

ENLARGEMENT OF MICROWAVE PLASMA REGION AND DIAMOND DEPOSITION AREA

Mutsukazu KAMO,¹ Fumio TAKAMURA,² and Yoichiro SATO¹

¹National Institute for Research in Inorganic Materials, 1-1 Namiki, Tsukuba, Ibaraki 305, Japan

²New Japan Radio Co. Ltd., 2-1-1 Fukuoka, Kamifukuoka, Saitama 356, Japan

Attempts were made to enlarge the microwave plasma region and diamond deposition area. Four techniques were tried to generate microwave plasma over a wide range. A method introducing microwaves into a rectangular cavity from two directions was considered to be suitable for diamond deposition on a large substrate. Diamond film was obtained on a silicon wafer 100 mm in diameter by the method.

1. Introduction

The microwave plasma method¹⁾ for diamond synthesis has a high stability of experimental conditions during deposition and the products are reproducible. But it is difficult to obtain a plasma region larger than 60 mm in diameter because of the loss of microwaves by leakage and the inhomogeneity of plasma intensity.

This report deals with methods for the enlargement of the plasma region and diamond deposition on substrates 100 mm in diameter. Methane concentration, flow rate, pressure, the addition of argon, rotation of the substrate holder, plasma intensity and the plasma position on the substrate were studied as to their effects on the homogeneity of diamond film thickness and on the film quality. Diamond was obtained as a film on a silicon wafer 100 mm in diameter using a method introducing microwaves into a rectangular cavity from two directions.

2. Experimental

2.1 Enlargement of plasma region

Four techniques, shown in Fig. 1(a)–(d), were tried to generate microwave plasma over a wide range. Plasma generated using these methods was studied with respect to the shape, size, and position in the reaction chamber.

Figure 1(a) shows a cylindrical cavity, in which a

substrate is placed perpendicular to the electric field. Plasma in the cavity shifted from the center area to the edges of the substrate with increasing microwave power. Therefore, a wide plasma area was not obtained by this method. Figure 1(b) shows another cylindrical cavity, in which a substrate is placed parallel to the electric field. The substrate in the cavity was not heated to the desired temperature range for diamond deposition because the substrate was not surrounded with plasma. Figure 1(c) shows a microwave oven capable of holding a large substrate. It was difficult to generate plasma on the substrate in the oven. Figure 1(d) shows a rectangular cavity introducing microwaves from two directions; plasma about 130 mm in length and 50 mm in width was obtained at the center of the cavity by this method. The substrate was covered by the plasma over the whole area under rotation. Figure 1(d) is therefore considered to be suitable for diamond deposition on a large substrate because of the wide plasma area and the capability to heat substrates to the proper temperature range without an additional heat source. No wide plasma area was obtained using the other three methods. In addition, it was difficult to heat substrates to the temperature range 700–1000°C, which is the desired range for diamond synthesis, without an additional heat source. In this study, diamond deposition was carried out using the apparatus shown in Fig. 1(d).

