



FINAL REPORT

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Production of Optical Quality Free Standing Diamond Wafer

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1. Overview

Valdosta Optics Laboratory, Incorporated (VOLI) is pursuing the capabilities of developing synthetic diamond and diamond-like materials, and integrating and implementing these materials in various applications, particularly as a high performance heat spreader for solid state laser (SSL) components, optical coatings, and active engineering materials for micro-electro-mechanical systems (MEMS) and sensors.

The current focus is on three key research and development directions, namely:

1) optical quality, highly oriented polycrystalline diamond (PCD) wafers as heat spreader for solid state laser crystals,

2) large area single crystal diamond synthesis, and

Further adjustments and additions to the research and development projects/programs will be made as the company's capabilities continue to grow.

In order to conduct the planned projects, experimental equipments and instruments, including microwave CVD reactor, DC/RF metal sputtering system, laser cutting system, sample pre- and post-deposition treatment facilities, and material characterization instruments, have been specified, designed, purchased, and installed.

2. Project Plan

2.1. Optical Quality Polycrystalline Diamond Wafers as Heat Spreaders for Solid State Laser Components

2.1.1. Objectives and Justifications

The goal of this project is to develop freestanding, optical quality, highly oriented polycrystalline diamond discs with a thickness up to 1 mm and a diameter over 2 inches. It will be bonded as the heat management layer with a laser crystal using Adhesive-Free Bond (AFB®) technology.

The importance of effective thermal management in the design of solid state laser system cannot be overemphasized. The demand for higher optical power from high power pump lasers requires efficient thermal management since their performance decreases drastically with the increasing temperature of the lasing materials. For example, with a diode laser as a pumping source, the lasing wavelength shifts significantly with changes in junction temperature, about 0.3 nm/K for a 980 nm pump laser. Thermal management of the SSL system is crucial to the device's performance and extended long term reliability.

Diamond has the highest thermal conductivity of any known material, almost three times higher than the widely used heat management material of copper. Therefore it is a superb candidate for thermal management purposes. However, the integration of the SSL crystal and the CVD diamond layer is a major obstacle. VOLI will approach this obstacle by initially depositing diamond onto a foreign substrate (e.g., silicon). After stripping the silicon substrate off, the SSL crystal and the free-standing diamond layer will be bonded together.

The synthesis method for PCD will be a microwave plasma enhanced chemical vapor deposition (CVD) technology. Plasma CVD technology has proven to be a stable, fast, and versatile means of growing synthetic diamond and similar materials. A microwave plasma reactor has been ordered and is being assembled by Lambda Technologies.

Since the thermal conductivity of the CVD diamond is directly related to its material quality, optical grade PCD is desirable in order to achieve high thermal conductivity up to 1800 W/m-K.

The crystal orientation of the as-grown PCD is critical with respect to the AFB® process. In order to achieve van der Waals bonding between two crystals, the bonding surfaces need to be extremely flat and smooth. Polishing of PCD discs is thus necessary to get the desired flatness and surface roughness (a few Å RMS). Polishing randomly orientated PCD surfaces, which is the crystal orientation in most cases for plasma CVD technology, to this level of smoothness is difficult and time consuming, if not impossible, due to the existence of the pyramidal (111) spikes (Fig.1a). On the other hand, by controlling the crystal growth of the as-grown PCD to be (100) orientation (Fig. 1b), it should be possible to polish the PCD surface to sub-nanometer roughness.

2.1.2. Technical Background

Recent technology developments in two areas, bias enhanced nucleation (BEN) and high growth rate CVD diamond synthesis, are key for realizing the PCD heat spreader.

Stoner et al. ¹ reported successful growth of azimuthally oriented diamond crystals on β -SiC using Yugo's BEN method ², which then led to the growth of (100)-oriented diamond films on Si (100) that later became known as highly oriented diamond (HOD) films. Most recently, Kawarada et al. ³ were successful in growing perfectly coalesced, (100)-oriented, 300- μ m thick HOD films, with no grain boundary at the film surface.

