

# Reaction products at brazed interface between Ag–Cu–V filler metal and diamond (111)

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**Abstract** The composition of a brazing-filler metal in an Ag–Cu–V system which was selected using lattice misfit data, was estimated using perturbation interface model and applied it to the brazing artificial single crystal diamond (111) by unidirectional-solidification brazing. An Ag-27.8Cu system containing less than 1mass%V brazing-filler metal provided stable joint strength (the shear strength at the brazed interface exceeded 200 MPa). Optical observation of the brazed interface revealed that silver crystal grains grew from vanadium carbide islands formed on the diamond. This behavior is consistent with a slight degree of lattice mismatch between silver and vanadium carbide crystals. Atomic force microscope observation revealed small scale islands of the reaction products with good adhesion are enough for brazing diamond (111). X-ray diffraction results indicated several types of vanadium carbides,  $V_8C_7$ ,  $V_4C_3$  and  $V_2C$  were formed there, and  $V_4C_3$  reaction product was considered to provide good adhesion between the filler metal and the diamond due to prefer solidification of silver on the reaction products islands.

## Introduction

A diamond (111) surface has superior properties of hardness and a lower wear rate than (100) and (110) surfaces [1, 2]. When a clean environment is required, the surface of diamond (111) is expected to provide a wear-free film for mechanical components (such as a precision guideway) by brazing the diamond to metal parts. For example, precision stages of a scanning probe microscope in ultra high vacuum (UHV) are made of sapphire plate.

Diamonds have been brazed to metal parts by a silver–copper (Ag–Cu) eutectic filler containing titanium as a reactive metal. The diamond (111) surfaces joined by this filler metal were sometimes fractured by shear stress as low as 56 MPa, whereas diamond (100) surfaces joined by the same filler in the same brazing condition were not fractured by the shear stress of exceeded 120 MPa [3]. It is thus necessary to search for a proper reactive metal based on a crystal lattice of a reaction product with diamond (111).

A reactive metal element was selected from  $IV_A$  and  $V_A$  groups in the periodic table in order to form a carbide via a reaction with diamond (111) for wetting [4, 5]. Titanium was replaced by vanadium because 0.1607 misfit between vanadium carbide (111) and diamond (111) is smaller than 0.2125 misfit between titanium carbide (111) and diamond (111) [3]. The misfit is used to explain the film growth morphology [6]. It is the difference between the film and substrate lattice constants normalized by the substrate lattice constant.

The analysis in the around study progress predicted that preferential solidification of silver on the VC islands would occur due to the slight degree (0.01298)

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of lattice mismatch between Ag (111) and VC (111) in the unidirectional-solidification brazing, where brazing-filler metal solidifies from the diamond surface by cooling the diamond. The lattice mismatch parameter called the interfacial planar disregistry was introduced to explain the heterogeneous nucleation. This detailed definition and the calculation are described in the references [3, 7–9]. Physical bonds such as Gleiter's lock-in model [10] from the VC islands to the filler through Ag crystal grains will be generated at the interface. The analysis also predicted that copper would solidify from the diamond due to the slight degree of lattice mismatch (0.01350) between Cu (111) and diamond (111). The Cu crystals that grew from the diamond relax stress at the brazed interface [3].

Two parameters, the temperature gradient and the growth rate during the unidirectional-solidification brazing process, play important roles in forming the columnar structure consisting of the Ag and the Cu crystals at the interface. An ideal joined boundary morphology produced by the brazing is considered to have a cell structure [9]. This is expected to join a large area of diamond (111) surface to metal parts without cracking.

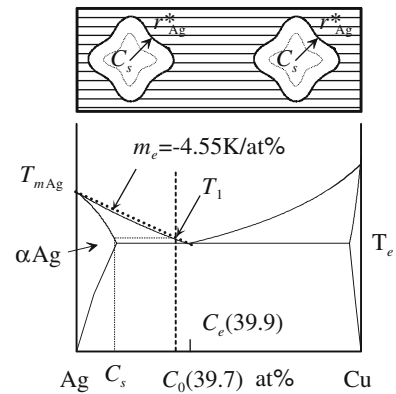
### Theoretical determination of composition of brazing-filler metal

The amount of vanadium added to the brazing-filler metal was determined as follows. First, it may be assumed from the following information that supercooling can arise in the Ag–Cu eutectic system.

