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### Formation of TiC via Interface Reaction between Diamond Grits and Sn-Ti Alloys at

### **Relatively Low Temperatures**

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#### Abstract

In this paper, interfacial reaction between diamond grit and Sn-6Ti alloy was systematically studied at brazing temperatures from 600 to 1030 °C. A thin and uniform layer of scallop-like nano-sized TiC grains was formed after brazing for 30 minutes at 600 °C, and interfacial TiC grains subsequently coarsened as brazing temperature increased to 740 and 880 °C. Strip-like columnar TiC grains in a bilayer structure was further grown as brazing temperature increased to 930 °C. After brazing at 1030 °C, a dense layer of columnar TiC grains were formed. Based on the TEM micrographs of interfacial TiC, the formation and evolution of the growth morphologies of interfacial TiC was believed to be controlled by the diffusion of C flux from diamond grits, which is dependent on the brazing temperatures.

**Key words**: Brazing, Diamond grits, Growth Morphology, Microstructure, Transmission electron Microscopy (TEM)

#### 1. Introduction

Brazing is a promising method for the manufacturing of diamond abrasive tools [1, 2], which are widely used for the machining of hard and/or brittle materials such as stones, ceramics and non-ferrous alloys [3]. Moreover, diamond brazing has been used for the fabrication of modern diamond devices for medical applications [4]. Conventionally, brazing of diamond is mainly implemented by reacting diamond grits with filler alloys at relatively high temperatures (approximately 930-1100 °C, depending on the filler alloys used) for ten or a few tens of minutes [5]. During brazing, diamond girts react with the active elements such as Ti, Cr and V in the filler alloys, which forms a layer of interfacial carbide [5].

Because established brazing approach involves the use of high brazing temperatures, the extensive formation of interfacial carbide inevitably results in undesirable thermal damage of brazed diamond grits [6, 7]. Therefore, the Cu-Sn-Ti and Ag-Cu-In-Ti alloys with lower melting points were attempted to replace Ni-Cr alloys, in order to reduce the thermal damage induced during brazing process [8]. When brazing diamond grits using these Ti-containing filler alloys, TiC is the carbide formed during interface reaction. The knowledge on the growth behaviors of TiC is essential for the understanding of failure mechanism of brazed diamond grits, and hence the optimization of brazing process. However, existing research focus more on the characterization of the interface products formed between diamond grits and commercial Ti-containing filler alloys at a certain brazing temperature [9-13], or the technology development of brazing process [14, 15]. To the best knowledge of authors, there is little information available regarding the growth behavior of interfacial TiC at variable brazing temperatures, especially at relatively low brazing temperatures [16].

This work thus aimed to understand the effect of brazing temperatures on the formation and growth behaviors of TiC formed during brazing of diamond grits at temperatures ranged from 600 to 1030 °C. In particular, a binary Sn-Ti filler alloys was employed to achieve the interface reaction between liquid Ti and

solid diamond grits at a brazing temperature as low as 600 °C, with minimal effects of alloying elements reported in the previous works using commercial filler alloys [9, 10].

#### 2. Experimental procedures

Sn-6Ti (in wt% unless mentioned elsewhere) ingots were prepared by melting commercial purity Sn and Sn-20Ti master alloy in a non-consumable arc furnace in high-purity Ar atmosphere. Chemical composition of the casted alloy was determined to be Sn-5.9Ti by inductively coupled plasma mass spectrometry (ICP-MS) analysis. The casted ingots were mechanically cut and rolled into foils of 3 mm in length and width and 1 mm in thickness. The foil specimens were then polished and ultrasonically cleaned in acetone bath after polishing. Some of the specimens were prepared using traditional polishing techniques for metallographic observation. To prepare brazed diamond samples, diamond grits (ISD-1650, ILJIN Co. Ltd, Korean) with a size ranged from 425 to 500 µm were used. As shown in Fig. 1, a diamond grit ultrasonically cleaned in acetone bath was placed on the a prepared Sn-6Ti alloy foil, under which a tungsten carbide (WC) substrate with a polycrystalline diamond (PCD) coating was used as the sample holder to minimize any effects of dissolved metal substrates. The assembled samples were placed in a quartz tube with flowing high-purity Ar and brazed by a resistance tube furnace for 30 minutes at 600, 740, 880, 930 and 1030 °C, respectively. Prior to brazing, a T-type thermocouple was used to measure and calibrate the brazing temperature with an accuracy of  $\pm 10$  °C. After brazing, the quartz tube was automatically withdrawn from the tube furnace and cooled in air. Brazed diamond girts were then etched in a 10%HNO<sub>3</sub>+1%HCl+ethyl alcohol solution to remove remaining Sn-Ti alloy. A Hitachi S4800 field emission scanning electron microscope (SEM), a Hirox KH8700 3D digital microscope, a confocal Raman microscope (inVia, Renishaw, Gloucestershire, UK), and a FEI TECNAI F20 field emission transmission electron microscope (TEM) with an energy dispersive spectrometer (EDS) were used for microstructure observation and phase analysis. TEM samples were prepared using a FEI Helios Nanolab-600i dual beam focused iron beam (FIB)

milling machine.