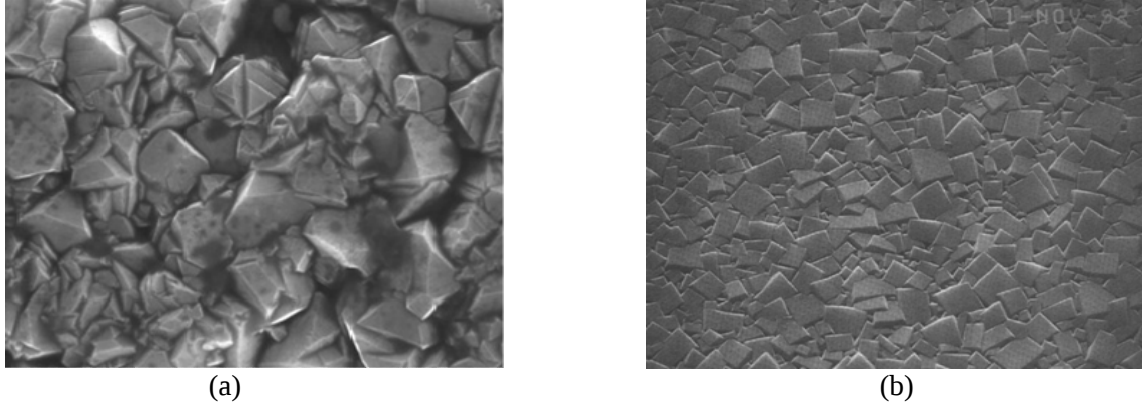


Figure 1. Scanning electron microscope images of polycrystalline diamond as-grown surface with (a) random orientation, and (b) (100) orientation.

For high growth rate CVD processes, Yan et al.⁴ reported the successful production of high-quality single-crystal diamond with microwave plasma CVD technology. The crystals can be produced at growth rates from 50 to 150 $\mu\text{m/h}$, which is up to 2 orders of magnitude higher than standard processes for making polycrystalline MPCVD diamond.

The CVD reactor DiamoTek-700 ordered from Lambda has the ability to realize the above growth condition. However, the diamond growth rate of the machine claimed by the supplier was around 5 $\mu\text{m/h}$ with standard methane-hydrogen chemistry, which is too low for a commercially value final diamond product (Fig. 2). Thus improvement work with respect to growth rate is needed in order to produce cost-effective CVD diamond discs.

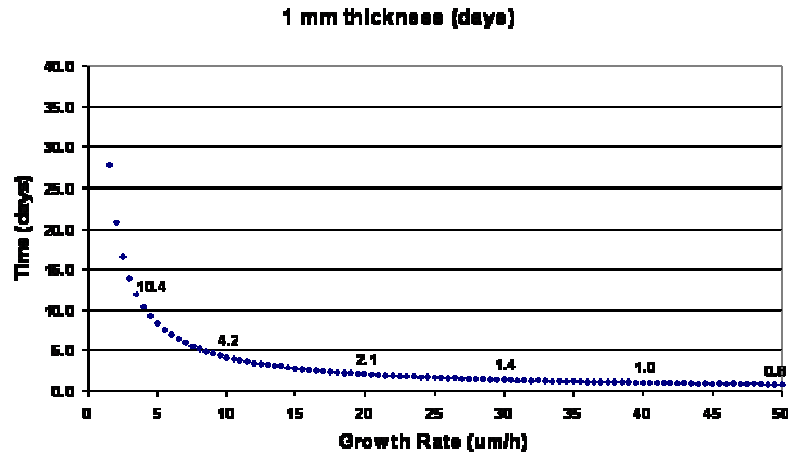


Figure 2. A schematic drawing of the relationship between growth rate and total growth time for a 1mm thick diamond disc.

2.2. Large Area Single Crystal Diamond Growth

2.2.1. Objectives and Justifications

A freestanding, single crystal diamond plate/disc with a thickness over 1 mm and a diameter over 1 inch is an ideal candidate as a heat spreader for large laser crystals such as disk-shaped YAG lasers and optical windowss in high power laser systems. Moreover, a large size single crystal diamond wafer will have a fundamental impact on other diamond-related applications, such as diamond electronics, diamond MEMS, and diamond-biological applications.

2.2.2. Technical Background

The major drawback for the CVD technology is that diamond film deposited on a non-diamond substrate is polycrystalline, consisting of sp^3 -bonded diamond grains and sp^2 -bonded grain boundaries. Due to the two-phase nature of PCD, the material properties of PCD are highly affected by its grain boundary content, even though the mass fraction of the grain boundary is small. Although high-quality polycrystalline diamond films are suitable for many applications, single crystal diamond is required in many other cases, especially for optical components and active electronic devices.

The unparalleled performance of single crystal diamond compared to polycrystalline diamond has led scientists to pursue large area single crystal diamond but with limited success. To some extent, the small area of available single crystal diamond has seriously restricted its application in other fields in spite of its high crystal quality. There are two major methods to synthesize single crystal diamond, namely 1) high temperature high pressure (HTHP), and 2) chemical vapor deposition (CVD). They each have their own limitations for large-area, large-size production. For HTHP method, it is technically impractical to form a wafer- or disc-shaped single crystal diamond. On the other hand, for the CVD approach, the size/area of the deposited single crystal diamond is confined by the size/area of the single crystal diamond seed, and the availability of large-area single crystal seeds is often limited and costly. For instance, the largest single crystal diamond plate in the market is 6.5mm×6.5mm×2mm with a list price of \$1,600 (Sumitomo Electronic Inc.).