The cell structure at the brazed interface can be formed by unidirectional solidification using two parameters, the temperature gradient,  $G_L$ , and the growth rate,  $V$ . Rapid cooling and a wide range of solidified temperature are needed for the structure. Figure 1 shows the Ag–Cu phase diagram and the schematic illustration of the primary crystals in the melt. Constitutional supercooling [11] is necessary to produce the structure at the brazed interface. The required condition is expressed in the following relation:

$$\frac{G_L}{V} \leq \frac{m_e C_0 (1 - k_e)}{k_e D_L}, \quad (1)$$

where  $m_e$  is the slope of the equilibrium liquidus curve,  $k_e$  is the equilibrium distribution coefficient,  $D_L = 5 \times 10^{-9} \text{ m}^2/\text{s}$  is the diffusion coefficient of the solute and  $C_0$  is its concentration in the melt. The value of  $D_L$  is one used as the metal solute in a



**Fig. 1** Ag–Cu phase diagram and schematic illustration of the primary crystals in the melt

typical liquid metal [11]. In this estimation case, the Cu concentration of the eutectic composition,  $C_e = 39.9$  at%, was substituted for  $C_0$ . The right side of Eq. 1 was calculated to be  $6.57 \times 10^{10}$  using  $m_e = 4.55 \text{ K/at\%}$  and  $k_e = 0.356$  in the Ag–Cu system. The maximum value of  $G_L$  produced by our unidirectional-solidification brazing is  $6 \times 10^6 \text{ K/m}$ . The minimum value of  $V$  is  $1 \times 10^{-4} \text{ m/s}$  when  $G_L$  is equal to  $6 \times 10^6 \text{ K/m}$ . These values satisfy our brazing conditions in a vacuum.

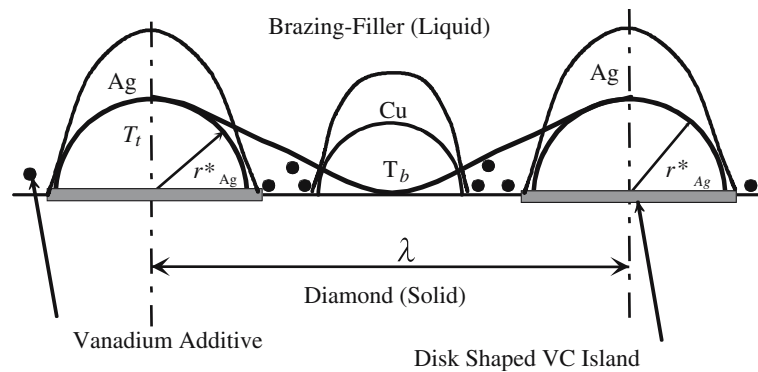
The radius of the Ag hemisphere formed on a VC island is related to the turbulence wavelength  $\lambda$  of the solid–liquid boundary in perturbation theory [12, 13]. The critical radius,  $r^*$ , is given by the relation of  $\partial G_{\text{hom}} / \partial r = 0$ , where  $G_{\text{hom}}$  is Gibbs free energy for homogeneous nucleation, and  $r$  is the radius of the nucleus.  $r^*$  is obtained from

$$r^* = \frac{2\sigma_{\text{LS}}T_m}{\rho\Delta H\Delta T}, \quad (2)$$

where  $T_m$  is the melting temperature,  $\sigma_{\text{LS}}$  is the interfacial free energy of a solid,  $\rho$  is its density,  $\Delta H$  is the heat of fusion, and  $\Delta T$  is the degree of supercooling. Here, the critical radius of silver,  $r^*_{\text{Ag}}$ , is estimated to be 113.4 nm using  $\Delta T = 2.0 \text{ K}$  based on a measured value for silver in a brazing system, experimentally,  $\Delta H/T_{\text{mAg}} = 11.3/1235 \text{ kJ/mol} \cdot \text{K}$ ,  $\sigma_{\text{LS}} = 101 \times 10^{-3} \text{ J/m}^2$  [14], and  $\rho_{\text{Ag}} = 9.733 \times 10^4 \text{ mol/m}^3$ . From a perturbation model of the cross-section of brazed interface as shown in Fig. 2, it is assumed that the growth of the Ag hemispherical embryo on the VC reaction product island occurred to the critical nucleus of  $r^*_{\text{Ag}}$  by preferred solidification of silver. The condition to produce the cell structure at the brazed interface is given by

