution utilizing Boron Nitride thin films (BN) primarily in the cubic phase (cBN) as a chemically inert, mechanically tough, low work-function material, for improved cold-cathode electron field emission technology. The desirable characteristics of leading electron emission materials such as molybdenum tips and Carbon Nanotubes (CNTs) are well known. However, the chemical reactivity of these materials in oxidizing environments, especially carbon, presents significant limitations in both operational and handling factors with respect to their application in Hall thrusters and other propulsion technologies in certain environments. The propellantless nature of this approach eliminates the neutralizer's potential contribution to efficiency degradation and its superior material properties offer the possibility of long lifetime operation.

Our previous studies indicate that cBN exhibits all of the superior properties of CNT as a field emitter, as well as having the additional advantage of chemical stability in oxidizing environments such as may be encountered in space propulsion applications. In particular, a comparison with existing coldcathode electron emitters shows that cBN should perform at background pressures some 2 orders of magnitude higher than the pressures at which CNT typically operate ( $\sim 10^{-5}$  Pa). In Phase I the BN cathode was consistently operated with higher background pressures (up to  $10^{-3}$  Pa) of oxygen, xenon and water vapor. These cathode emission characteristics changed slightly at elevated pressures of oxygen but reverted to their original behavior when returned to the low-pressure operation. Under typical operating conditions, only a few volts per micron were needed to initiate emission.

#### 2. PROJECT OBJECTIVES

The work is divided into three primary tasks as described in the proposal of this contract including:

- 1) Evaluate the emission characteristics of BN
- 2) Assess, model, and design BN electron emitter for low-power Hall thruster application.
- 3) Develop a plan for Phase-II prototype development and testing of BN electron emitter.

The timeline that was outlined along with the tasks is shown in Table 1.

	Month								
TASK	Sept	Oct	Nov	Dec	Jan	Feb	Mar	Apr	May
Organize Initial Testing Plan									
Fabricate cBN samples	-								
Conduct Emission Tests			4						
Develop Design Concept									
Develop Phase II Fabrication Plan							2		
Develop Phase II Test Plan									
Status Reports	x				х			х	
Final Report					4				3

<b>Table</b> (	1 -	Timeline	for	the	Phase-I	STTR	Project
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#### 3. WORK CARRIED OUT AND RESULTS

### 3.1 Evaluate the emission characteristics of BN

# 3.1.1 Organize Initial Testing Plan

The initial subtask of developing a test plan for this phase was accomplished. The proposed work included testing a cBN sample in the environment of  $O_2$ , Xe, air and  $H_2O$  vapor. These tests serve as a baseline for further evaluations which will be carried out in Phase II using a MEMS machined array of holes in between the cBN sample and the anode. We were able to accomplish the testing plan as it was laid out for Phase I.

#### 3.1.2 Fabricate cBN samples

Initial samples of the cBN have been produced and were prepared for insertion into the field emission test chamber. Figure 1 shows one of these samples. The scale in the background is in inches. These samples are a thin film of cBN deposited on a conductively doped silicon wafer.



Figure 1: cBN Sample

Of the samples fabricated, nine boron nitride (BN) thin film samples were identified for possible emission threshold measurement and characterization. These films were selected based primarily on their structure phase, determined using Fourier transform infrared spectroscopy (FTIR). The surface morphology of the samples, in terms of nanostructured tip arrays favorable for electron emission, was also an important characteristic for these samples, and was characterized by means of Atom Force Microscopy (AFM). A typical surface imaged by AFM is shown in Figure 2.

### 3.1.3 Conduct Emission Tests

#### 3.1.3.1 Experimental Setup

Some of the cBN samples produced were prepared and mounted in the vacuum chamber for testing. The setup of the sample/anode is depicted in Figure 3. The cBN film surface is separated from a phosphor screen and anode by  $25.4 \mu m$  diameter glass fibers. The phosphor enables the imaging of the electron emission, and was not used in the testing in this Phase. Figure 4 is a photograph of a sample mounted. The sample is sandwiched using clamps manufactured of an Ultra High Vacuum (UHV) compatible insulating material. There is a slit in the aluminum holder so that the phosphor is visible from the opposite side. The wire leads are visible from the back side of the cBN sample and from the anode.