Some efforts have realized large area single crystal diamonds. A formation of so called “mosaic” structures has been attempted as early as 1991 by Geis et al.⁵ Other groups including Findeling-Dufour’s⁶, Janssen’s⁷, and Meguro’s⁸ also have done similar work during 1993-1997. Kobashi et al.⁹ have tried similar research in 2003, in which he deposited a single crystal diamond layer on the top of a 4x4 matrix of single crystal diamond plates as seed (Fig. 4). Although there was a successful proof of concept, this diamond layer grown on top of the seed matrix often suffers from large internal stress, lattice mismatch, embedded defect points/lines, and even catastrophic crack line formations. The major hurdle is the microscopically huge gap ($\sim 1\mu\text{m}$) between two adjacent diamond seed plates which caused inhomogeneous lattice structure along the interface line.

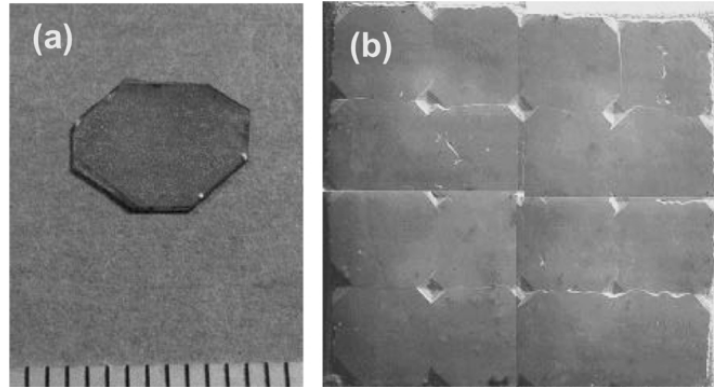


Figure 4. (a) 8 mm single crystal of CVD diamond; and (b) 12x12 mm² size mosaic diamond of 1 mm thickness.

A summary of their research results:

1) The assembled mosaic diamond plates were soldered onto a molybdenum substrate holder using a thin layer (10 μm) of high-temperature solder (melting point $> 1250\text{ }^{\circ}\text{C}$). A small, tight-fitting, recess in the molybdenum prevented the plates floating apart during soldering. This procedure ensured the alignment of the seed crystals within $0.2''$. The gap between the crystals, at room temperature, was typically less than 1 μm , while the height difference varied between - 1 and + 3 μm .

2) A band of enhanced growth is present parallel to the interface. The main difference between the samples is the position of the band relative to the original position of the. These shifts correspond to the imposed mis-orientation between the crystal plates.

3) Crack lines often appear along (111) direction, which limit the diamond layer thickness. Therefore, internal stress is a critical problem in getting large area single crystal diamond.

The diamond growth at the contact boundaries was strongly influenced by the factors listed below:

- 1) The crystal orientations of the basal diamond plates.
- 2) The difference in heights and crystal orientations between adjacent diamond plates.
- 3) The difference in off-angles between the adjacent basal diamond plates.
- 4) CVD growing conditions.

As a conclusion of above discussion, to realize a perfect single crystal diamond layer across the different diamond plates, the offset of the crystal orientations among the seed matrix must be less than 2° , and the plates must have the same height.

Equipment and Facility Design, Procurement, and Installation

2.3. Material Synthesis and Deposition Facilities

2.3.1. Microwave Plasma Chemical Vapor Deposition Reactor and Supporting Facilities

A decision has been made to order the Lambda Technology reactor as opposed to the Seki reactor because both reactors appear capable of the same quality of CVD diamond deposition but Lambda is less expensive, closer in location (North Carolina vs. Japan), no need to break the vacuum when switching from bias enhanced nucleation to high growth rate, more post processing support and potential cooperation with MSU. The microwave CVD reactor is being assembled at Lambda Technology's NC facility. The system is expected to be delivered to VOLI's facility in mid-April. Figure 17 shows the current assembly status of the system.



(a)



(b)

Figure 17. Pictures of the Lambda CVD reactor being assembled.

2.3.2. DC/RF Sputtering System

The MRC sputtering system has been delivered to VOLI. This system is able to perform DC and RF sputtering of metal and other materials with three 6" targets. The re-assembly of this system is in progress.

2.4. Laser Cutting System

2.4.1. Overview

Diamond, as an extreme material, is difficult to process because of their unparalleled hardness and wear resistance. Laser processing, such as cutting and marking, has become an effective method in machining diamonds. In laser machining, a laser beam heats up and evaporates diamond carbon to realize material removal.