$$\lambda \geq 4r^*_{\text{Ag}}. \quad (3)$$

**Fig. 2** Ideal model of cross-section of diamond boundary joined by unidirectional-solidification brazing method



The minimum amount of vanadium content is estimated as follows. The shapes of the VC islands are assumed to be disk-shapes. The minimum diameter of the island is assumed to be  $2r^*_{Ag}$ . The minimum distance between them is considered to be  $\lambda$  as shown in Fig. 2. One island exists in an area of a parallelogram with one side of  $\lambda$  based on the orientation of diamond (111) surface as shown in Fig. 3. The surface-area percentage of the disk-shaped VC islands with the minimum radius formed on the diamond surface is calculated to be 22.5%. A fine brazed interface is considered to be formed when less than 22.5% of the diamond surface is covered by the islands. The disk thickness is assumed to be 5 nm, and it is twice as high as the average height of the islands measured by the AFM observation in section ‘Reaction products for heterogeneous nucleation’. The reaction product is supposed to be a stoichiometric VC (NaCl-type crystal) compound [15], and it is based on the X-ray dif-

fraction results in section ‘Solidified structures’. There are  $1.138 \times 10^7$  V atoms in one disk. When a 6-mm-wide, 6-mm-long and 0.1-mm-thick sheet of the Ag–Cu eutectic brazing-filler metal is used, the theoretical minimum amount of vanadium is 0.2 micrograms, which means 0.0005 mass%.

The Cu concentration is determined by the difference between the temperature at the top of the Ag critical nucleus,  $T_t$ , and that at the diamond surface (bottom of the perturbation wave),  $T_b$ , which is given by Eq. 4

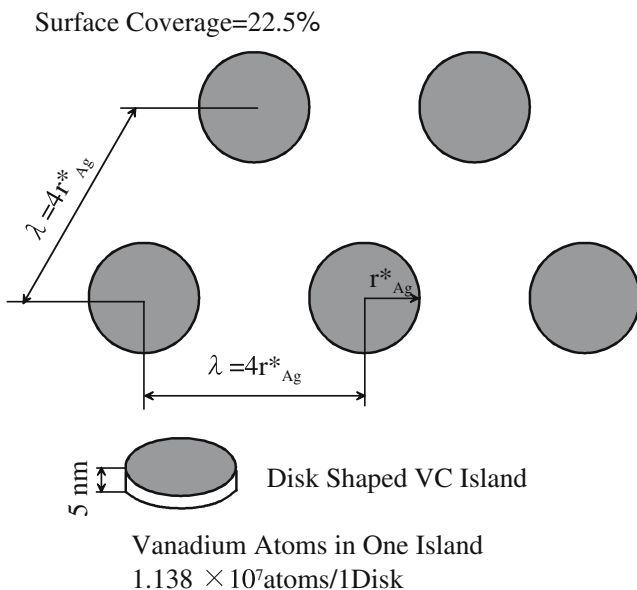
$$T_t - T_b = r^*_{Ag} G_L \tag{4}$$

The heterogeneous nucleation of copper from the diamond surface occurs when  $T_b = T_e$ , where  $T_e$  is the eutectic temperature of the Ag–Cu system. From the equilibrium phase diagram of the Ag–Cu system and from the relation,  $T_t \geq T_{mAg} + m_e C_0$ , the following relationship for the concentration of copper in a new brazing-filler metal is derived.

$$T_{mAg} + m_e C_0 < T_e + r^*_{Ag} G_L \tag{5}$$

As a result,  $C_0 = 39.7$  at% is obtained. This indicates that 0.2 at% copper should be decreased as compared with  $C_e = 39.9$  at%, and that the  $\alpha$ Ag primary crystals should grow from the VC islands prior to the Cu crystal growth.

As a result, the theoretically determined composition of the brazing-filler metal for diamond (111) is Ag-27.9Cu-0.0005V (mass%).



**Fig. 3** Vanadium carbide reaction product island model on diamond (111)

**Experimental**

**Specimens**

Artificial diamonds (Sumicrystal: Ib type) were joined to 42 Invar alloy plates (Nilaco corporation: Ni + Co 41–43%, Mn 0.7–1.25%, Si < 0.3%, C < 0.1%, Fe Bal.). The plates were 0.2 mm in thick. This alloy has

such a small coefficient of thermal expansion as a glass at 670 K that thermal stress in the diamond is expected to be relaxed.

The brazing surfaces of diamond (111) were prepared by cleaving the diamonds. These cleaved surfaces were not polished because it was hard to polish these wear-free surfaces.

Silver powder (purity 99.9%) containing vanadium hydride powder (purity 99%) that had passed through a 325-mesh screen was sandwiched between two  $6 \times 6 \times 0.05$  mm sheets of the Ag–Cu eutectic foils at a ratio of 1 mass%. The mixed powder did not contact with the diamond, till the eutectic foil metal melted in order to investigate the vanadium reaction products with the diamond. The content percentage of vanadium was the minimum one because it is difficult to mix small amounts of the powder. The composition for the experiment became Ag-27.8Cu- less than 1 V (mass%).

