HOMOEPITAXIAL HIGH TEMPERATURE DIAMOND GROWTH FROM OXYACETYLENE FLAMES

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ABSTRACT

High quality single crystal diamond is grown from natural seeds in a fuel-rich oxyacetylene torch at substrate temperatures of 1150-1350°C. The flame-grown diamonds are optically clear and have a Raman and photoluminescence spectrum comparable to natural, type IIa diamond with very low defect density. Laue X-Ray diffraction patterns indicate the growth is epitaxial. Growth rates were 100-200 μm/hr, both vertically and laterally. {100} top seeds showed macroscopic terraces on the top surface after growth. Scanning electron microscope pictures show that initially round seed crystals grew into octagons.

Introduction

Among the new materials that have appeared recently, diamond has received much interest for its superlative properties. With its high thermal conductivity, optical transmission range, index of refraction, bandgap and breakdown voltage, diamond is important for heat sink, electronic and optical applications (1). A method of producing large single crystal boules of diamond would be particularly important for use as windows in optical materials and as wafers in semiconductor applications.

Experimental

Polycrystalline diamond films have been grown in oxyacetylene torches by several groups(2-6). As in our group's previous work(7,8), a torch system was used to grow diamond homoepitaxially on seeds as shown in the apparatus diagram in figure 1. The torch was a standard #0 brazing torch with an orifice diameter of 0.89 mm. Feed gases were purified 99.6% C_2H_2 dissolved in acetone and ultra-high purity, 99.99% O_2, their flow rates controlled with mass flow controllers to a C_2H_2/O_2 ratio of 1.08. Acetone was removed from the C_2H_2 line by adsorption onto activated charcoal, while the O_2 was used without further purification. The seed was brazed with a Au-Ta braze to the face of a Mo screw, the height of which could be adjusted in a water-cooled Cu block to control the temperature. A two-color infrared pyrometer moitored the seed surface temperature, which was kept at 1250±20 °C. The flame jet impinged upon the screw, with the seed crystal remaining within the flame feather. A Mo screw with a pinhole in its top face was connected to a differentially pumped mass spectrometer. The gases in the boundary layer will be sampled and the ratio of C, H and O atoms will be correlated to the growth conditions and rates.

Results and Discussion

Figures 2a and 2b show scanning electron micrographs of the diamond seed crystal before and after a one hour-long growth. The top {100} face grew into a regular octagon. The growth rate was ~100 μm/hr. The top face of the crystal is smooth except for 10 μm wide steps and optical microscopy shows the grown diamond to be remarkably clear. The Raman spectrum of the flame-grown diamond, shown in figure 3, has the same peak position and width as natural type IIa diamond within the limits of the instrument. A very small fluorescence background can be seen, but no non-diamond carbon can be detected in the spectrum.

The Laue X-Ray diffraction photograph shown in figure 4 shows that the diamond is a single crystal and that the growth is epitaxial. This is also seen from the trapezoidal side faces in figure 2b which correspond to the {111} faces on a cubo-octahedron.

Figure 5 shows a photoluminescence spectrum of the flame-grown diamond at 6 K. It was collected at a wavelength of 488 nm and a power of 20 mW. The major features seen besides the normal diamond Raman peak at 2.375 eV (b) are at 2.155eV and 1.946 eV (d, e) and correspond to a N-vacancy complex and pair respectively, which are normally seen in flame-grown diamond (5). A small background luminescence seen across the entire spectrum is probably due to recombination of extended defects, which is seen in natural diamond as well.

Conclusions

High quality single crystal diamonds were grown in flames. The diamonds were exceptionally clear and have Raman and photoluminescene spectra showing high purity and low defect densities. Flame-grown diamonds grow at high rates and high temperatures suggesting a possible method for growing diamond boules.

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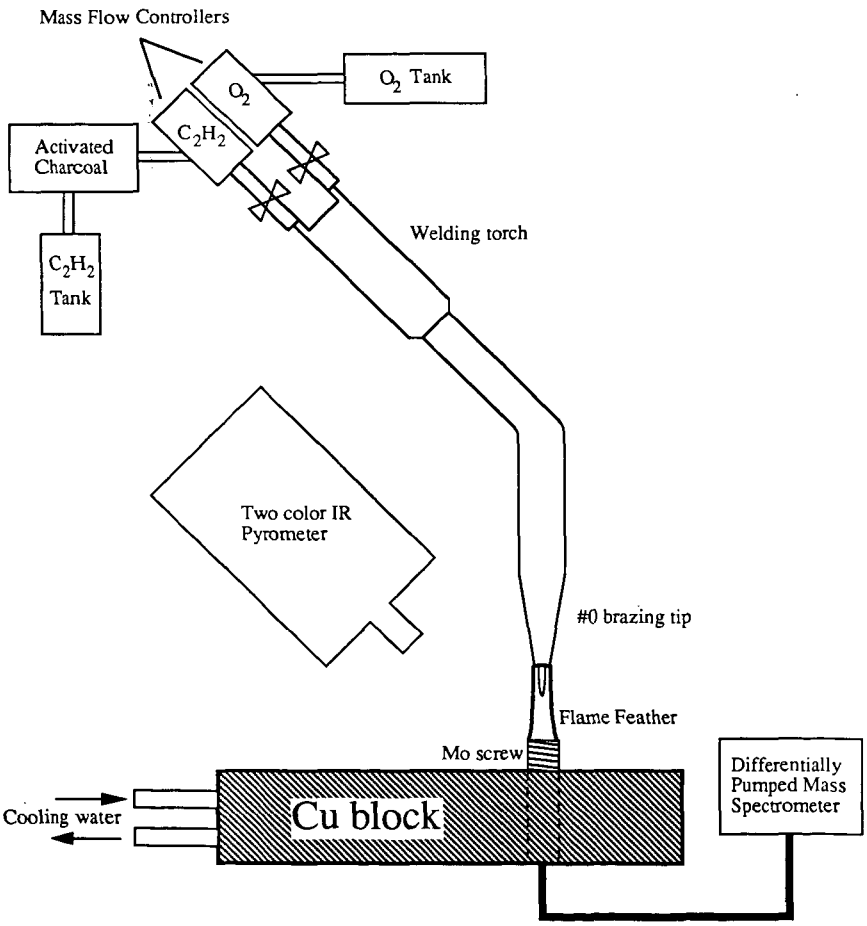