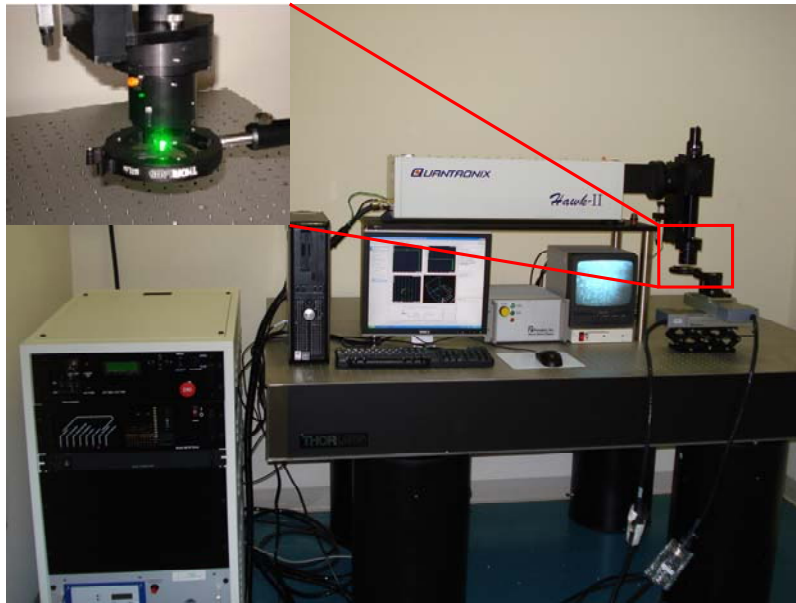


Figure 18. Picture of the laser cutting system. (Insert: picture of the laser spot.)

The objective of this project is to realize 1) Complex 2D diamond cutting with smooth cutting cross-section, narrow kerf-width, and no heat damage. 2) Diamond laser micro-marking of complex 2D shapes.

2.4.2. Diamond Laser Cutting

First, diamond is the best known thermal conductor. Theoretically it is difficult to heat diamond to very high temperature with laser in CW mode, even focused properly. Therefore, the laser must be set in Q-Switch mode with pulse rate in kHz range. In this way, the laser will not only be spatially focused to tens of microns area, but also be compressed in time domain to hundreds of ns. In short, ultra-fast heating is the key.

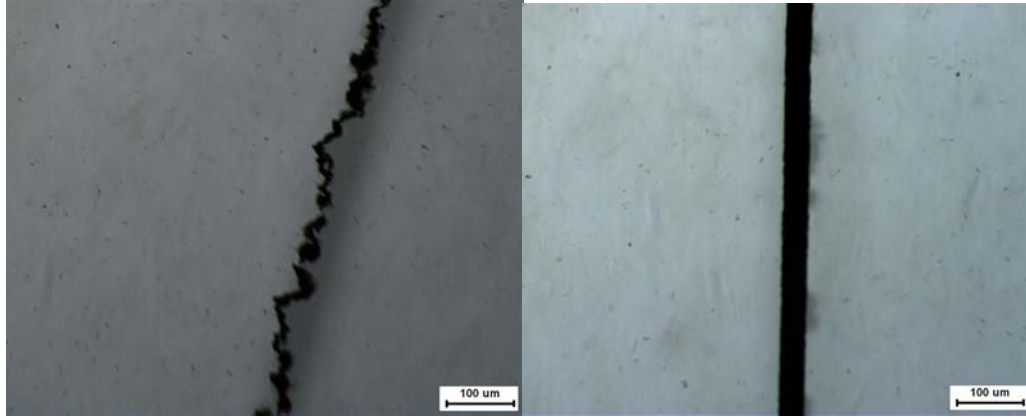
Secondly, optical absorption of diamond is very weak in visual light band. The effective laser cutting will be after the realization of initial graphite layer, which locally improve the optical absorption. Therefore, the diamond cutting procedure is a two-step procedure: diamond is first transformed to graphite, and then the graphite is burned in the air.

Many factors we need to carefully evaluate in order to realize the high quality laser cutting of diamond, some of them are listed in the following.