Figure 2: AFM imaging of surface of a typical BN film prepared by ion-beam assisted sputter deposition a) side view showing a self-assembled array of sharp BN nanotips (vertical scale = 144 nm). Mean roughness = 5.43 nm; b) plan view showing lateral extent of tips ~20 nm giving a density of tips  $\sim 10^{11}$  cm<sup>-2</sup>.



Figure 4 – Picture of Sample/Anode Setup

Figure 3 – Sample/Anode Setup

Figure 5 shows the overall experimental setup. A UHV system capable of a base pressure (without the presence of any environmental gases) of 10<sup>-8</sup> Torr or lower contains the sample viewed from the front window. Oxygen and other gases can be introduced through a gas manifold and then measured using a Residual Gas Analyzer (RGA) in order to determine the partial pressures of each gas species.





Figure 5 – Field Emission Measurement Setup

# 3.1.3.2 Experimental Results

Of the nine BN samples identified, four samples have been analyzed (SN104, 124, 133 and 134). Field emission measurements were taken at base pressure,  $\sim 10^{-8}$  Torr. In the results shown in Figure 6 the anode-sample field was increased slowly (0.1 V/µm per minute) and the emission current was recorded at each value of the field. Occasionally we observed that the limits of emission for a given emission site would be exceeded, ostensibly resulting in that emission site's irreversible extinction. Consequently, for subsequent tests we placed an upper limit on the emission current of 10 µA. Evidence for emission from localized sites can be observed in the upper left and right panels of Figure 6, where small peaks in the emission can be seen prior to the main turn-on point.

The measured field emission threshold for these films varies from ~0.5 V/ $\mu$  to ~37 V/ $\mu$ , with the difference being attributed to the following:

- Variations in the surface morphology of the films, which can contribute significantly to field enhancement experienced by emitted electrons at an emission site on the film surface.
- The possible effects of adsorbates (e.g., hydrogen or water vapor) on the BN surface.



SN104						
FE Chamber Pressure	$\sim 8 \times 10^{-8}$ torr					
Emission Threshold	37.9 V/μ					
SN133						
FE Chamber Pressure	$\sim 2 \times 10^{-7}$ torr					
Emission Threshold	3.07 V/μ					
SN134						
FE Chamber Pressure	$\sim 1.5 \times 10^{-7}$ torr					
Emission Threshold	26 V/µ					
SN124						
FE Chamber Pressure	$\sim 1.7 \times 10^{-7}$ torr					
Emission Threshold	0.55 V/µ					

Figure 6: Threshold fields for several BN field emission samples.

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Experiments to better characterize the effects of these two possible contributors to the field emission of the boron nitride films were performed. These experiments included a careful analysis of the field emission of a particular emission site on a given sample at a field *slightly below and above* the emission threshold field, allowing for a systematic analysis of the robustness and stability of the emission site. The samples were evaluated by maintaining a constant voltage once emission occurred rather than continuing to increase the voltage. Once stable emission is achieved, gases such as oxygen, air, Xe and water vapor were introduced to evaluate the performance during and after gas exposure. In this way we could test the stability of emission in terms of sensitivity to various environmental gases that are likely to be encountered under actual operating conditions. Additionally, a system has been designed to heat samples *under UHV conditions* to remove any physisorbed substances that may affect subsequent field emission measurements. This process should facilitate establishing handling procedures for BN electron emitters. It is interesting to note that sample SN124 was tested over 2 years prior to these tests and similar emission characteristics were achieved without requiring any special storage or handling (simple closed container storage with no special purging). This attests to the excellent chemical and physical stability of BN emitters.