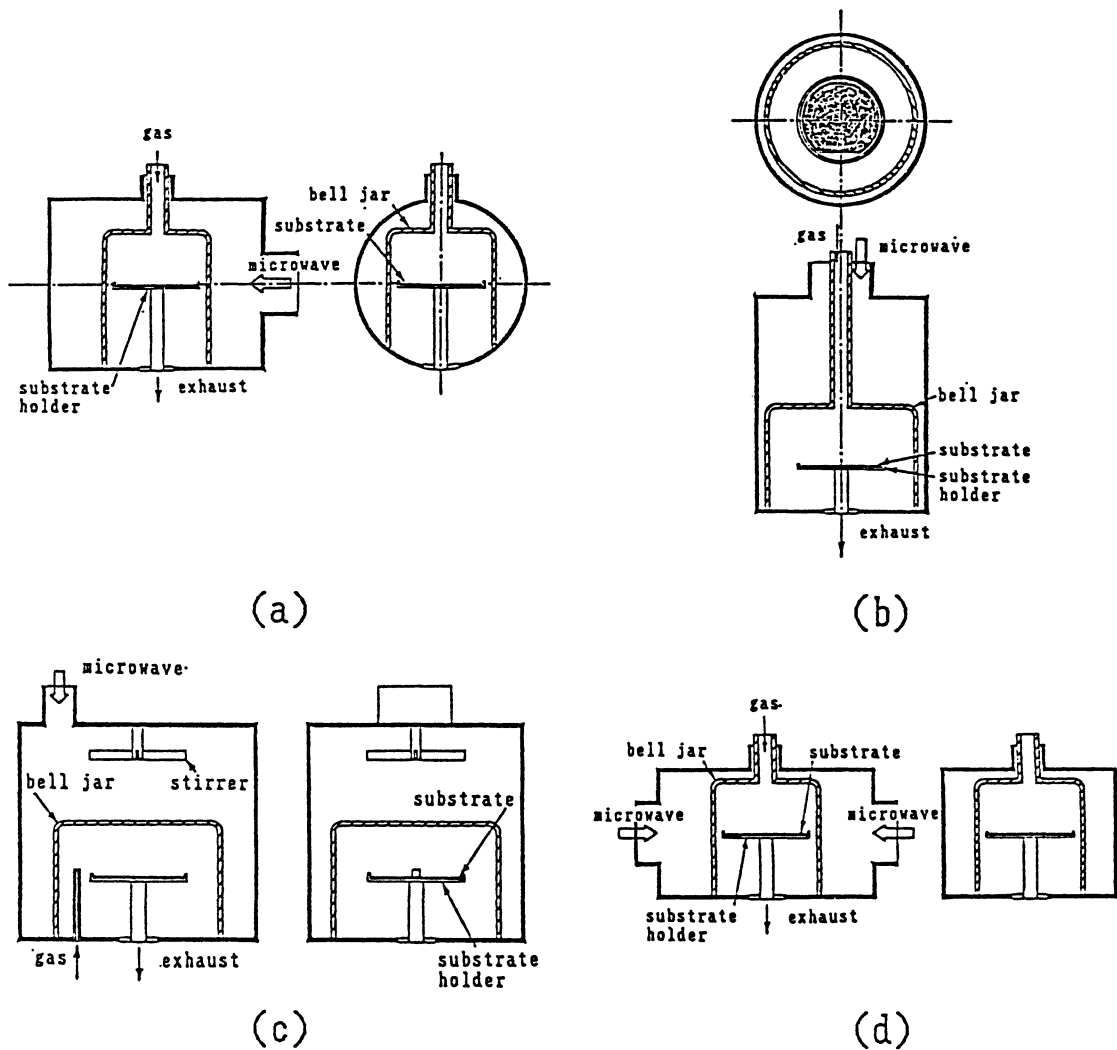


Fig. 1. Schematic diagram of the apparatus for enlargement of microwave plasma region. (a) and (b); cylindrical cavities, (c); microwave oven, (d); rectangular cavity.

2.2 Synthesis of diamond

A silicon wafer 100 mm in diameter was used as a substrate. The mirror-polished surface of a commercially available silicon wafer was roughened using diamond powder 8–16 μm in particle size. After diamond deposition, the substrate was divided into 20 fragments 5 mm \times 5 mm in size from the center to the edge. Each fragment was studied by scanning electron microscopy (SEM) and Raman spectroscopy. Experimental conditions are shown in Table 1.

Table 1. Conditions of diamond film synthesis

Microwave power (kW)	1.5–2.6
Source gas	H ₂ , CH ₄
Methane concentration (vol%)	0.5, 1.0
Flow rate (sccm)	100, 250, 500
Pressure (kPa)	2.7, 3.3, 4.0
Rotation of	
substrate holder (r.p.m.)	4, 8, 16
Substrate temperature at	
the center area (°C)	860
Argon concentration (vol%)	0, 1, 20
Reaction time (hour)	20

3. Results and discussion

Plasma distribution in the cavity is considered to be one of most effective factors in obtaining uniform diamond film on the whole substrate area. It may be predicted that diamond film with maximum thickness at the substrate center would be prepared using a plasma about 130 mm in length and 50 mm in width in the cavity shown in Fig. 1(d), because the center always contacts plasma while the edge intermittently contacts plasma. Three kinds of plasma, with different intensities at the center of the cavity, were used for diamond synthesis. The lines labeled (a), (b) and (c) in Fig. 2 show the decreasing thickness distributions of diamond films obtained using the three kinds of plasma. Thickness distributions (a), (b) and (c) in Fig. 2 were obtained under the plasma with different intensities at the center, which decrease in that order. The results in Fig. 2 indicate that the alteration of plasma intensity is effective for obtaining diamond film with uniform thickness.