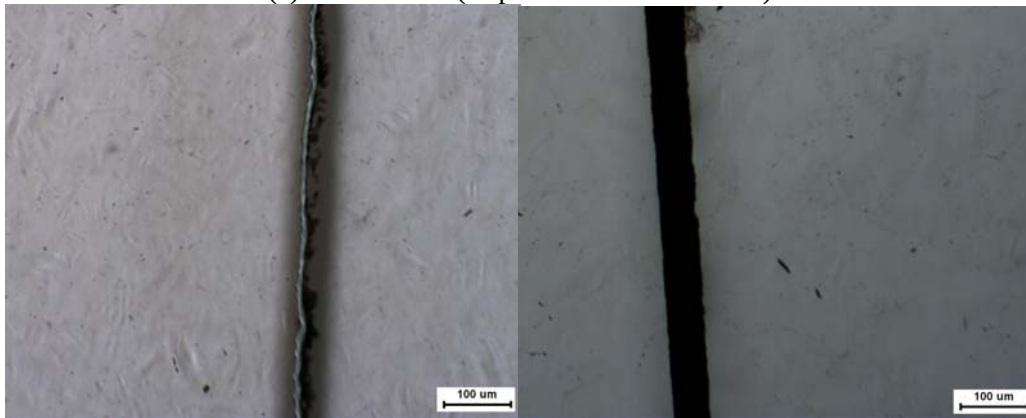
3.2.2.1 Influence of Laser Power and Pulse Rate

Laser power and pulse rate are combined together to decide the final cutting performance.

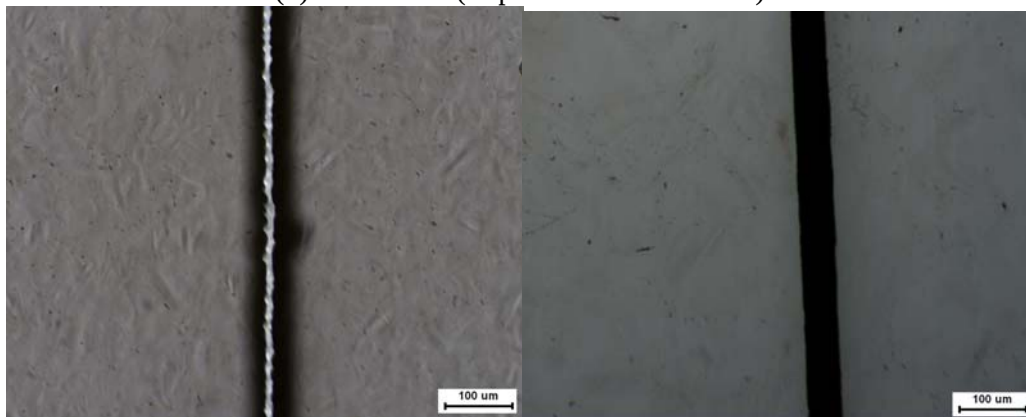
It does not necessarily mean that a laser with a higher power density is better in diamond cutting. Too high power density can result in more material damage, such as heated affected zone (HAZ) and heat checking. We need find an optimal laser power and pulse rate to make the laser pulse energy just greater than the critical evaporation energy (CEE) of diamond. We here compared the cutting results of different pulse rate under the same power of 9W.



(a) 20 kHz (Top Kerf-Width = 35 µm)



(b) 3 kHz (Top Kerf-Width = 55µm)



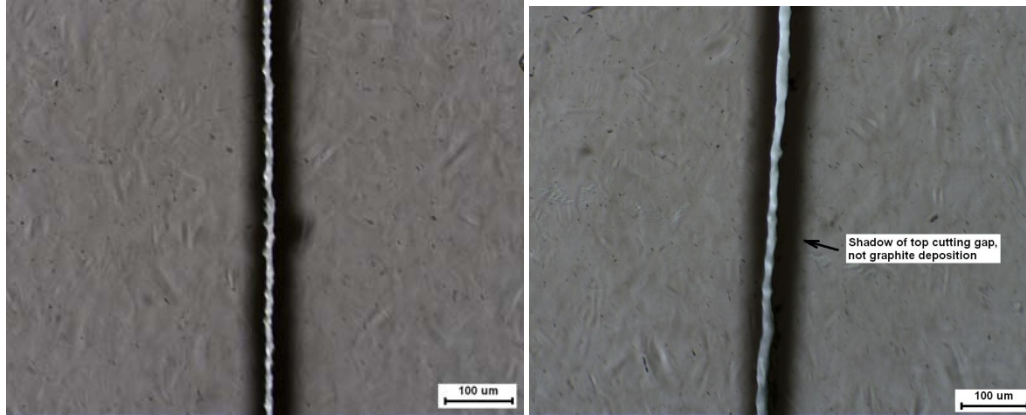
(c) 1 kHz (Top Kerf-Width = 65 µm)

Figure 19. SEM images of laser cutting patterns at various laser powers and pulse frequencies.

We can see that higher pulse rate was related to weaker cutting ability, with 1 kHz pulse rate giving best comprehensive cutting performance.

3.2.2.2. Influence of Sample Motion Speed

Too slow sample motion speed may cause local heat damage on cutting sample. However, too fast sample motion speed may cause cutting trajectory shifting, which widens the cutting kerf-width. We here further compared the influence of two motion speeds to the final cutting with the same 9W power and 1kHz pulse rate.



