

# Improvement of mechanical properties of electroplated diamond tools by microwave plasma CVD diamond process

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Received 5 January 1998; accepted 8 June 1998

## Abstract

The growth and surface modification of irregular diamond grains of electroplated diamond tools have been developed successfully. Results show that the adherence, cutting ability, and wear resistance of diamond grains are improved by this process. In this study, the crystallization and quality of diamond grains were determined by SEM (SE and BSE), Raman spectroscopy, and XRD determination. Tests using a block-on-ring tribotester were also carried out to examine the adherence, cutting ability, and wearing resistance of the electroplated diamond tools. The adherence of diamond grains was also observed on a SEM microphotograph of the cross-section view of specimens. SEM line scanning was performed to determine whether the alloy elements are effectual in promoting the diffusion bonding strength among diamonds, the interlayer and substrate. © 1998 Elsevier Science S.A. All rights reserved.

*Keywords:* Electroplated diamond tools; Microwave plasma CVD diamond process

## 1. Introduction

Diamonds are extensively applied in machining, such as grinding, cutting, lapping and polishing, and their use may even be extended to drilling bits, core drill and single point tools [1–4]. Diamond tools are indispensable tools for sawing architectural materials. When the significance of the cutting speed is more important than the tool's life, electroplated diamond tools are often used. Because these diamond tools can produce sophisticated curves at a faster cutting speed and low costs, their use is favoured by several industries. Because the temperature in the electrolytic process is low and nickel is not wetting well to diamond, diamond grains only adhere to the surface of the tool by mechanical interlocking. Such an interlocking action has many shortcomings, and so the adherence of diamond grains is not secure and grains may fall off easily during the cutting operation, thereby possibly leading to a reduction in a tool's service life. However, diamond grains must be covered by metal so that the diamond grains can adhere to the

substrate. The extra coverage height on this part of the diamond grains may mean that the grains are unable to cut into the workpiece as a result of the reduced cutting depth reduce, and the chips discharge with difficulty. Moreover, since the nickel layer is too thick, the discharge of chips are often blocked, so the cutting speed is greatly reduced, and the cutting even completely disrupted in some extreme cases. It is advantageous to electroplated diamond tools when diamond grains with strong blocky shape, edges sharpness and marked facets. The objectives of this study are to improve the aforementioned shortcomings in, and meet the needs of, electroplated diamond tools.

The LPSSS (low-pressure solid state source) process is suited for synthesizing high-quality diamonds under low-pressure conditions [5–7]. The main idea of the LPSSS process is to produce liquid metal–carbon–hydrogen alloys in the presence of plasma to enhance diamond nucleation and growth [5–11]. In this study, using a similar idea, a process has been successfully developed to improve the mechanical properties by diamond growth and surface modification on the electroplated diamond tools.

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## 2. Experimental

The specimen for electroplating nickel was JIS-SKD11 tool steel with nominal compositions of 1.4~1.6% C, 11.0~13.0% Cr, and 0.8~1.2% Mo. The specimen of the substrate was designed to be triangular with sides of 15.0 mm and a thickness of 4.1 mm. After mechanical polishing, the specimen was electroplated with a layer of nickel at a current density of 1.0~1.4 A cm<sup>-2</sup> under a temperature of 30~40 °C by using an electrolyte, which was dispersed with micro-diamonds. Diamond grains are commercial-grade HPHT synthetic diamonds with irregular shapes and an average size of 16 µm in diameter. The electrolyte consists of nickel sulphamate (600 g l<sup>-1</sup>), nickel chloride (4.4 g l<sup>-1</sup>), boric acid (40 g l<sup>-1</sup>) and a few drops of H<sub>2</sub>O<sub>2</sub>.

Diamond grains of the electroplated diamond tools were grown by a 5-kW MPCVD system with hydrogen and methane as the source gases. The typical growth conditions were: CH<sub>4</sub>/H<sub>2</sub> = 18/300 (sccm), 2.5-kW microwave power, 70-Torr total pressure, 880 °C substrate temperature and a 3-h deposition time.

The morphology and crystal quality of these specimens were characterized by SEM (SE and BSE image), Raman spectroscopy (HeNe laser, λ=633 nm) and XRD. To account for the better adherence, cutting ability and wearing resistance of diamond grains in these tools by this MPCVD diamond process improvement, comparisons can be made in two ways: one using a block-on-ring tribotester test, with the materials wearing against electroplated diamond tools is Al alloy AA6061T4, with a surface roughness, R<sub>a</sub>=1.2 µm, and hardness, HRB=80 and the surface of the fracture being observed by SEM; and the other by SEM micro-photograph comparison of the adherence of diamond grains from a cross-sectional view of the specimens obtained before and after this process. Possible diffusion bonding to enhance the adherence at the interface was examined by SEM line scanning.

