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# Thermally Induced Defects in Industrial Diamond

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#### ABSTRACT

In this study we make use of laser heating of HTHP industrial diamond, as well as the optically measured temperature profile of the diamond surface, to study temperature induced changes to the diamond structure, both chemically and mechanically, in the absence of mechanical forces. This has relevance to the efficacy of diamond as a hard material in such applications as rock drilling and material processing. We report on the induced defects when the diamond is irradiated with high power CO<sub>2</sub> and Nd:YAG lasers respectively, and show that the thermal induced stresses in the diamond are sufficient to radically alter its physical properties, resulting in critical fracture. Raman spectroscopy, X-ray diffraction and scanning electron microscopy indicate that the heating does not result in graphitisation of the diamond, but rather diffusion from the non-diamond base results in cobalt and tungsten oxides forming on the diamond surface. This has a deleterious effect on the diamond performance.

XRD was used to determine the chemical structure of the irradiated samples. Spectrum **A1** was taken on the surface of the PCD layer while spectrum **B1** was taken inside the substrate. Figure 3 shows XRD spectra for industrial diamond heated with the Nd:YAG laser and the CO<sub>2</sub> laser, respectively. Spectra **A1** in both figures indicates that Co diffused to the surface of the PCD layer to form oxides such as Co<sub>2</sub>O<sub>2</sub>.

Keywords: laser heating; thermal stresses; polycrystalline diamond.

### INTRODUCTION

Industrial diamond is a form of polycrystalline diamond (PCD) layer on supporting tungsten carbide (WC) substrate that is sintered at high temperature and high pressure (HTHP). The nature of such a diamond has been previously described [1-2]. Industrial diamond is often called polycrystalline diamond compact (PDC). Industrial diamond has gained wide commercial acceptance in the drilling industry, since its introduction in 1973 years ago. Drill bits equipped with industrial diamond now account for a significant portion of the total footage drilled. The popularity of the use of these bits is because of their effectiveness in drilling soft to medium formations at relativity high penetration rates, but they are rarely used in cutting hard formations. However, the bits nevertheless reach very high temperature during a typical drilling or cutting process due to friction. It is believed that the high temperatures lead to stress induced damage through dislocations of the lattice. Studies on these bits were devoted to experimental and theoretical work related to the mechanical aspects of cutting and residual stress in industrial diamond. It was discovered that the industrial diamond material that is currently used deteriorates rapidly at temperatures exceeding 700 – 750 °C [3]. This is thought to be one of the main reasons that successful drilling of harder formations with industrial diamond bits has not been possible because harder formation means higher temperature.

The objective of this investigation was to study the temperature induced changes to the industrial diamond structure, both chemically and mechanically by making use of laser heating of the industrial diamond. The results of a comprehensive investigation of the structure of industrial diamond samples are presented.

# **MATERIAL AND METHODS**

The HTHP diamond that was used here, as provided by the supplier, is deposited on an alloy substrate containing WC with trace elements of iron (Fe) and cobalt (Co). The samples were cylindrical in shape with a diameter of 15 mm. The total height of the samples was 15 mm with 12 mm of WC and 3 mm of PCD. The picture of the industrial diamond that was used in this study is shown in figure 1(a).

The samples were heated by a Trump Laser Frequency excited (TLF) series  $CO_2$  laser and a Rosin Sinar DY044 diode pumped Nd:YAG laser, respectively. The Nd:YAG laser (1.064 µm) has a maximum power of 4.4 kW and the  $CO_2$  laser (10.6 µm) has maximum power 5 kW. The diameters of laser beam were 600 µm for Nd:YAG laser and 250 µm for  $CO_2$  laser. The samples were heated at the centre of the PCD surface for 8 seconds with the same laser power of 4 kW respectively.