0.5 mm/s
5 mm/s (edge more smooth)
Figure 20. SEM images of laser cutting patterns at various moving speeds.

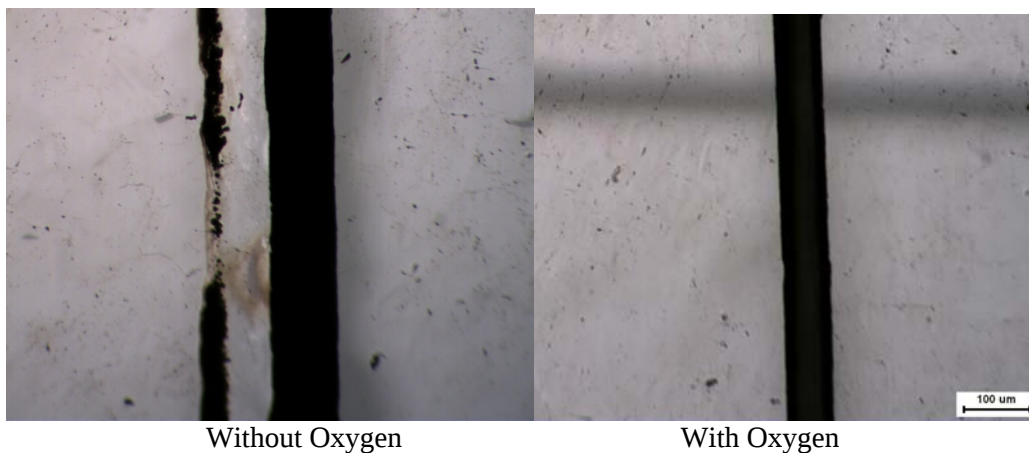
3.2.2.3. Influence of Oxygen Feeding

1) Before adding the oxygen feeding nozzle, it was difficult to cut through the diamond plate. The reason is that, without the help of enough oxygen, the transformed graphite inside the deep cutting gap worked just as an excellent isolation layer to prevent further laser cutting. Therefore, oxygen feeding is another key for efficient diamond laser cutting.

2) Because of the sufficient burning of graphite, it will prevent of the deposition of graphite on the cutting edge.

3) The oxygen flow to the cutting point also works as an effective cooling method to reduce the thermal damage on the diamond cutting edge.

4) Such cooling effects will narrow and sharpen the temperature profile at the cutting point, and lead to a narrower cutting kerf-width.



Without Oxygen
With Oxygen
Figure 21. SEM images of laser cutting patterns with or without feeding oxygen.

3.2.2.4. SEM Research of Cutting Cross-Section

Our optimal cutting parameters are 9 W, 1 kHz, 1 mm/s, and 20 PSI oxygen blowing. Under such working conditions, the diamond sample plate can be successfully cut within 10 passes. The SEM research of the cutting cross-section confirmed a successful laser cutting of diamond plate with smooth cutting edge, no heat damage.

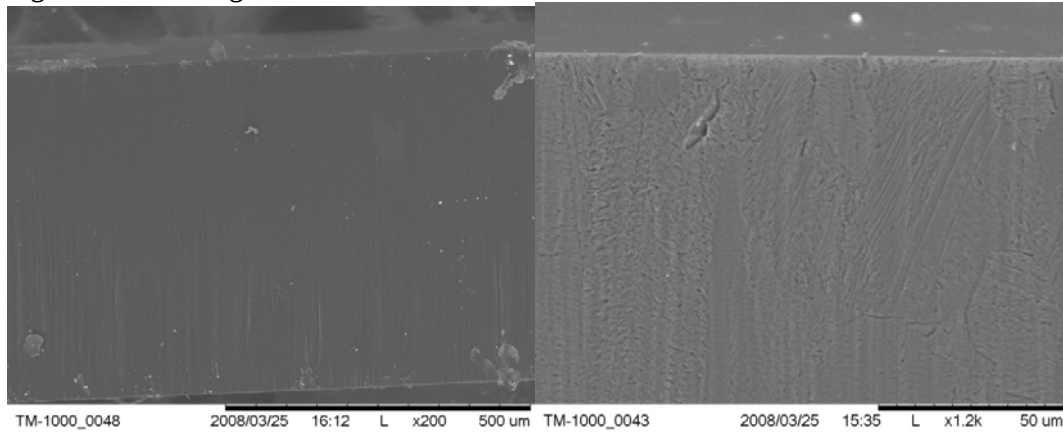


Figure 22. SEM images of the cross section view of the laser cutting patterns.