## 3. Results and discussion

### 3.1. Growth and surface modification of diamond grains by the MPCVD diamond process

From the C–Ni binary phase diagram under ambient temperature conditions, it can be seen that the carbon exhibits a very low solubility in metals and consequently will precipitate to graphite. In the HPHT process, high pressure is utilized to promote carbon's solubility in metals. Diamond is produced from the carbon–metal catalyzing process; but Roy et al. [5–11] found that the solubility of carbon could be enhanced with carbon and metals as mixtures exposed in a hydrogen atmosphere. In hydrogen plasma, it will vastly promote the solubility

of carbon in metals. A new process for the low-pressure synthesis of diamonds has been successfully developed [5–7]. The mixture used in Roy et al.'s studies could be obtained by mixing carbon powders with any group of metals comprising: Au, Ag, Fe, Cu, Ni, La, Mn, and Sn. When the mixture is exposed at 600–1100 °C in a hydrogen plasma to create a carbon–metal–hydrogen liquid, the diamond will crystallize from the alloy liquid under suitable conditions. Herewith, we speculate that the process of hydrogen and methane plasma can shed some light on to electroplating diamond tools, and can lead to diamond nucleation and growth on the surface of micro-diamonds and the Ni carbide layer. Under such conditions, the micro-diamonds eventually form seeds that develop into high-quality diamonds.

Fig. 1(a) shows the morphology of diamond grains by electroplating with an averaging size of 16 µm, with angular corners and an irregularly weak surface. It also shows concave and clear gaps between diamond grains and Ni metal. The above phenomena occur because

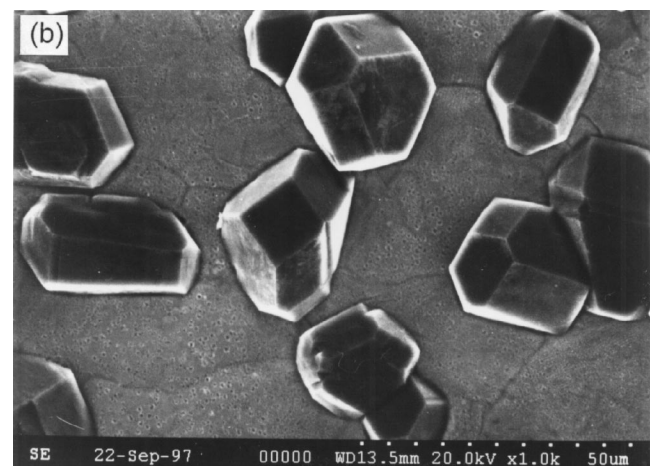
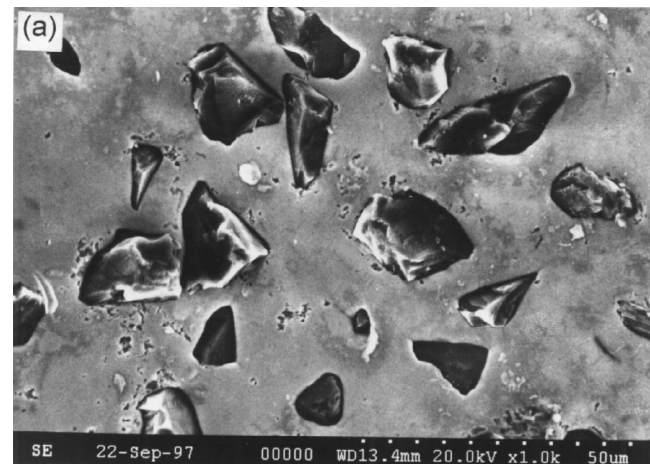


Fig. 1. SEM morphology of electroplated diamond tools: (a) original and (b) diamond grains through the growth and surface modification by this MPCVD diamond process.

nickel is not wetted as well as diamond, and diamond is also the best insulator. It is advantageous for machine-sawing for diamond grains are good enough to be with high-quality, sharp edges, obvious and strong blocky shape, the results of our efforts substantiated that objective in the long run that can be showed in Fig. 1(b). Fig. 1(b) is a SEM micrograph of the growth and surface modification of diamond grains produced by this microwave plasma CVD diamond process. It shows an average grain size of approx.  $25\ \mu\text{m}$  from the electroplated diamond tools under the MPCVD process for 3-h deposition. The results also show a high degree of crystalline growth of these diamond grains with a high quality, high nucleation density, good crystalline morphology, marked crystal facets, apex, hills and edge sharpness. The SE (SEM) and BSE (SEM) images of these strong block-shaped diamond grains are shown in Fig. 2(a and b). Electroplated diamond tools for this MPCVD diamond process are signified by four narrow XRD diamond peaks. Fig. 3 presents the typical Raman spectra with a peak at  $1333.8\ \text{cm}^{-1}$  and f.w.h.m. of  $3.6\ \text{cm}^{-1}$ , which confirms a good diamond crystalline quality.

