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16. Abstract (Limit: 200 words)

This interim report describes the work performed during the first month of SBIR Phase I research on the laser ablation methodology of cubic boron nitride deposition on electronic materials. Specifically, the report presents our initial efforts dealing with material procurement, experimental design and setup, preliminary experiments, results and some discussion. The work shows the potential of laser for the synthesis of cubic boron nitride.

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Figure A1
Report Documentation Page

SBIR Phase I Research Progress Report

During the period June 15 through July 14, 1991, initial tasks of the project including some experiments were carried out and are described below.

Materials Procurement

The project requires a hexagonal boron nitride (HBN) target and silicon substrate for laser ablation synthesis of cubic boron nitride (CBN). A high purity grade of HBN was obtained from the Advanced Ceramics section of Union Carbide. The HBN rod received has a purity of 99% (HBC grade), a diameter of 12 mm and a length of 150 mm. Substrate materials were available in-house and are n-type Si <111> and Si <100> wafers with thicknesses of 0.6 mm and 1.6 mm respectively.

Experimental Arrangement

Figure 1 shows the experimental setup for laser ablation. A six-way vacuum chamber which can be evacuated to less than 10^{-7} torr by means of diffusion and mechanical pumps is used in the work. This chamber has provisions for the laser beam window, heating the substrate up to $1000^{\circ}C$ and mounting of the target. The HBN target can be spun within the chamber using a magnetic device external to the chamber. The substrate-to-target distance was held at about 5 mm but can be varied up to 25 mm. The HBN rod can rotate at various speeds upto a maximum of 50 rpm. An inlet for N_2 gas flow into the chamber is also shown in Figure 1. A lens behind the window is located in order to focus the beam on the HBN rod.

An KrF-excimer laser beam, operating at 248 nm, was used for ablation. The 248 - nm excimer laser offers the following specifications:

pulse width 23 nsec
pulse energy 30 - 400 mJ
pulse repetition rate 1 - 100 Hz

Experiments, Results and Discussion

Two experiments were conducted to determine the feasibility of CBN growth on silicon substrates.

In experiment # 1, a Si <111> wafer was used as the substrate. The sample with typical dimensions of 25 mm x 25 mm x 0.6 mm in the mirror-polished condition was ultrasonically cleaned in methanol followed by etching in HF acid (49%) for 30 seconds. The sample was then rinsed in water and dried. The wafer was mounted on the sample holder parallel to the laser beam axis and about 5 mm below the target. The vacuum chamber was evacuated to 10^{-7} torr while the specimen being heated to $400^{\circ}C$. The HBN target rod was then spun at 50 rpm. The excimer beam was focused on the bottom of HBN rod (see Figure 1) using a 100 mm focal length quartz lens. The laser parameters included:

Energy density = 24 J/cm^2
Pulse rate = 5 Hz

A total of 18,000 pulses was used for ablation. Figures 2a and b are the photographs of the laser ablation experiment. After completion of the experiment, the sample was visually examined and then in scanning electron microscope (SEM) attached with an X-ray microprobe for elemental analysis.

The sample exhibited a brown-colored film distributed over 350 mm^2. In the center of film area, the deposit was not adherent and, in fact, was stripped off. SEM/EDAX analysis showed that the film contained 1-3 micron sized particles (Figure 3). The particles were identified to consist of boron and nitrogen in EDAX.

Although BN could be deposited by this experiment, additional analysis is required to identify its crystal structure and stoichiometry. The key problems in this experiment were particulate formation and stripping of the film at the center.

A second experiment was designed to minimize the particulate formation as well as reduce the adherent problem of the film. In this experiment we have used a Si <100> sample as the substrate and the substrate temperature was reduced to $100^{o}C$. Other parameters and conditions were essentially same as experiment # 1. Results of visual and SEM examination indicated a strongly adherent, continuous film of BN (Figure 4). However, the micron-sized particles were still present although the particulate density is slightly reduced.