#### Unidirectional-solidification brazing

Equipment for unidirectional-solidification brazing in a vacuum chamber at  $3 \times 10^{-2}$  Pa is shown in Fig. 4. The diamond specimen and the brazing-filler metal were placed on 42 Invar alloy plate Number 2. The plate was directly heated by electricity to melt the brazing-filler metal. Four thermocouples were used during the brazing process. Thermocouple A (Number 1) is located in the center of the plate to measure the brazing temperature. Thermocouple B (Number 4) is inserted in the copper cooling mass (Number 3) to measure its temperature. Thermocouples C (Number 5) and D (Number 6) are located near the edge of the copper cooling mass to measure the heat flux. The gray objects are insulators.

After the brazing-filler metal had entirely melted, the temperature of the brazing specimen was kept at the fixed brazing temperature for the brazing time. The brazing temperature and time were set to the values from several experimental combinations that let the vanadium additive react with the diamond according to

a differential thermal analysis for vanadium reaction products with graphite. The brazing temperatures were 1060 K, 1090 K and 1120 K, and the brazing time was set to 240 s. The joined specimen began to cool when the diamond floating on the melt contacted the cooling mass, which acted as a chill, and the stage (Number 8) was moved up by the springs as the stainless steel wire (Number 9) was gradually loosened. The melt started to solidify from the diamond interface when the heat flux in the cooling mass was measured to be about  $1.8 \times 10^6$  W/m<sup>2</sup>. When the melt solidified entirely, the alloy plate was also cooled by turning off the electricity.

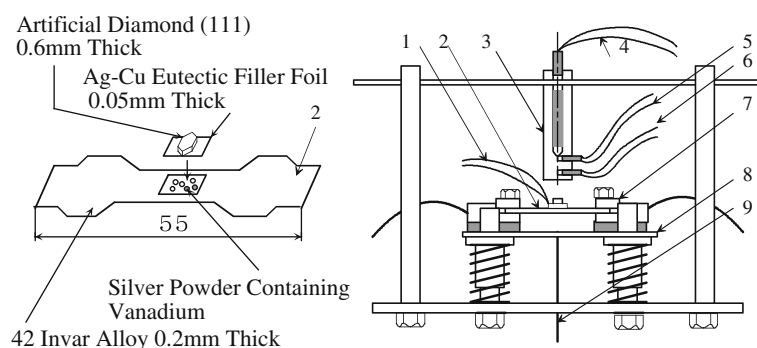
#### Observation procedure for NC-AFM

A flat-brazed interface specimen for a non-contact mode atomic force microscope (NC-AFM) observation was prepared in order to investigate the shape and size of the reaction product island of the nucleant for the brazing-filler metal. The alloy plate and the most brazing-filler-metal were removed from the interface. The interface was polished until it became as flat as the diamond surface. A small amount of the brazing-filler metal still remained there. The specimen was put in a vacuum chamber and was heated to the melting temperature of the brazing-filler metal. The melt on the diamond surface was vaporized, and a much smaller amount of it remained in a hemispherical shape. We observed the surfaces where it had adhered by NC-AFM.

#### Strength test method

The strength of the brazed interface was examined using shear test equipment previously developed [3]. A slit was made in every strength-test specimen. The alloy plate and the brazing-filler metal were cut perpendicular to the tensile direction using a thin grindstone, and applied a tensile load to the alloy plate parts. Thus, the shear strength of the brazed interface could be measured with the least effect from the fillet made by the brazing-filler metal around the diamond.

**Fig. 4** Schematic illustrations of unidirectional solidification brazing apparatus and specimen. 1. Thermocouple A, 2. 42 Invar alloy plate, 3. Cooling mass, 4. Thermocouple B, 5. Thermocouple C, 6. Thermocouple D, 7. Electrode, 8. Stage, 9. Wire



After performing the shear strength test, the flat separated metal surface was observed with an electron probe micro analyzer equipped with a scanning electron microscope (SEM/EPMA) in order to investigate the morphology of the solidified structure at the brazed interface.

On the other hand, the brazing-filler which remained on the brazing diamond surface was removed from the interface. The interface was polished, until it became as flat as the diamond surface. The interface was investigated by X-ray diffractometer in order to detect the peaks of the reaction products of vanadium with diamond.

## Results and discussion

### Reaction products for heterogeneous nucleation

A differential thermal analysis (DTA) was performed to investigate the reaction between vanadium and carbon using vanadium hydride powder and graphite powder in argon gas atmosphere. Its temperature elevated slowly at the rate of 10 K per minute. DTA signal was gradually changed at temperature ranged from 720 K to 820 K. A clear DTA signal was obtained at 1120 K. According to the results, brazing was conducted at every temperature of 1060 K exceeding 10 K above the melting point of the Ag–Cu eutectic filler metal, 1090 K, and 1120 K. The brazing time was set to 240 s. The amount of the V additive was small, and its reaction with diamond is considered to be moderate according to the DTA results. As a result, since the brazed surface of the diamond was eroded slightly, a silver translucent interface without scattering the light on the VC islands was obtained.