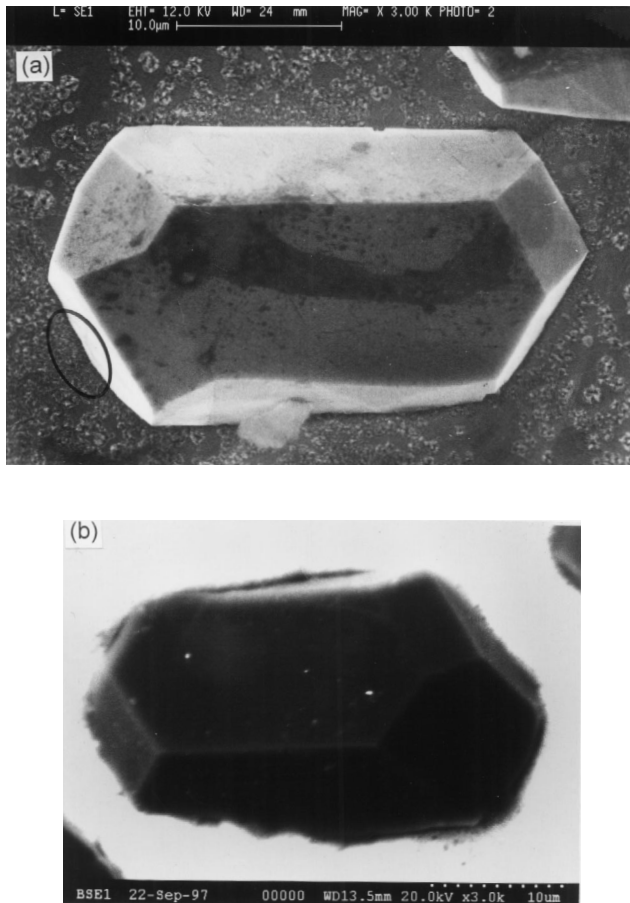


Fig. 2. Higher magnification of the strong blocky shaped diamond grain showing (a) SE (SEM) image and (b) BSE (SEM) image.

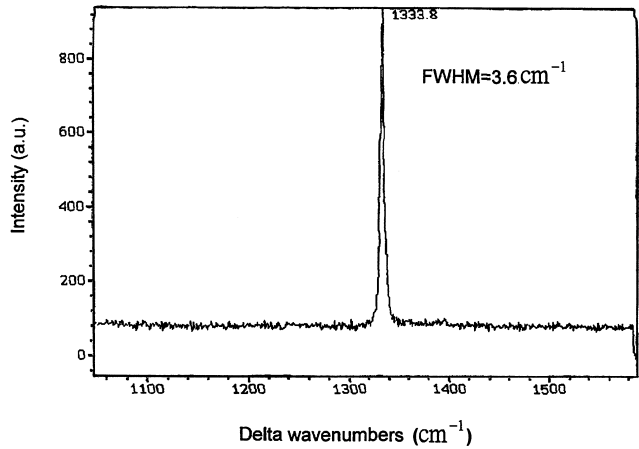


Fig. 3. The typical Raman spectra with a peak at  $1333.8\ \text{cm}^{-1}$  and f.w.h.m. of  $3.6\ \text{cm}^{-1}$ .