Diamond films with uniform thickness were obtained at the center area 60 mm in diameter using the plasma of which the intensity was weaker at the center area than at the edge area. The effects

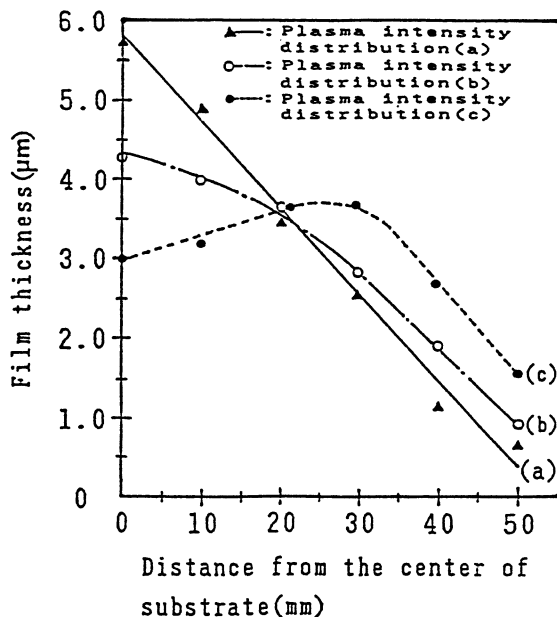


Fig. 2. Thickness distributions of diamond films obtained under the plasma with different intensities at the center area.

of the addition of argon, rotation of the substrate holder, plasma distribution and plasma position on the substrate were studied to obtain homogeneous diamond thickness over the whole substrate area.

Plasma enlargement due to the Penning effect is expected by the addition of argon to reaction gas. The addition of argon somewhat improved the distribution of diamond film thickness, but film with a homogeneous thickness was not obtained over the whole area.

In addition to the alteration of plasma intensity, the effect of plasma position on the substrate on film thickness was also studied. Figure 3 shows the thickness distributions of diamond films synthesized using plasma generated at different positions. Figure 3(a) shows the thickness distribution of film prepared using plasma generated on the center line of the rotating substrate. Thickness distribution of film synthesized under plasma about 15 mm from the center line is shown in Fig. 3(b). These results indicate that the thickness distribution may be controlled by the plasma position on the substrate.

Furthermore, a diamond film with uniform thickness seems to be obtained by the alteration of

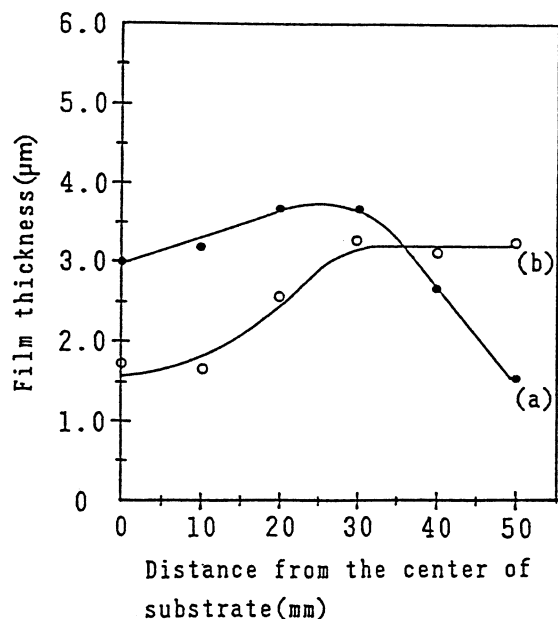


Fig. 3. Thickness distributions of diamond films prepared under the plasma generated at different positions on a substrate.

the reaction time under the plasma at different positions, as well as alteration of the plasma intensity. Figure 4 shows the plasma shape used for the synthesis of diamond film shown in Fig. 5. The reaction times of (a), (b) and (c) in Fig. 5 under the plasma (I) and (II) shown in Fig. 4 were as follows: (a); 14 hrs in (I) and 6 hrs in (II), (b); 15 hrs and 5 hrs, (c); 16 hrs and 4 hrs, respectively.