#### 3. Results and discussion

#### 3.1 Growth Morphologies and microstructure of interfacial TiC

Fig. 2 shows the images of the microstructure of Sn-6Ti alloy and the brazed joint of a diamond grit and Sn-6Ti alloy. As shown in Fig.2 (a), the solidified Sn-6Ti alloy has a composite microstructure with primary intermetallics (dark grey phase) homogeneously distributed in Sn matrix and no evidence of segregation of Ti that sometimes occurs during casting was observed. The ICP-MS analysis revealed that the casted alloy had a composition of Sn-5.9%Ti, corresponding to a solidus of 231.9°C and a liquidus of around 580°C under equilibrium [17]. Fig. 2(b) shows the optical image of a diamond grit brazed at 600 °C. It can be seen that no contamination of diamond grits can be observed and Sn-6Ti alloy has been slightly lifted towards to the surface of diamond grits, indicating that the Sn-6Ti alloy did wet the diamond grits during brazing at 600 °C.

Fig. 3 shows the surface morphologies of interfacial TiC formed on diamond grits at brazing temperatures ranged from 600 to 1030 °C (note that the interfaces were examined after the Sn-6Ti alloy was removed and the surface morphology of interfacial TiC formed on PCD coating on WC substrate can be found in supplementary information). After brazed at 600 °C for 30 minutes, a continuous layer of scallop-like TiC grains of ~20 nm in diameter formed on diamond grits, as shown in Fig. 3(a). When brazing temperature increased to 740 °C, faceted scallop-like grains of ~50 nm in diameter formed among the existing nano-sized TiC grains, as shown in Fig. 3(b). When the temperature increased to 880 °C, the faceted scallop-like TiC grains coarsened into a strip-like morphology, as can be seen in Fig. 3(c). When temperature increased to 930 and 1030 °C, strip-like TiC grains further grew to a length of ~1  $\mu$ m, with nano-sized particles precipitated on the surface of strip-like TiC grains, as shown in Figs. 3(d) and 3(e).

Fig. 4 provides the TEM bright field micrographs of cross-sectional morphology of interfacial TiC after brazing for 30 minutes at 600, 740, 880, 930 and 1030 °C. In Fig. 4(a), a continuous layer of scallop-like TiC grains can be clearly observed on the surface of diamond without coarsening or faceting of TiC grains. In Fig. 4(b), it can be found that the TiC grains grew to a thickness of ~50 nm. Moreover, the continuous layer of TiC grains became irregular, which indicates the TiC grains started to coarsen after brazing at 740 °C, as observed from the top-view image in Fig. 3(c). In Fig. 4(c), TiC grains further coarsened to a size of ~100 nm after brazing at 880 °C. When the brazing temperature raised to 930 °C, the interface TiC layer displayed a bilayer structure after brazing: a layer of relative dense TiC grains of less than 100 nm in diameter formed at the diamond side (Fig. 4d), and the other layer of strip-like TiC grains irregularly grew towards to filler alloy matrix. As temperature was further increased to 1030 °C, as shown in Fig.4 (e), TiC grains grew into a dense and continuous layer, showing a columnar morphology. We can conclude from Fig.4 and 5 that the brazing temperature has remarkable effects on the growth morphologies of interfacial TiC grains: as brazing temperatures increased from 600 to 1030 °C, the interfacial TiC grains gradually changed from a nano-sized scallop-like morphology to a dense columnar strip-like morphology.

#### 3.2 Formation and evolution of TiC brazed at increasing temperatures

Thermodynamically, the reaction, i.e.  $Ti+C\rightarrow TiC$ , is spontaneous at 0 °C [18]. Therefore, if liquid Ti can wet solid diamond, the interface formation of TiC during diamond brazing should be viable at a temperature significantly lower than that used in the conventional brazing route. However, research on the interface formation of TiC was limited in published literature due to the high melting points of existing brazing filler alloys [5, 12]. In this study, the interface reaction between liquid Ti and diamond grits was achieved at a brazing temperature as low as 600 °C, by using a binary Sn-Ti filler alloy. Fig. 5 shows the high resolution TEM image and TEM EDS spectrum of the interfacial phase formed during brazing reaction at 600°C. As