# 3.2 Assess, model, and design BN electron emitter for low-power Hall thruster application.

There are four major questions addressed by this task relating to the integration of the electron emitter to the low power Hall thruster system. The first question is, "what is the ideal geometry (location and size) for neutralization?" There are several factors that affect what this might be. Optimum size and location are influenced by the magnetic field of the thruster, space charge limitations, mechanical integration and thermal dissipation. These affect not only the efficiency of the thruster, but also its manufacturability. Three concepts have been fleshed out regarding the integration of field emitter devices into Hall thrusters (see Figure 7). These are 1) simply mounted to the exterior of the spacecraft near a Hall Thruster, 2) mounted annularly around the external radius of the thruster and 3) mounted on the exterior surface of the thruster's cylinder.



Figure 7 Proposed neutralizer locations

One of the major factors influencing the location of the neutralizer is the magnetic field of the Hall thruster. Design 1 or 2 could place the neutralizer along field lines that would lead into the thruster. However, design 3 would require the electrons to cross field lines in order to be utilized. Figure 8 depicts how a field line would cross through the neutralizer and direct the electrons to the ionization region in the second design. The location relative to the magnetic field will have a significant influence in determining the usefulness of the electrons produced. Additional work on the magnetic field circuit might further improve the ability of the cathodes to produce electrons for ionization. Plans for Phase II include modeling and measuring the magnetic field in order to better determine how the thruster's magnetic field can be modified to enhance the collection of the electrons from the neutralizer. An assessment will also be made of the current magnetic field in use with additional circuit designs evaluated.





Figure 8 Field Lines on a Hall Thruster

Designs 1 and 2 should also allow for adequate management of heat dissipation from the thruster. Design 1 allows for the cathode to be placed almost anywhere on the spacecraft, but Design 2 enables the cathode to remain part of the Hall thruster. The final design may not be completely annular since that much area may not be needed. The third design would be more problematic from a thermal standpoint since it would hinder heat dissipation from the thruster. The exact location will be facilitated by the testing in Phase II.

The size of the emitter that will be needed will be driven by space charge limitations and the amount of current required for the thruster. The space charge limitation on the cathode is driven primarily by the plasma density, the electron temperature, the gate voltage and the cathode potential.<sup>1</sup> Beal tested a 200 W thruster from Busek and found the electron temperature to be around 1-2 eV.<sup>2</sup> He also measured the plasma density as shown in Figure 9. Marrese determined that a Hall thruster with a plasma density of  $8 \times 10^8$  /cm<sup>3</sup>, an electron temperature of 1 eV, gate potential of 30 V and a cathode potential of 20 V could support a maximum electron emission current of 17 mA/cm<sup>2</sup> to avoid space charge limitations. This was based on measurements for an external cathode on a 1.4 kW thruster. However, the measurements shown in Figure 9 indicate plasma densities on the order of  $5 \times 10^{10}$  /cm<sup>3</sup> around the location of an external cathode. Marrese calculated a maximum current for this density to be 1700 mA/cm<sup>2</sup> to avoid space charge effects based on previously mentioned temperatures and densities. This model was also based on a planar sheath that was the most conservative in estimating maximum current.



Figure 9 - Plasma density in the far-field plume of a BHT-200 measured using a triple probe.<sup>2</sup>

Beal measured the discharge current to be nominally 0.8 A for the BHT-200 thruster. Using the worst case scenario of  $17 \text{ mA/cm}^2$ , the minimum size of the cathode would need to be  $47 \text{ cm}^2$ . However, the more realistic size of the cathode based on the higher plasma density would be  $0.5 \text{ cm}^2$ . The performance of the BN field emitter cathodes is expected to be  $10-100 \text{ mA/cm}^2$ . We anticipate therefore that space charge will not be the limiting factor in the emitter size. The BN field emitter cathode would thus be  $1-10 \text{ cm}^2$  for the 200 W Hall thrusters. driven primarily by the capacity of the emitter.