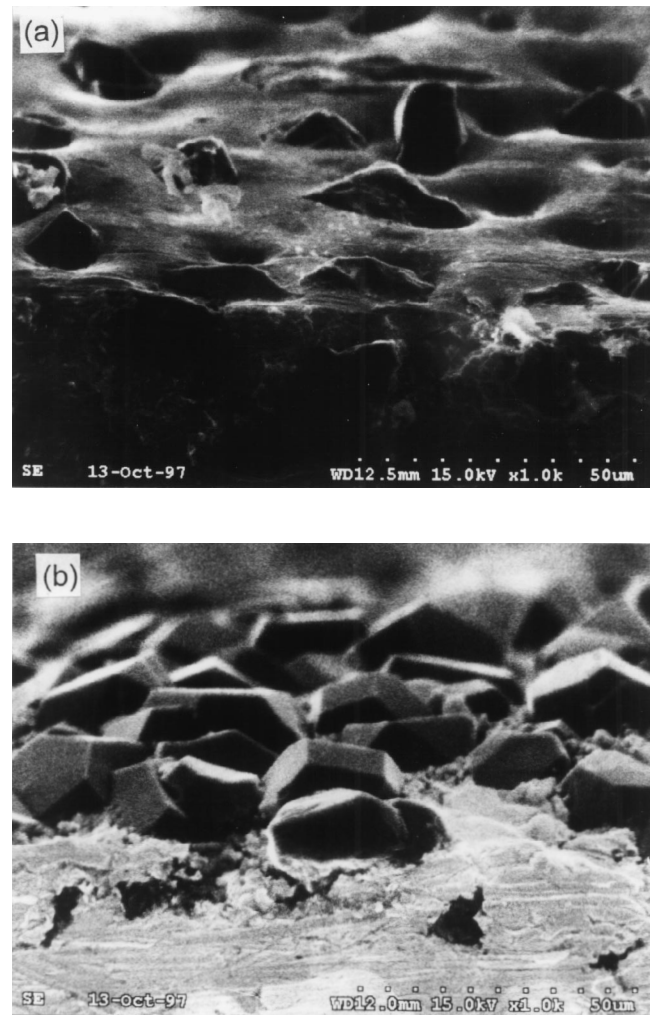


Fig. 4. Cross-section of the electroplated diamond tools: (a) original and (b) diamond grains through the growth and surface modification by this MPCVD diamond process.

### 3.2. Improvements of the adherence, cutting ability and wear resistance of diamond grains

Since the wetting ability between diamond and nickel is not good, nickel with diamond forms a concave contact in the electroplating process, and diamond grains only engage mechanically with the nickel layer. Here, the electroplated diamond grains are used as seeds to grow upwards as a continuation of this MPCVD diamond process, and these seeds impart similar anchoring effects. The originally concave contact in the electroplating process forms a flat and tight contact between the nickel layer and the diamond grains due to the formation of carbon–metal–hydrogen liquid; this result is shown in Figs. 1(b) and 2(a). However, the line between two intersecting facets [shown as a circled area in Fig. 2(a)] indicates a curve instead of a straight line. It is speculated that the carbon–metal–hydrogen liquid is splashed over the diamond grain, and this phenomenon is evidenced from the BSE (SEM) image in Fig. 2(b). Diamond crystals protrude from the surface, and therefore, the sawing height is increased. These improvements are advantageous for the cutting operation when increasing the chips-discharging speeds. The above-mentioned differences are also shown in Fig. 4(a and b). Fig. 5 shows the corresponding Ni, Cr, and Fe line scanning at the interface of the diamond grains and the nickel layer for the MPCVD diamond process. It shows that Cr and Fe from the steel substrate effectively diffused into the nickel layer. The results suggest a good diffusion

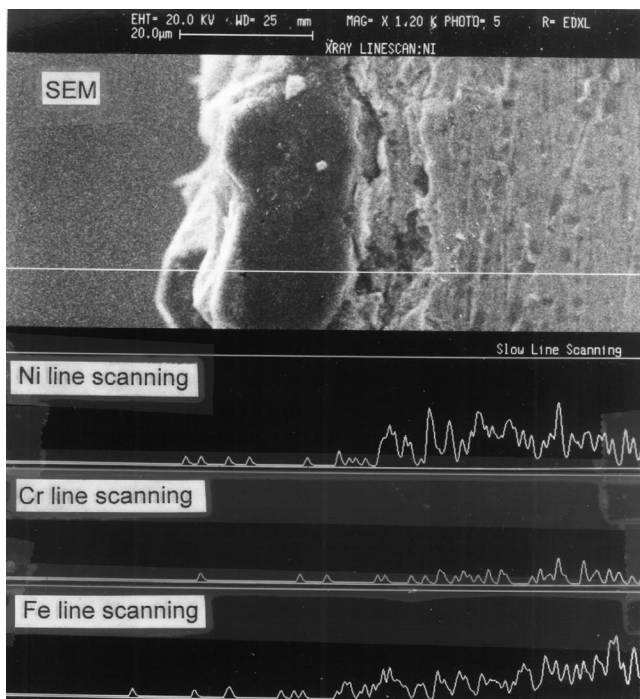


Fig. 5. Corresponding Ni, Cr, and Fe line scanning at the interface of diamond grains and the nickel layer for the present process.

bonding between the nickel layer and the steel substrate. In addition, there is a layer within the Ni layer and closer to the diamond grains side, which shows a high possibility of forming liquid Ni–Fe–Cr–C–H alloys during diamond deposition. This is in agreement with the statement that metal–carbon–hydrogen alloys can greatly enhance diamond nucleation and growth [5–11].