3.2.3. Diamond Laser Marking

LabView program to realize complex laser cutting and marking has been developed, with interface of simplified version shown below.

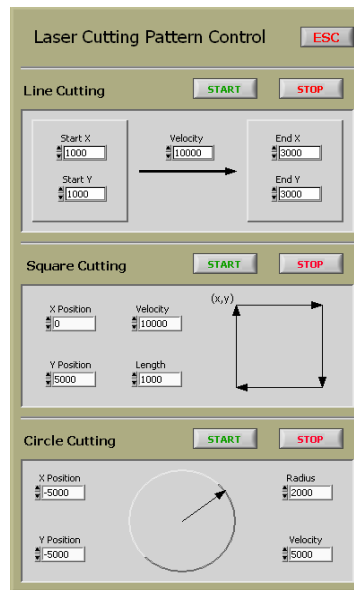


Figure 23. Programmed LabView interface for motion control.

Figure 24. Optical images of the laser marking results.

3.2.4. Conclusion

Through intensive research and development, VOLI now has the ability to cut 2D shapes on a diamond plate of thickness 2 mm or less. The cutting kerf-width is 60 um on the top surface and 25 um on the bottom surface. The cutting edge is clean, smooth, and of no heat damage. VOLI has also successfully owned the ability of laser micro-marking of complex 2D shapes on a diamond plate.

2.5. Material Characterization and Quality Control Instruments

2.5.1. Raman Spectroscope

In selecting a Raman microscope, there are two main concerns in choosing the right wavelength laser. First is the cross-section of the species, in our case diamond bonding as sp^3 .

Second is the photoluminescence background. A UV laser is more sensitive to SP^3 bonding than a longer wavelength, which is critical for very thin diamond film. Longer wavelength (Red) laser will have less photoluminescence background, which is important to get good S/N ratio. As a result, the common choice of Raman laser wavelength for diamond research is a green laser, a compromise of UV and Red. It is always good to have both UV and green lasers as Raman laser source to provide more information of sample film.

Raman spectroscope Model LabRamHR/UV ordered from Horiba has arrived and been installed. Fig. 24 shows the actual system and a spectrum measured from polycrystalline diamond sample.

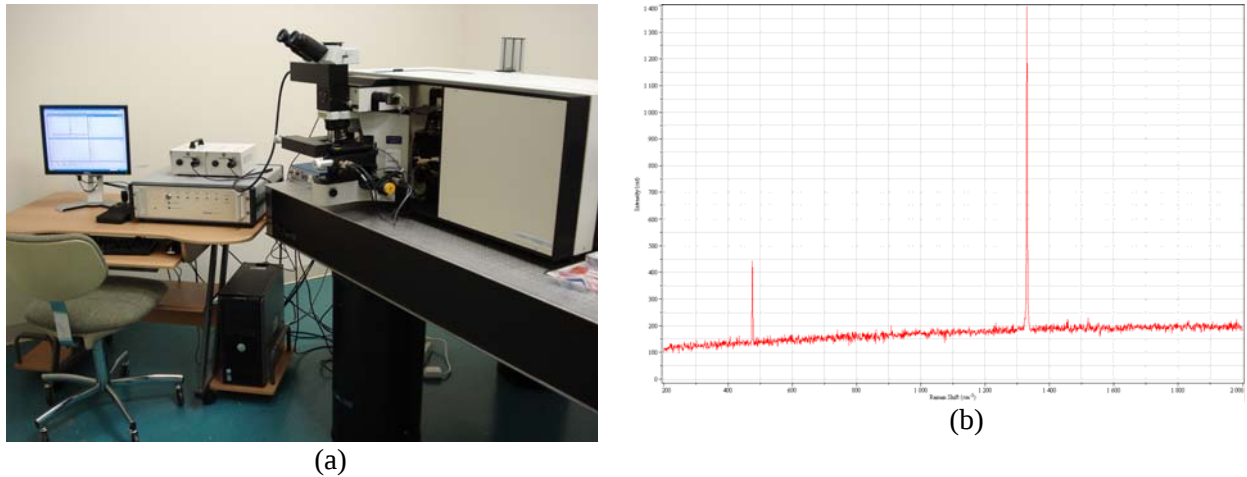


Figure 24. (a) A picture of the Raman Spectroscope and (b) a Raman spectrum of polycrystalline diamond sample.

2.5.2. Scanning Electron Microscope (SEM)

A SEM machine from Hitachi has been installed. This SEM is capable of providing high-quality SEM images with maximum magnification of 10,000X. It is suitable for low-conductivity samples, such as high-quality poly- and single-crystal diamond, with its charge release function.