Figure 1. Oxyacetylene torch diamond growth apparatus.

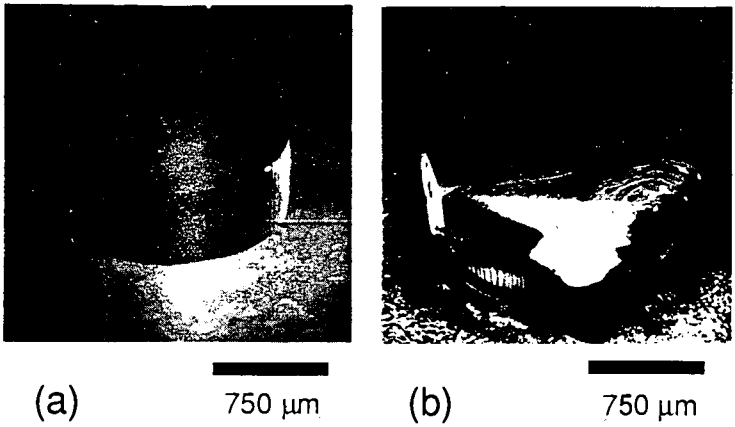

Figure 2. SEM photo of the seed diamond before (a) and after (b) growth in torch.

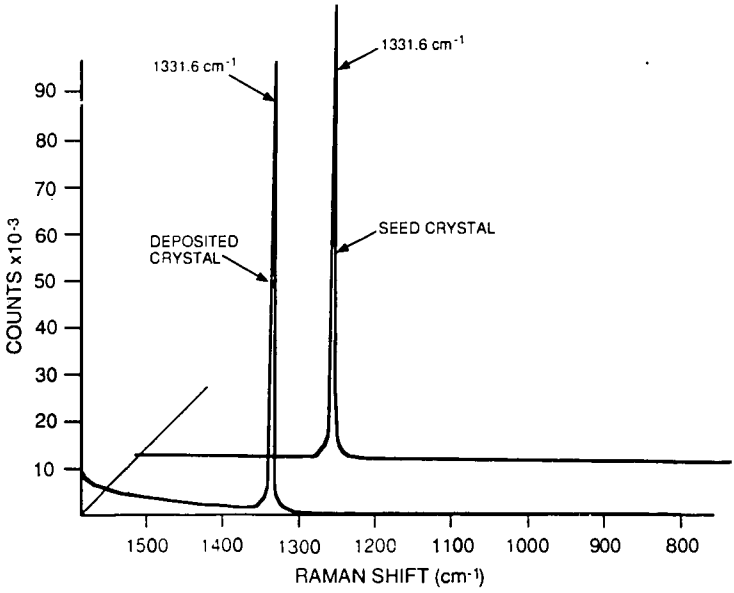

Figure 3. Raman Spectrum of the seed and flame-grown diamonds.

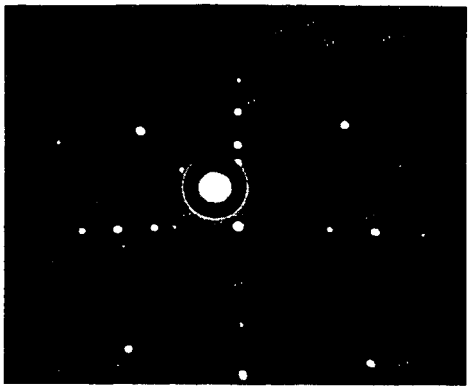

Figure 4. LaueX-Ray diffraction pattern of the flame-grown diamond.

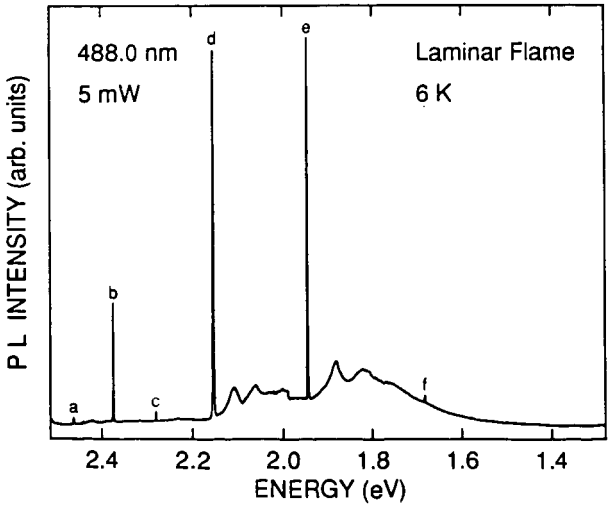

Figure 5. Photoluminescence Spectrum of the flame-grown diamond.