We performed the NC-AFM observation on the brazed diamond surface prepared by the process described in the section ‘Observation procedure for

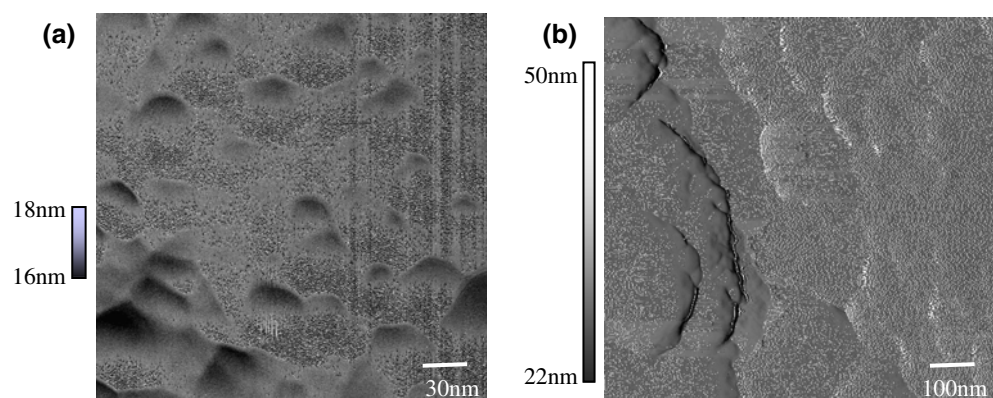
NC-AFM’, from where the brazing-filler metal was clearly removed to where it still remained. Reaction-product islands of about 15 nm in radius, which were detected as VC islands by EPMA, can be observed in Fig. 5a. These small islands are not considered to be effective nucleants for the brazing-filler metal. Larger islands can be observed in the bottom of the figure. Their average height from the diamond surface is about 2 nm based on the measurement of a topographic image by NC-AFM. Continuous VC islands covered with the brazing-filler metal can be observed on the left side in Fig. 5b. Their surfaces exceed 1000 nm in diameter and can facilitate heterogeneous nucleation as effective nucleants. These NC-AFM observation results are consistent with the calculated result of  $r_{Ag}^* = 113.4$  nm since the VC island must exceed the critical radius of silver to act as a nucleant.

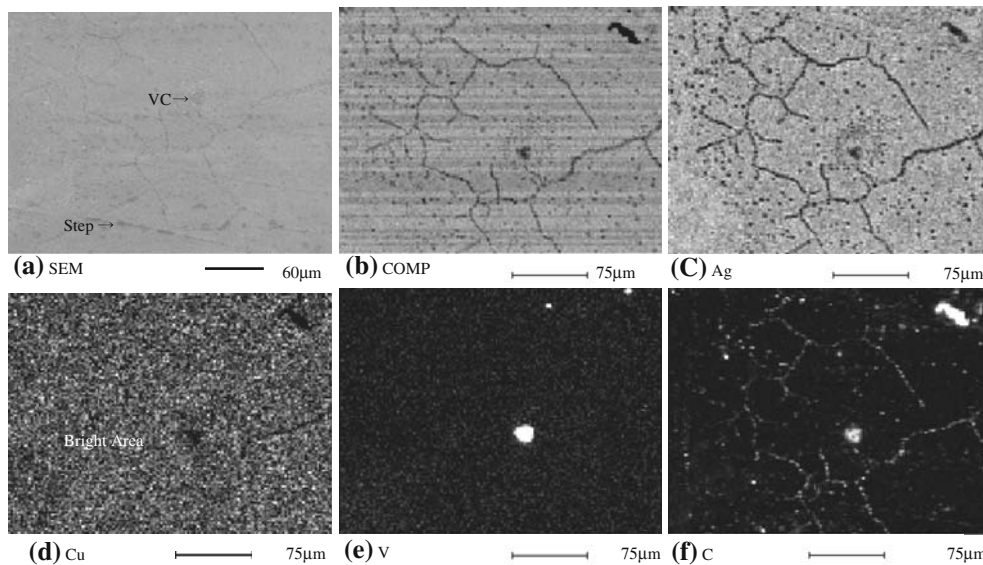
### Solidified structure

We performed SEM/EPMA analysis on the filler-metal surface of the specimen fractured between the diamond and the filler metal with a flat fractured surface by the shear strength test. Figure 6 presents the SEM (a) and composition (b) images, and the maps of silver (c), copper (d), vanadium (e) and carbon (f). The marks of the step edges of the cleaved diamond surface can be observed in Fig. 6a. This brazed interface was considered to be weak because only three VC islands were observed in the V and the C maps as shown in Figs. 6e and f. The VC island shown in the center of every map was created in the center of one Ag grain in the Ag (c) map. This suggests that the  $\alpha$ Ag primary crystal started to grow on the VC island as the nucleant due to a slight degree (0.01298) of lattice mismatch between Ag (111) and VC (111).