The wearing test of the block-on-ring tribotester was performed under a load of 20 N, rotating speed of 100 rpm, ambient temperature of 25 °C, environmental relative humidity of 80%, and a 10-min testing duration with no lubricant. Fig. 6(a and b) show the worn surfaces of these diamond tools used in the tribological properties test. Fig. 6(a) shows that the diamond grains had almost fallen off, indicating that the adherence of diamonds is not good enough. The deep worn surface also suggests that the wear resistance is poor. Fig. 6(b) shows that the diamond grains are stripped off less than those in Fig. 6(a), and the worn surface is shallow, suggesting that the adherence and wearing resistance

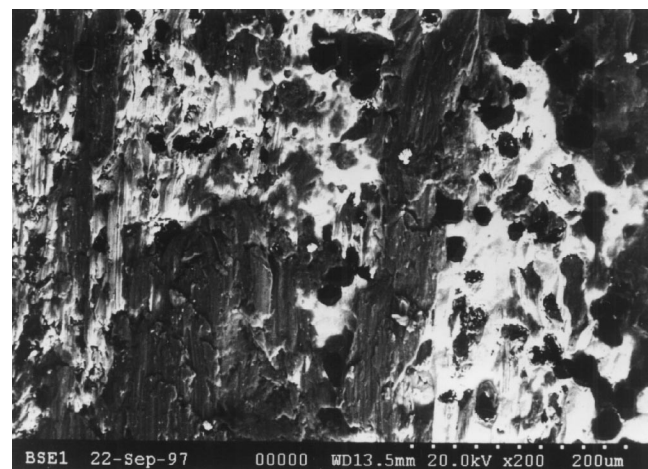
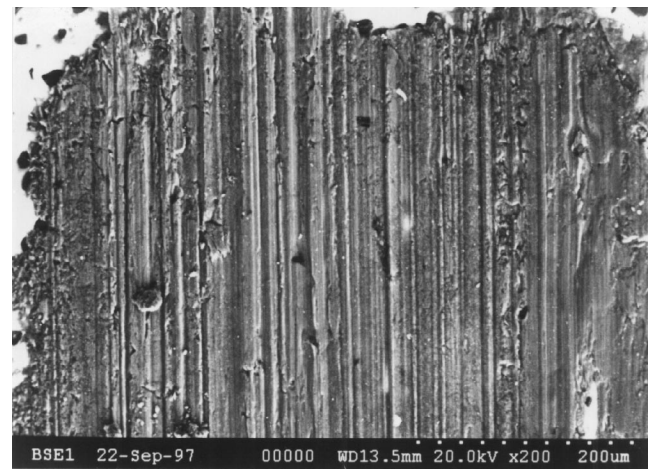


Fig. 6. Worn surface of electroplated diamond tools after the tribological properties test: (a) original, and (b) diamond grains through the growth and surface modification by this MPCVD diamond process.

are better than that in Fig. 6(a). The worn quantities of Al alloy AA6061T4 by these tools through the growth and surface modification is more than that of the original electroplated diamond tools, which also suggests that the cutting ability has been improved by this process.

#### 4. Conclusions

The growth and surface modification of electroplated diamond tools have been developed successfully in the MPCVD diamond process. The results show that diamond tools modified using this process will perform better than untreated electroplated diamond tools in several aspects, including high quality, high nucleation density, marked crystalline morphology, edge sharpness, better cutting ability, better adhesion, and better wear resistance. The reasons for these advantages in this study are as follows:

- (1) the nickel–carbon–hydrogen alloy system can effectively enhance diamond nucleation and growth,
- (2) the diamond grains are effective seeding crystals for diamond nucleation and growth, and they are effectively embedded into the nickel carbide layer to form a good mechanical interlocking, so that the adherence, wear resistance and cutting ability are improved,
- (3) Fe and Cr can form a good diffusion bonding

between nickel and the steel substrate through this process; the adherence, cutting ability and wear resistance are also promoted.

#### Acknowledgement

This work was supported by the National Science Council of R.O.C. under the contract No. NSC87-2216-E-027-003. Prof. I-Nan Lin at NTHU and Prof. K.H. Chen at Academia Sinica are gratefully acknowledged for their assistance.

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