The second question addressed was determining the necessary biasing requirements. We are still working to refine the answer to this question. Currently, we have found that we are able to initiate emission at 1-2 volts per micron. Our goal would be to maintain this low threshold electric field and correspondingly low flight gate voltage. The density of emission sites and the overall current requirement will resolve the final gate voltage value. Further testing in Phase II will determine these voltages for the case when many emission sites are simultaneously active. We have designed a MEMS gate structure to facilitate these measurements. Testing in phase II will also determine the optimum cathode potential relative to the thruster. Currently, we assume that bias to be similar to that currently used for the hollow cathodes ( $\sim$ -5 to -20 V). These voltages will contribute significantly to the resulting efficiency of the thruster Using Equation 1 we can determine the approximate efficiency of the thruster based on its cathode potential.<sup>3</sup>

$$\eta = \frac{a}{1 + \frac{b + V_{cath}}{V_D}}$$
 EQ (1)

Using Equation 1, where  $\eta$  is the efficiency, *a* is approximately 0.8 (unity minus the electron loss ratio), *b* is the ion production cost (100 eV/ion, worst case) and V<sub>D</sub> is the discharge voltage (250 V), we can plot how efficiency varies with the cathode potential. Figure 10 depicts the resulting *total* efficiency for the potential range anticipated for the BN cathode (Note: gate potentials are also included). The figure shows that 200 W Hall thrusters using hollow cathode technology have efficiencies from 20% to 45% (nominally 42%).<sup>4</sup> Since our testing currently demonstrates that we should have potentials less than 50 V, we anticipate improving the efficiency of the thruster by up to 30%.

The specific impulse (Isp) can be calculated based on the efficiency. Equations 2 and 3 show this relationship.

$$\eta = \frac{1/2}{m_A(1 + \frac{\dot{m}_C}{\dot{m}_A})(gIsp)^2}/Pin \qquad EQ(2) \qquad \Rightarrow \qquad Isp = \sqrt{\frac{2\eta Pin}{\dot{m}_A(1 + \frac{\dot{m}_C}{\dot{m}_A})g^2}} \qquad EQ(3)$$

Figure 10 also includes the specific impulse expected using the BN emitter. The power corresponds to the 200 W thruster using the nominal flow rate ( $\dot{m}_A$ ) of 0.74 mg/sec reported for the Busek Hall thruster.<sup>4</sup> Note that the cathode flow rate ( $\dot{m}_C$ ) will be 0 for the BN emitter. The nominal specific impulse for the Busek thruster was reported as 1570 sec (ranging over 1200-1600 sec). Figure 10 depicts an expected improvement of at least a few hundred seconds.



Figure 10 – Total Efficiency and I<sub>sp</sub> versus Cathode Potential for 200 W Hall Thruster with BN Emitter.

The third question addressed by this task was identifying what protection is needed for the BN device in integration and ground processing. We have found that minimal protection is needed with the BN samples. The samples are currently stored in plastic containers under ambient conditions. These samples have consistently performed even after storage of a few years. However, we must also consider contamination issues from particulate matter which could be an issue depending on the gate structure used with the BN material. There is also the issue of how well these samples recover from operating in higher pressure environments ( $10^{-5}$  torr). Fig. 11 shows the BN emitter performance during and after the introduction of Xe gas, a common propellant in space propulsion applications.

The main result of this test is that there is no significant change of the emission yield either upon introduction of the Xe gas nor on removal of the Xe gas. Similar results (i.e., no change in residual gas) were obtained for Air.

We have also tested the performance of BN emitters in the presence of molecular oxygen. The results are shown in Figure 12. On introduction of oxygen we observed a ~30% drop in emission current which thereafter remains constant for at least several hours at constant background pressure of ~2 µtorr. However, upon removal of the oxygen gas (after ~7.5 hours) the original emission current is fully restored. It is interesting to note that there appears to be a time lag of about 30 minutes between removing the gas and recovery of the emission (see Figure 12). We believe this effect is due to surface adsorbed water vapor which slowly desorbs from the surface when the vacuum conditions return to UHV. A residual gas analysis (RGA) spectrum (Figure 13) showing the presence of residual water vapor supports this contention.



Figure 11: Exposure of BN emitter to Xe gas at 5µtorr followed by gas removal. The gas is pumped out after about 2.8 hours of exposure and monitoring continued for a further 9 hours. No deterioration of the emission is observed. Note also that there is no significant change in emission due to a transient overshoot in pressure (to >100 µtorr) that occurred on introduction of the Xe gas. Red curve: Gas pressure; Blue curve: emission current.