The Cu concentration is expected to be higher near the grain boundaries when the  $\alpha$ Ag crystal is large. The slightly bright area surrounding the dark VC island can

**Fig. 5** NC-AFM images of brazed interfaces. **(a)** Grayscale of height ranged from 16 nm to 18 nm. Small islands are reaction product islands. **(b)** Grayscale of height ranged from 22 nm to 50 nm. Brazing-filler metal can be observed on the reaction product islands in the left side of the figure





**Fig. 6** SEM/EPMA analyses on the brazing-filler-side of fractured surface made by shear test. SEM and composition images (a), (b), and maps of silver (c), copper (d), vanadium (e), and carbon (f)

be observed in the Cu (d) map. The slightly dark area surrounding it can also be observed in the Ag (c) map. These results indicate that the copper concentration increases with the  $\alpha$ Ag crystal growth. The network formed by dark lines around the VC island as shown in both composition (b) image and the Ag (c) map cannot be observed in the Cu (d) map, although the bright area along them (indicated by an upward arrow) can be observed. However, the network can be observed in the C (f) map. This differs from the cell structure found in the previous studies for diamond (100) [9].

### Shear strength test

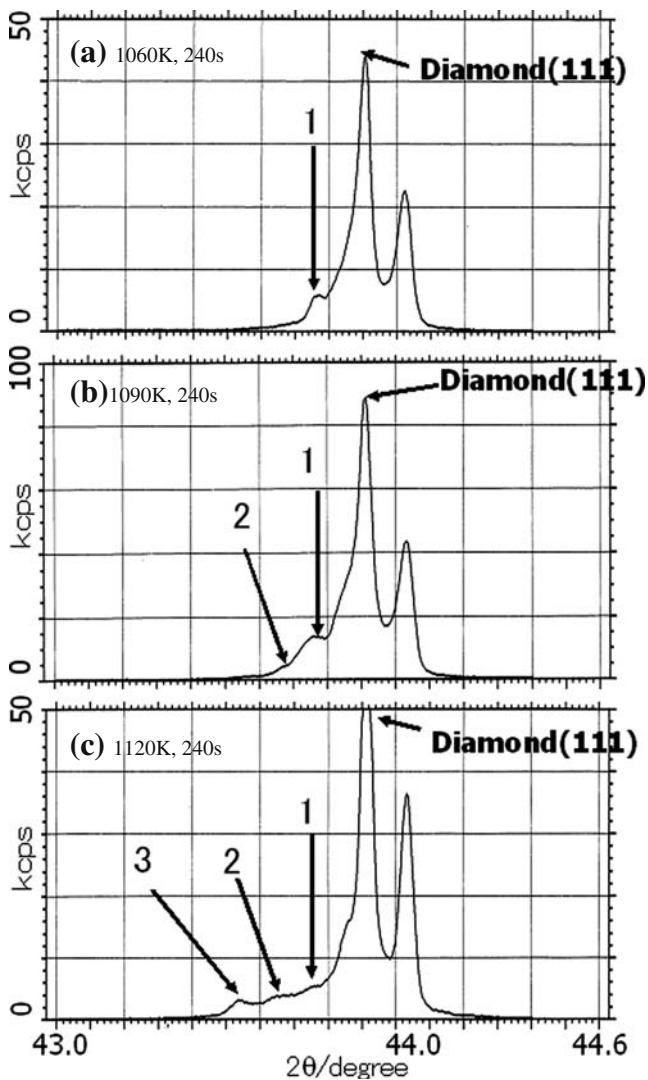
We performed strength tests for four specimens. The number of the specimens is so limited because our first aim is to braze diamond (111) surface with a large area and because it is difficult to produce the strength test specimens because of the thin (0.6 mm thick) diamond.