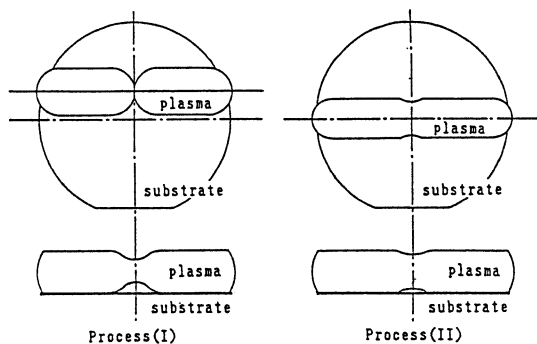


Fig. 4. Illustration of the position and shape of the plasma used for the improvement of the thickness distribution shown in Fig. 5.

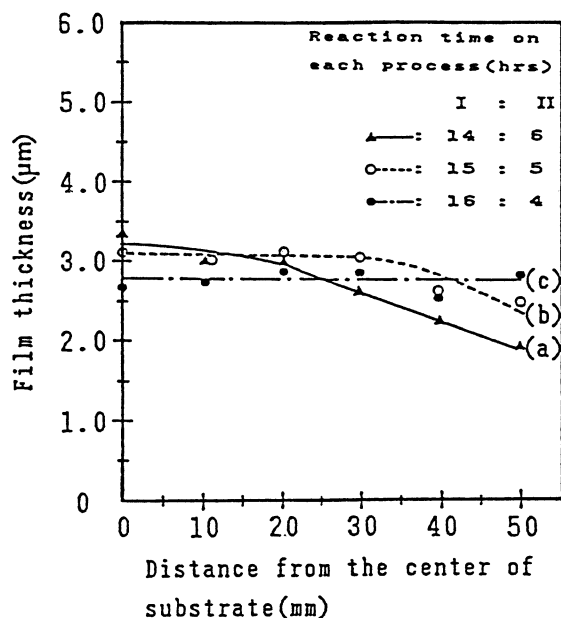


Fig. 5. Thickness distributions of diamond film synthesized by the combination of the methods shown in Fig. 4.

The results shown in Fig. 5 indicate that the combination of the plasma intensity and the plasma position is effective in obtaining the diamond film with uniform thickness; such a uniform film on the whole area of the substrate 100 mm in diameter was prepared by the method shown in Fig. 5(c).

The Raman spectra of diamond films obtained by the method of Fig. 5(c) are shown in Fig. 6. Good quality diamond generally tends to be obtained at the edges of a substrate compared with the central part. The inhomogeneity seems to be due to heterogeneous substrate temperature.

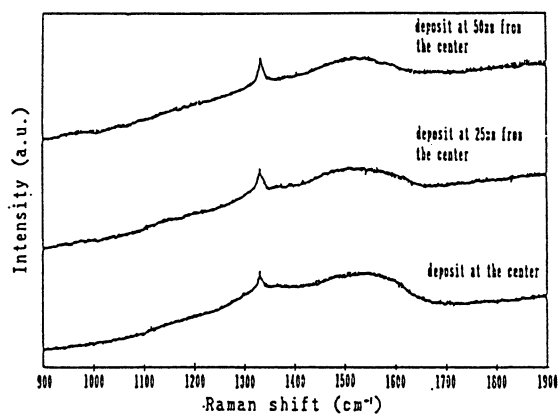


Fig. 6. Raman spectra of a diamond film obtained on the substrate 100 mm in diameter (Fig. 5(c)).

4. Summary

- 1) The microwave plasma region was enlarged by a method introducing microwaves from two directions into a rectangular cavity.
- 2) Diamond film was obtained uniformly on the whole area of a substrate 100 mm in diameter by the combination of two types of plasma modes.
- 3) Good quality diamond tends to be obtained at the edge of a substrate compared with the central part.

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