Figure 12: Emission characteristics (blue curve) at constant field (~5 V/ $\mu$ m) for nanostructured BN surface in the presence of oxygen (2  $\mu$ torr) followed by removal of the oxygen gas. The final pressure is ~10<sup>-8</sup> torr.



Figure 13: RGA scan showing water vapor as well as the expected oxygen peak.

We investigated the effects of presence of water vapor in more detail. Figure 14 shows the emission current from nanostructured BN in a partial pressure of water vapor equal to  $\sim 10 \mu$ torr followed by removal of the water vapor by evacuating the measurement chamber.

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Figure 14: Emission from BN in water vapor. See Figure 15 for an expanded view. Red curve: gas pressure; blue curve: emission current. The BN emitter seems to be very insensitive to the presence of water vapor at the levels tested here (< 10  $\mu$ torr), although a slight enhancement is apparent (see Figure 15). The transients in gas pressure are due to difficulties in manually adjusting the pressure to a set value. Even with large pressure overshoots (mtorr) the emission is very stable.



Figure 15: Exploded view of emission current (blue curve) from BN sample in atmosphere of water vapor followed by removal of gas. Note the small (few %) enhancement of the emission yield in the presence of water vapor.

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### **3.3 MEMS Prototype Emitter**

The final question addressed in this task was the design of the details for a prototype emitter to be used in Phase II for demonstration testing. Several versions of the emitter have been designed for use in Phase II. The basic design is depicted along with testing apparatus in Figure 16. The bottom layer of the emitter consists of BN deposited onto conductively doped silicon (depicted in orange). A MEMS gate structure (light blue) is then placed on top of the BN to facilitate extraction of the electrons. An exploded view of the gate structure is also depicted in Figure 17. The thickness of the dielectric is on the order of 1  $\mu$ m and will allow a maximum of ~1000 V to be applied between the gate and the BN emitter surface, which is significantly higher than what will be needed.





# Figure 17 - MEMS Gate Structure

The MEMS gate structures are designed with areas that are populated with holes  $1-2 \mu m$  in diameter. These areas are depicted in Figure 18. The reason for the thicker portions between these areas is for structural integrity. To facilitate testing several versions of these gate structures are being produced. Table 2 lists the various layouts that are planned to be produced for testing with the BN samples.

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Figure 18 - Layout of Emission Areas on MEMS Gate Structure

Sample Number	Hole Diameter (µm)	Hole Spacing (µm) Center-to-center	Approximate Number of Holes on Sample*	Approximate Current Performance**
Α	1	5	444,411	8.9 mA
В	1	8	174,636	3.5 mA
С	1	10	112,211	2.2 mA
D	2	5	444,411	8.9 mA
Е	2	8	174,636	3.5 mA
F	2	10	112,211	2.2 mA
G	2	300	99	2 μΑ
Ι	1-2 (TO-5)	8	15,876	0.3 mA

 Table 2 - Various Gate Structure Designs

\*Note: These numbers assume 11 emission openings of 1 mm<sup>2</sup>

\*\*Note: These numbers assume 0.1 μA/hole with 20% emission efficiency (0.1 μA/hole is independent on hole diameter)

#### 3.3 Develop a plan for Phase-II prototype development and testing of BN electron emitter.

The plan we developed for Phase II is broken into two stages with a set of initial tasks leading into the prototype development tasks. At each stage the tasks are related to 1) system definition/planning/reporting, 2) Technology related to BN use for electron field emission, 3) cBN-MEMS field emitters, and 4) integrated thruster/plasma testing.