The shear strength of the brazed interface produced with the brazing-filler metal containing titanium exceeded 120 MPa, but defects were detected in a large percentage in the case of diamond (111) [3]. The shear strengths of three specimens made with the new brazing-filler metal containing vanadium were 205 MPa, 202 MPa and 98 MPa. Their condition of the brazing temperature and time was 1090 K and 240 s. These specimens were not fractured at the brazed interface but in diamonds, so their interfaces are considered to be stronger than their shear strength. On the other hand, the specimen used for the EPMA observation

exhibited low shear strength of 47 MPa. This brazing condition of the temperature and time was 1060 K and 240 s. When brazing was conducted at 1120 K for 240 s, it was difficult to produce a joint. The data of the shear strength tests indicate that the brazed interfaces of diamond (111) with higher strength and lower strength were produced by the vanadium reactions with the diamonds.

Figure 7 shows X-ray diffraction results for the brazed diamonds produced in the conditions of the brazing temperature and the time (a) 1060 K, 240 s, (b) 1090 K, 240 s, and (c) 1120 K, 240 s. The  $2\theta$  angle ranged from 43 to 44.6°, where diamond (111) peak appeared. The diamond peaks are at 43.92 and 44.2°. Changing the brazing temperatures, the diamond peak formations are changed as shown in the figures (a), (b) and (c). One peak of Number 1 can be observed in Fig. 7a (brazed at 1060 K). Two peaks of Number 1 and Number 2 can be observed in Fig. 7b (brazed at 1090 K), and three peaks of Number 1, Number 2 and Number 3 can be observed in Fig. 7(c) (brazed at 1120 K). These peaks are considered to be detected by varied lattice space at the diamond interfaces reacted with vanadium additive.

Figure 8 shows the X-ray diffraction results ranged from 25 to 43.4°. A small peak at 40.8° can be observed in Fig. 8a (brazed at 1060 K). It is due to  $V_2C$  reaction product. Another peak at 42.2° is considered to be Vanadium (200) [16]. Heterogeneous nucleation and crystal growth of silver would not occur, because Ag (111) peak is small. The Number 1 peak is considered to be related to the formation of  $V_2C$  reaction



**Fig. 7** X-ray diffractometer results for brazed diamonds in the brazing conditions of (a) 1060 K, 240 s, (b) 1090 K, 240 s, (c) 1120 K, 240 s. The  $2\theta$  angle ranged from 43 to  $44.6^\circ$

product. In Fig. 8b (brazed at 1090 K), a large Ag (111) peak can be observed. A peak of Ag (222) at  $82.0^\circ$  could be also detected. Silver preferential solidification is considered to occur from vanadium carbide reaction products in the brazing condition of 1090 K, 240 s.  $V_4C_3$  (111) peak is considered to exist near the large peak of Ag (111) shown by an arrow of the  $V_4C_3$  (111), although the peak is slightly sifted from the JCPDS angle of  $37.44^\circ$  [16]. This reaction product is reported to be cubic lattice and NaCl type crystal. Thus, vanadium atoms and carbon atoms binding closely provides bonding strength. As a result, diamond (111)- $V_4C_3$  (111)-Ag (111) bond-bridges are considered to be formed. In Fig. 8c (brazed at 1120 K),  $V_8C_7$  (400) peak and  $V_2C$  (200)