In both experiments, the particulate formation was partly attributed to the intermittent rotation of the target (caused by the bearing friction) in the chamber during the experiment. This problem was now corrected by redesigning the bearings that support the HBN rod. Currently experiments are being planned to reduce the particulate formation by increasing the distance between the substrate-to-target and reducing the energy fluence. Other effective measures are being studied. Raman spectroscopy and X-ray diffraction of the films will soon be carried out to confirm the presence of CBN and reported later.

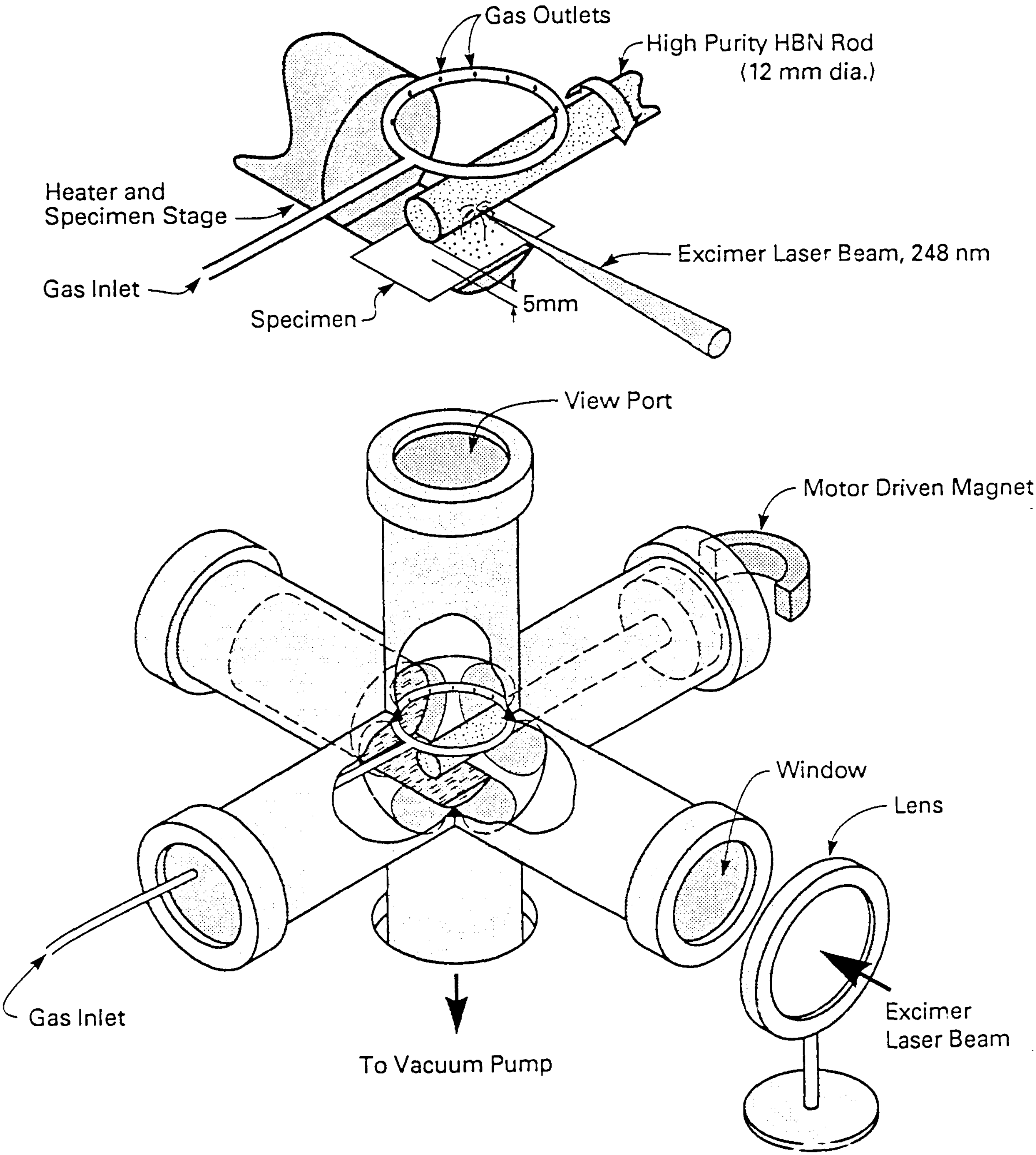

Figure 1. Experimental setup for laser ablation synthesis of cubic boron nitride

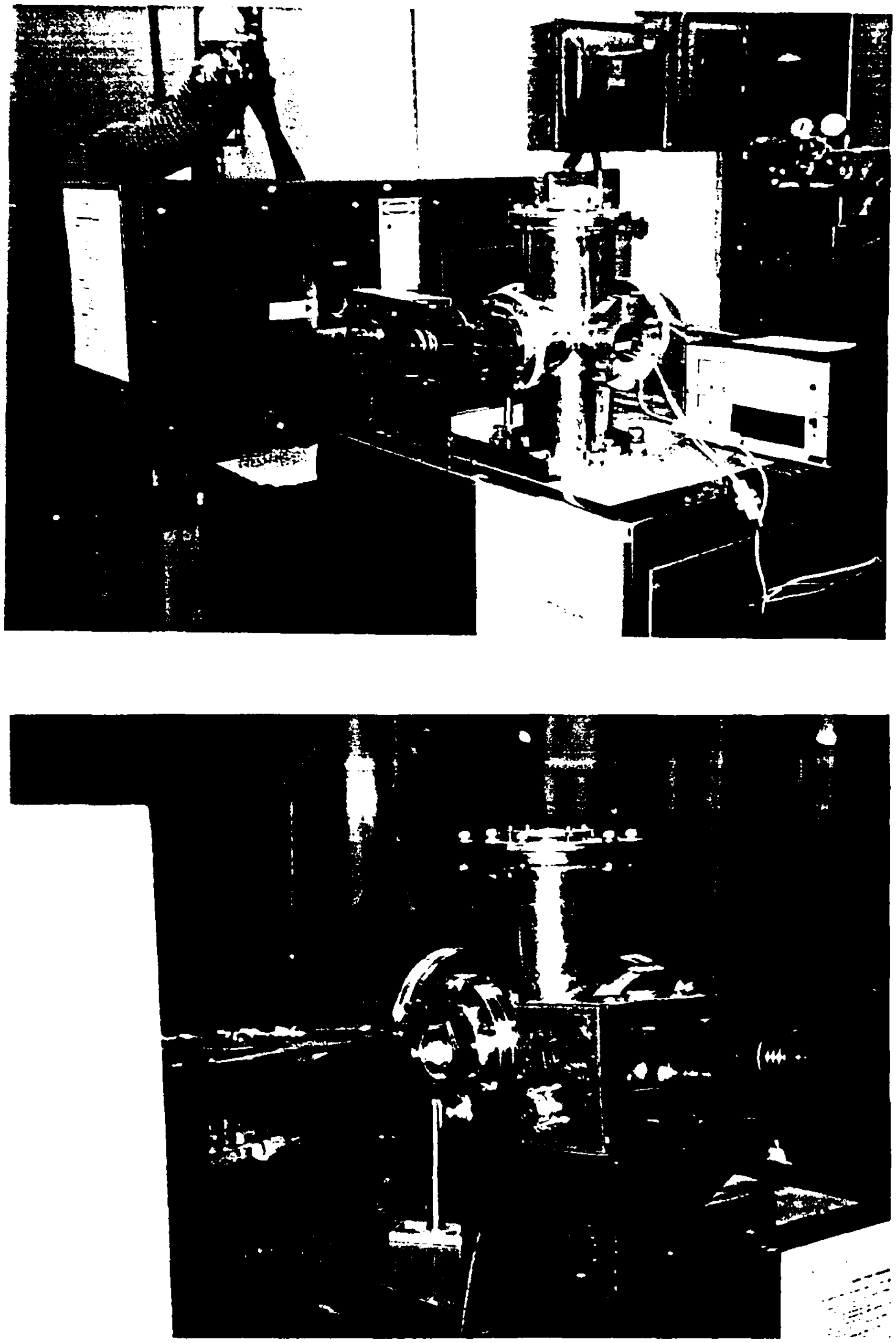

Figure 2. Photographs showing the excimer laser experiment for synthesis of thin films