The goal related to the system definition is to have a clear, up-to-date evaluation of the requirements for the emitter. The goal for the second task is to determine and provide consistent BN samples for integration into the emitters. The third task is to identify and produce the related hardware for the emitter (i.e. MEMS gate structure). The point of the fourth task is to test the environment around the Hall thruster with and without the emitter to demonstrate and improve the emitter. These tasks are broken down in the two stages as follows:

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# 1. Initial Tasks

- 1.1 System definition/planning/reporting
  - 1.1.1 Establish system requirements
    - 1.1.1.1 Low power Hall Thruster
    - 1.1.1.2 Electrodynamic Tether
  - 1.1.2 Conduct integration issues requirements analysis
  - 1.1.3 Refine neutralizer concept for Hall Thruster and associated market analysis
  - 1.1.4 Refine neutralizer concept for ED Tether and associated market analysis
  - 1.1.5 Provide interim report
- 1.2 BN Technology for Electron Field Emission
  - 1.2.1 Establish material refinement plan
  - 1.2.2 Upgrade BN fabrication chamber
  - 1.2.3 Fabricate and conduct tests for enhanced nanostructures
  - 1.2.4 Conduct surface contamination effects testing
- 1.3 cBN-MEMS Field Emitter
  - 1.3.1 Configure test chamber for cBN-MEMS emitter testing
  - 1.3.2 Design and produce updated prototype MEMS gate structure for electron emission testing
  - 1.3.3 Characterize cBN-MEMS structure performance
  - 1.3.4 Conduct laboratory performance tests and demonstrations (emission levels, space charge effects, etc.)
  - 2.4.5 Conduct robustness tests and initial life evaluation
- 1.4 Integrated thruster/plasma testing (Plasma and Electric Propulsion Lab –PEPL- at University of Michigan)
  - 1.4.1 Develop and implement testing system for integrated operation with low-power Hall thruster and plasma sources

## 2. Engineering Prototype Development Tasks

- 2.1 System definition/planning/reporting
  - 2.1.1 Revise system requirements and market analysis
  - 2.3.1 Revise material and integrated architecture plan as needed for performance and producibility
  - 2.1.2 Provide Interim Report
  - 2.1.3 Provide Final Report
- 2.4 BN Technology for Electron Field Emission
  - 2.4.1 Update growth chamber for larger surface areas and enhanced nanostructures
  - 2.4.2 Produce second generation BN structures
  - 2.4.3 Conduct laboratory performance evaluation of second generation material
- 2.5 cBN-MEMS Field Emitter Engineering Prototype
  - 2.5.1 Produce second generation MEMS structures
  - 2.5.2 Design and fabricate engineering prototype
    - 2.5.2.1 Mechanical (structure and protection covering)
    - 2.5.2.2 Thermal factors
    - 2.5.2.3 Bias circuitry
  - 2.5.3 Conduct laboratory performance tests and demonstrations
  - 2.5.4 Conduct robustness and initial engineering prototype lifetime evaluation
- 2.6 Integrated thruster/plasma testing (PEPL at University of Michigan)
  - 2.6.1 Conduct field emitter thruster interactions testing
  - 2.6.2 Conduct field emitter placement optimization testing
  - 2.6.3 Conduct initial integrated life testing

# 4. ESTIMATE OF TECHNICAL FEASIBILITY

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The BN emitters have proven to be robust when exposed to higher levels of oxygen, xenon, and atmosphere (air and water vapor). They have also shown rather low biasing voltage requirements to initiate emission (a few volts/micron). The important role of surface morphology (self-assembled arrays of very sharp BN nano-tips) is evident from these Phase I studies. It will be important in the next phase to produce optimized samples with homogenous surface characteristics so that emission will be drawn concurrently from most of the tips in these arrays. There is confidence that producing more homogenous samples will lead to a flight version of the emitter that will have immediate applications for low power Hall thrusters, electrodynamic tethers and related technologies.

#### 5. PEOPLE INVOLVED

The people involved with this Phase I STTR include:

- Dr. Jonathan Van Noord (EDA)
- Dr. Brian Gilchrist (EDA)
- Dr. Alec Gallimore (EDA)
- Dr. Roy Clarke (University of Michigan)
- Pedro A. Encarnación (University of Michigan)
- Hannah Goldberg (University of Michigan)

#### 6. PUBLICATIONS FROM RESEARCH

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