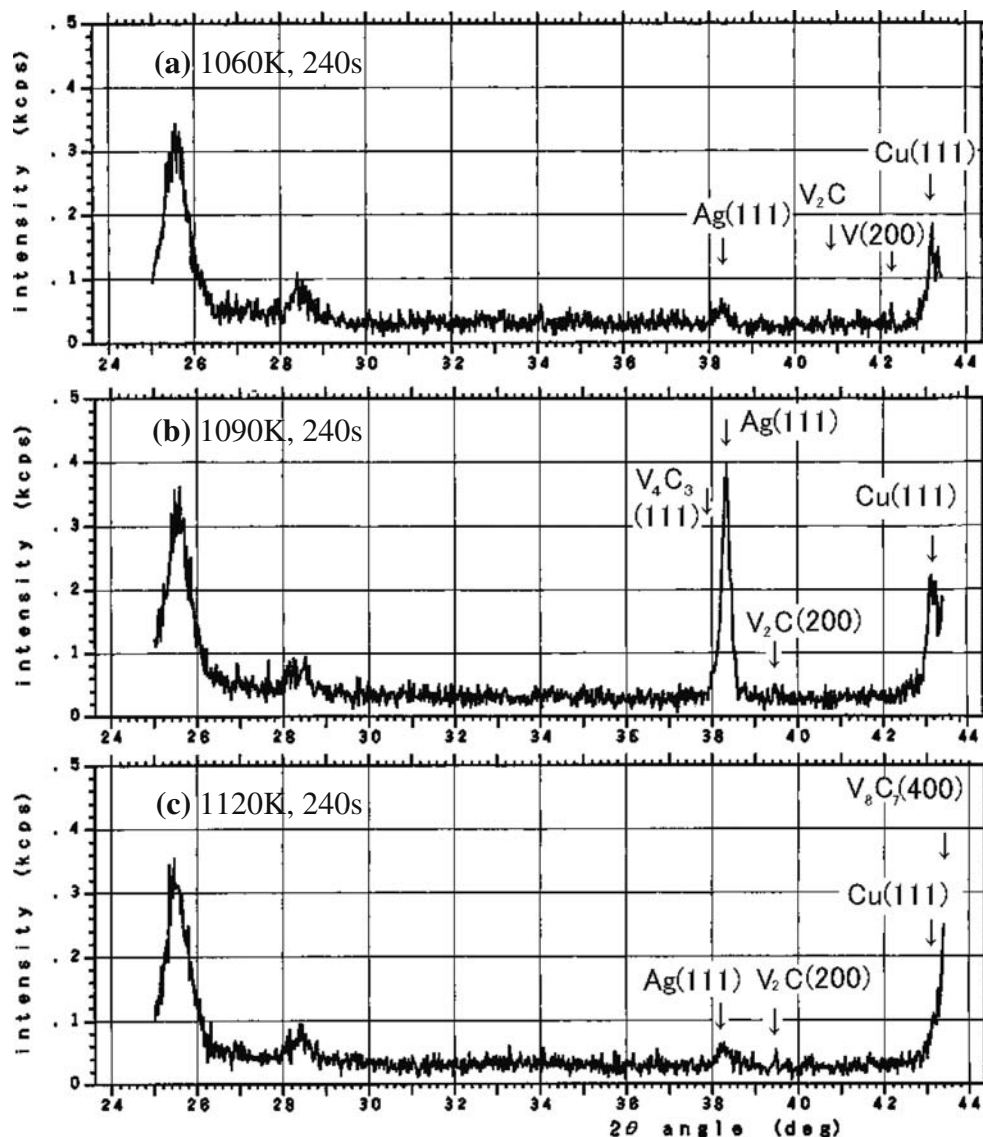
peak are detected. The preferential orientation of Ag (111) cannot be detected but also the Ag peaks become small. Wetting and heterogeneous nucleation of silver from the  $V_8C_7$  islands was considered be difficult to occur. The  $V_8C_7$  reaction product islands formed interface is considered to exhibit the low shear strength.

## Conclusions

The composition of the brazing-filler metal in Ag–Cu–V system for joining the diamond (111) surface was determined theoretically and attempted to join artificial single-crystal diamonds to metal plates by the unidirectional-solidification brazing method. Titanium was replaced by vanadium as the reactive metal additive for diamond (111) taking into account the slight degree of misfit between vanadium carbide and diamond.

The perturbation wavelength,  $\lambda$ , and the critical radius of the Ag nucleus,  $r^*_{Ag}$ , are important parameters for calculating the amount of the additive and the Cu concentration in the brazing-filler metal. The condition is  $\lambda \geq 4r^*_{Ag}$  at the brazed interface of the diamond. The ideal coverage ratio of the reaction product islands on the brazed diamond surface was calculated to be 22.5%, where the amount of vanadium and Cu concentration is also calculated to be 0.0005 mass% and 39.7 at%, which is 0.2 at% less than the eutectic composition. The theoretically determined composition was Ag-27.9Cu-0.0005V (mass%).

Using Ag-27.8Cu-containing less than 1mass%V brazing-filler metal, the artificial diamond could be joined to the 42 Invar alloy plate by brazing at 1090 K for 240 s. The shear strength at the joined interface exceeded 200 MPa, where the silver crystal grains grew from the vanadium carbide islands of  $V_4C_3$  formed on the brazed diamond surface, conforming with the theory of the lattice mismatch between silver and vanadium carbide. In the cases of the other brazing conditions of 1060 K and 1120 K, diamond-metal joints with high shear strength could not be produced, whereas  $V_2C$  or  $V_8C_7$  reaction products were formed. X-ray diffraction analysis results revealed heterogeneous nucleation and crystal growth of silver in brazing filler would not occur on these reaction products in spite of adding silver powder to the brazing-filler metal. The new brazing-filler metal containing vanadium provided higher bonding strength than that containing titanium, when the reaction product islands of  $V_4C_3$  were formed on the diamond surfaces.



**Fig. 8** X-ray diffractometer analysis results for brazed diamonds in the brazing conditions of (a) 1060 K, 240 s, (b) 1090 K, 240 s, (c) 1120 K, 240 s. The  $2\theta$  angle ranged from 25 to  $43.4^\circ$

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