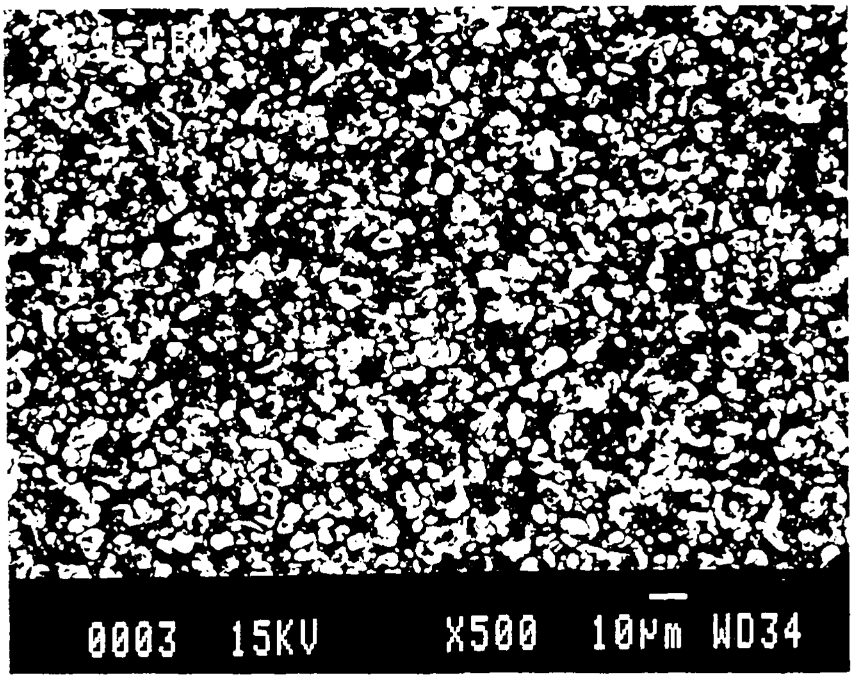

Figure 3. SEM micrograph of laser ablation deposited film on Si <111> substrate

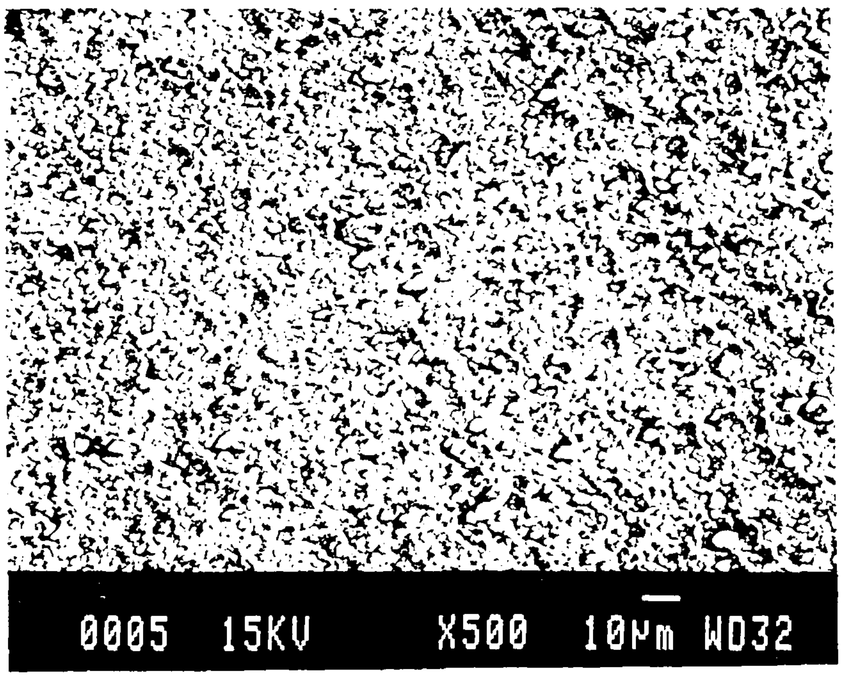

Figure 4. SEM micrograph of laser ablation deposited film on Si <100> substrate