The analyses of the two samples were carried out using Raman spectroscopy, X-ray diffraction (XRD) and Scanning electron microscopy (SEM). Raman scattering analyses were acquired with a Jobin-Yvon T64000 Raman spectrometer operated in single spectrograph mode. The 514.5 nm line of Coherent Innova 308 argon ion laser was used as the source of light in the Raman experiments. The powder X-ray diffraction generator used in this study was a Phillips PW 1830 generator operating at 45 kV and 40 mA. A Phillips X-ray diffractometer equipped with a graphite monochromator and a copper tube to collect the powder data. In the present study the investigation of morphology and qualitative analyses of industrial diamond were performed using a LEO 1525 SEM. The SEM had a high resolution and image quality of 1.5 nm at 20 kV and 3.5 nm at 1 kV for metallic and non metallic materials and also magnification of 20 – 500 000 X for morphological characterisation.



#### **RESULTS AND DISCUSSION**

The sample exposed to the CO<sub>2</sub> laser beam became visibly damaged, appearing bluish and also there was a yellowish-white powder denoted C at the interface between the PCD and substrate. There was a hole in the centre of the sample as shown in figure 1 (b). The sample was severed into two pieces with the crevice running through the centre of the hole (L1, L2). EDS spectrum confirmed that the yellowish-white powder from labelled C in figure 1(b) was WO<sub>x</sub>. The industrial diamond exposed to the Nd:YAG laser beam became visibly damaged too, with small and big material spheres on the surface of the sample. As expected, the Nd:YAG laser (1.064  $\mu$ m) led to more significant modification on the substrate and surface since the Nd:YAG laser has a shorter wavelength beam than the CO<sub>2</sub> laser and therefore can be more penetrating.



**Figure 1:** Comparison of the three samples that are: (a) initial sample, (b) heated with the CO<sub>2</sub> laser and (c) heated with Nd:YAG laser.



Figure 2 is a comparison of Raman spectra on the surface of the PCD before and after heating. These spectra show that there was chemical change on the surface of PCD due to the laser heating [6], i.e. both samples the diamond peaks (1332 cm<sup>-1</sup>) in PCD after heating are higher than that before heating. This suggests that the laser radiation has an annealing effect creating more crystalline diamond. The Nd:YAG laser seem is more effective in the annealing effect than the CO<sub>2</sub> laser. Badzian et al. [7] also observed an increase in this peak after they heated PCD with a laser and suggested that this indicates an increase in the parameter of atomic order.

Figure 3: The XRD spectra of the industrial diamond after heating with Nd:YAG laser and CO<sub>2</sub> laser, respectively. Spectra A1 in both figures indicate that Co diffuses on the surface of PCD layer to form oxides such as CoxOx for both samples and also indicate that both samples still have C on the surface of the samples.

The SEM analysis for both samples shows that the Co and W that diffused to the surface resulted in the formation of geometric structures on an otherwise smooth surface, such as spheres and rhombics, as shown in figure 4. The EDS spectra indicate that the spheres and rhombics were made of mostly Co and a trace amount of W.



**Figure 4:** SEM image of industrial diamond after heating with the (a) Nd:YAG laser and (b) CO<sub>2</sub> laser, respectively. The SEM analysis for both samples shows that Co and W diffused to the surface and result in interesting geometric structures such as spheres and rhombics.

# **CONCLUSIONS**

The industrial diamonds were studied using Raman spectroscopy, XRD and SEM to quantify the damage after irradiation by higher power lasers. Raman spectra show that both samples still contain diamond on the surface, with no graphite formation, but with the formation of metal oxides formed as a result of Co and W diffusing from the substrate. The SEM analysis for both heated samples show that Co and W (in the substrate) diffused to the surface (PCD) and resulted in geometric structures of spheres and rhombics. The XRD spectra recorded on the inside and outside of the PCD confirmed that indeed the WC diffused to the surface to form some compounds such as CoO and Co<sub>3</sub>O<sub>4</sub>.

**Figure 2:** Comparison of the three samples that are initial sample, heated with the  $CO_2$  laser and heated with the Nd: YAG laser.

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