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Microstructure and Oxidation Resistance of Nanometer Sized SiC Coated Diamond Particles[†]

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Abstract

Diamond particles were completely covered with a very thin SiC layer of ~200nm thick by a reaction of SiO vapor with diamond. The oxidation temperature of the SiC coated diamond particles started at about 920 $^{\circ}$ C, which is 400 $^{\circ}$ C higher than that of diamond without SiC coating. The growth mechanism of the SiC layer and the oxidation resistance of the SiC coated diamond is reported.

KEY WORDS: (SiC coating), (Diamond particles), (Activation energy), (Oxidation resistance)

1. Introduction

Diamond particles are used industrially for cutting tools and abrasives. However, the diamond is unstable under normal pressure and easily oxidized to carbon dioxide in air above 530°C. Additionally, transition metals such as iron, nickel, and cobalt react with diamond at around 1150°C and convert it to graphite. We have developed a new coating process of SiC on diamond particles to prevent oxidation and corrosion^{1) 2)}. In this study, the growth mechanism of the SiC layer and the oxidation resistance of the SiC coated diamond were investigated.

2. Experimental procedure

Diamond powders with the particle size of $20\sim30\,\mu$ m (Sumitomo Electric Industries Co., Itami, Japan) were used for the SiC coating. SiO powders (99.9% pure, Nacalai Tesque Co., Kyoto, Japan) were provided as the silicon source. The diamond powders were placed on the SiO powder bed via a carbon felt as illustrated in Fig. 1. This assembly was covered with carbon sheets in an alumina crucible to keep the gas pressure of SiO in the crucible and heated at $1250^{\circ}\text{C}\sim1450^{\circ}\text{C}$ in vacuum at about 0.03Pa for $1\sim90\text{min}$.

The diamond powders after the coating treatment were characterized by XRD and EDX. The surface morphology was observed using SEM. The thickness of the SiC layer and the oxidation resistance of the SiC coated diamond were evaluated by thermogravimetric (TG) measurement.

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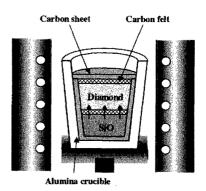


Fig. 1 An assembly for SiC coating of diamond particles.

3. Results and discussion

3.1 Growth mechanism of the SiC layer

Figure 2 shows the relationship between the thickness of the SiC layer, which was calculated using the mass of SiC layer measured by TG, and the holding time at different treatment temperatures. The thickness of the SiC layer increased linearly with increases of the holding time.

Figure 3 plots the mass gain ratio due to the formation of the SiC layer on diamond as a function of coating time at various temperatures. The weight of the SiC layer increases linearly with time. This result suggests that the growth of the SiC layer is not controlled by the self-diffusion of Si or C atoms through SiC, but mainly by precipitation or deposition of SiC from the vapor

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phase reactions. The following vapor-solid reactions account for the linear growth of the SiC layer with coating time.

$$SiO(g) + 3CO(g) \rightarrow SiC(s) + 2CO_2(g) \cdot \cdot (1)$$

$$CO_2(g) + C(s) \rightarrow 2CO(g) \cdot \cdot \cdot \cdot \cdot (2)$$

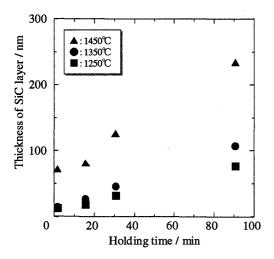


Fig. 2 Relationship between thickness of the SiC layer and holding time.

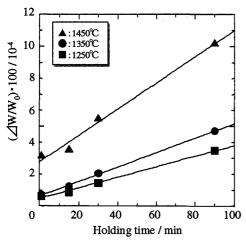


Fig. 3 Mass gain with reaction between SiO(g) and C(dia). W_o is the initial weight of diamond before SiC coating. ΔW is the weight gain after coating.

Figure 4 shows the Arrhenius plot of the rate constants that was calculated using the mass gain data by the least-square method. The apparent activation energy is 100 kJ/mol. Shimoo et al. calculated the apparent activation energy for the formation of a SiC layer on a graphite plate based on the reaction (1) and obtained 97 kJ/mol³). Both activation energies show an excellent agreement.

Figure 5 shows SEM photographs of the surface of SiC coated diamond particles treated at 1350°C. Tiny granules

of SiC were deposited and aggregated with the increase of the holding time. Even for samples treated for 1min and 15min, the whole surface is considered to be covered with a thin SiC layer because they show good oxidation resistance, as shown later. EDX analysis shows the uniform distribution of Si atoms on the whole surface of a SiC coated diamond particle as well.

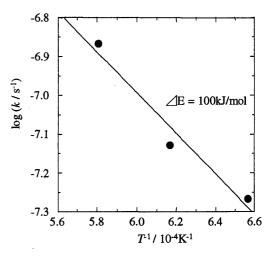


Fig. 4 Arrhenius plots for liner rate constant k of reaction SiO(g) and C(dia).

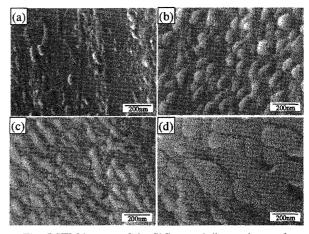


Fig. 5 SEM images of the SiC coated diamond treated at 1350°C for (a) 1min, (b) 15min, (c) 30min, and (d) 90min.

3.2 Oxidation resistance of the SiC coated diamond particles

Figure 6 shows the starting temperature for oxidation of SiC coated diamond particles as a function of coating time at various temperatures. The coating at 1350°C shows superior oxidation resistance. When the total coating time is 90min (3 times coating of 30min), the starting temperature of oxidation reaches about 920°C which is 400°C higher than that of diamond without coating. Even treated only for 1min, it increased to 750°C. This result suggests the formation of a thin protective layer of SiC on the diamond. Other coatings at

1250 °C and 1450 °C showed lower oxidation resistance. It is reported that the transformation to graphite from diamond and the generation of cracks in diamond starts at over 1400 °C $^{4)}$. The coating at 1250 °C for 90 min exhibits no improvement against oxidation compared to the coating for 1 min. The SiC coating has poor adhesion at 1250 °C.

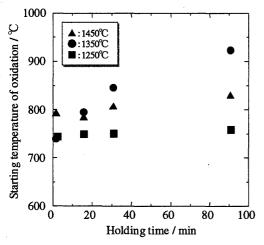


Fig. 6 Starting temperature of oxidation for SiC coated diamond particles.

4. Conclusions

Nanometer sized β -SiC granules were coated on diamond particles by the reaction of SiO and diamond in vacuum. The growth mechanism of the SiC layer on

diamond particle and their oxidation behavior were studied. The results can be summarized as follows.

- (1) The growth mechanism of the SiC layer on diamond occurs in two steps. In the first step, a thin SiC layer with a thickness of about 15 nm is formed by the reaction between SiO(g) and diamond. In the second step, nanometer sized SiC granules are deposited on the SiC layer by the vapor phase reaction between SiO(g) and CO(g).
- (2) The apparent activation energy for the formation of the SiC layer on diamond surface is 100 kJ/mol, which is almost the same energy reported for the SiC formation on graphite by the reaction of SiO(g) and CO(g).
- (3) The oxidation resistance of diamond particles is remarkably improved by SiC coating treatment at 1350 °C.

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