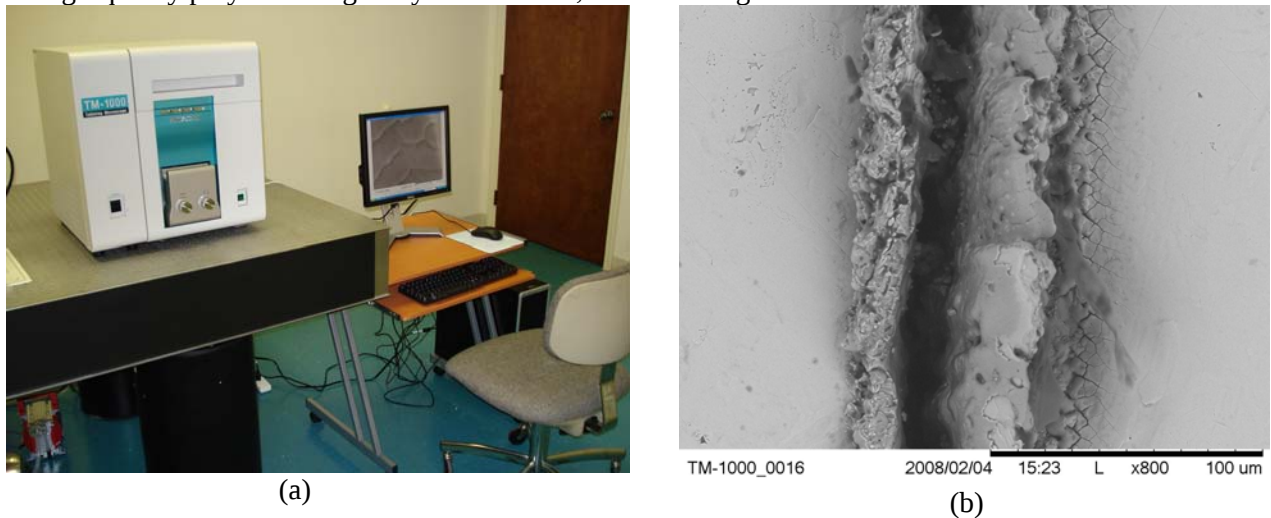


Figure 25. (a) A picture of the Raman Spectroscope and (b) an SEM image of the laser cutting groove on copper alloy.

2.6. Wet Chemical Laboratory Facility

The wet chemical laboratory has been set up with facilities and instruments including two ventilation hoods, two chemical storage cabinets, a DI water generator, a polishing machine, two ultrasonic cleaners, and an emergency shower and eye wash station. Fig. 26 shows the picture of the wet chemical lab.



(a)



(b)

Figure 26. Pictures of wet chemical laboratory.

3. Collaboration with Other Research Institutions

3.1. Intern Program for Valdosta State University (VSU)

The intern program between VSU and VOLI was initiated in January 2008, and has been progressing smoothly ever since. Four VSU undergraduate students have been accepted by VOLI and research projects have been assigned to individual students. Training of facilities and instruments, including general safety training, scanning electron microscope (SEM), Raman spectroscope, and laser cutting system, has been carried out. Workshops on SEM technology and applications, Raman technology and applications, motion control, and laser cutting technology, were carried out. Presentations were given on above topics by Dr. Liu, Dr. Qiu, and intern students. The Phase I of the VSU intern program has been completed in March 2008. Both VSU and VOLI are satisfied with the progress of the intern program. As a result, we decided to renew the contract between VOLI and VSU, and add two more intern opportunities at VOLI during the summer semester.

3.2. Research and Development Collaborations

VOLI collaborated with the Chemistry Department, Valdosta State University on an instrument proposal submitted to National Science Foundation for a high-performance X-ray diffraction machine. VOLI also worked together with the Electrical and Computer Engineering Department, Auburn University on a STTR proposal submitted to DARPA, Department of Defense.

4. Accounting of Federal Funds Expended and References

Accounting of Federal Funds Expended

Year 1 Contract	\$1,614,000
VSU Subcontract	\$60,000
VOLI Subcontract	\$1,182,239

Date	SF270#	Total Invoiced	Amount Invoiced to Date	Total Remaining for Contract
07/11/07	1	\$129,896	\$129,896	\$1,484,104
08/07/07	2	\$38,716	\$168,612	\$1,445,388
09/28/07	3	\$266,301	\$434,913	\$1,179,087
10/25/07	4	\$356,659	\$791,572	\$822,428
11/29/07	5	\$97,181	\$888,753	\$725,247
12/28/07	6	\$58,831	\$947,584	\$666,416
01/29/08	7	\$174,625	\$1,122,209	\$491,791
02/29/08	8	\$332,657	\$1,454,866	\$159,134
03/19/08	9	\$159,134	\$1,614,000	\$0

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