shown by the TEM EDS spectrum in Fig. 5(a), although Mo signal from sample holder was detected during TEM EDS point analysis, the interfacial nano-sized grains mainly consisted of C and T, which should be TiC, as suggested by Ti-C phase diagram [19]. According to the high resolution TEM image given in Fig. 5(b), the inter-plane distance of the interfacial phase formed at 600 °C was measured as 0.322 nm, corresponding to the (110) plane of TiC [20]. Thise evidenced that, the interface formation of TiC phase can be initiated at 600 °C. Moreover, as shown in Fig.5 (b), no textured growth orientation of TiC grains can be observed from the high resolution TEM image, which confirms a random nucleation of TiC grains at the early stage of interface reaction. The formation of randomly nucleated TiC grains is in contrast to the previously reported epitaxial growth of interfacial TiC formed at 930 °C [13], highly likely due to the different reaction conditions resulted in the different grains sizes and morphologies of interfacial TiC [13]. The newly formed TiC grains of ~12 nm in diameter between the existing TiC grains further indicated the early stage nucleation of interfacial TiC was completed after brazing at 600 °C for 30 minutes. Based on above analysis and the Ti-C phase diagram [19], it could be inferred that a layer of nano-sized TiC grains was formed at the brazing temperature of 600°C and the early stage reaction was completed after reaction for 30 minutes.

The evolution of interfacial TiC morphologies can be correlated to the progress of interface reaction, and the evolution of TiC morphologies with the increased brazing temperature are thus schematically illustrated in Fig. 6. As discussed earlier, Sn did not react with C in the range of temperatures used in this study [16]. The previous studies also indicated Ti was preferentially concentrated at the reaction interface and the Ti content of 5.9 wt% was sufficient to drive the interface formation of TiC during brazing reaction [21]. Therefore, the factor controlling the formation and growth morphologies of interfacial TiC should be the flux of C atoms dissolved from diamond grits. According to the general sequence of solid/liquid interface reaction [22], the prerequisite for the initiation of interface reaction between diamond and liquid Ti is that some C atoms in

unit cell of diamond could be dissolved in liquid Ti to establish a supersaturated zone (Fig. 6(a)). As the C concentration continuously increased, the supersaturated C and Ti atoms randomly nucleated as TiC grains on the diamond surface (Fig. 6(b)). After the nucleation of TiC, C atoms diffused preferentially through the grain boundaries between nano-sized TiC grains, reacting with Ti and continuously driving the growth of TiC. Since C is the diffusing species in TiC in the range of temperatures used in this study and the diffusion of C is dependent on temperature [23, 24], the interfacial growth of TiC was sluggish at relatively low temperature, i.e. 600 °C in this study. As a result, only a layer of TiC grains of ~20 nm in diameter was formed at 600 °C. As temperature increased to 740 °C, the diffusion of C flux became faster, promoting the growth of TiC grains and resulting in the formation of faceted scallop-like TiC grains. When temperature was increased to 880 and 930 °C, the TiC grains previously formed were further ripened and thus the number of grain boundaries was reduced (Fig. 6(c)), through which enhanced C flux at elevated temperature diffused to react with Ti atoms in the liquid filler alloy. The enhanced diffusion of C flux through a reduced number of grain boundaries consequently resulted in the formation of TiC grains vertically elongated to the filler alloys matrix (Fig. 6(d)). As a result, the interfacial TiC had a bilayer structure with dense nano-sized TiC grains formed at diamond side and strip-like TiC grains irregularly formed at filler alloy side. Consequently, interfacial Ti of a columnar morphology was observed in the TEM micrograph in Fig. 5(d), which is consistent with previous study [9]. As temperature increased to 1030 °C, the dissolution of diamond became even faster and dissolved C flux diffused into filler alloys to form a dense layer of Columnar TiC grains (see Fig. 6(e)).

Fig. 7 shows the microstructure and the TEM spot elemental analysis of C and Ti using sample brazed at 1030°C. In Fig. 7(a), an uneven interface between diamond and TiC grains was observed for the sample brazed at 1030 °C, which indicates that diamond grits were dissolved at a higher rate at 1030 °C. In Fig. 7(b), a clear gradient of C element can be identified as the TiC grains near to the diamond surface has a higher C

content (61.68 at%, averaged from points 1-4) than that at the Sn-6Ti alloy side (52.73 at%, averaged from points 5-7), evidencing the existence of a C flux from diamond grits to filler alloy. It should be noted that the C content measured in this study was significantly higher than the maximum content of C in TiC (48.8 at%) in C-Ti phase diagram, suggesting that the TiC grains formed during brazing reaction was a metastable phase with surplus C atoms in TiC unit cells. Isothermal aging experiment was undertaken to investigate the effect of the solid state phase transformation of interface TiC on the formation of nano-sized unidentified phase observed in Figs. 3(e) and 3(f). Nevertheless, the present study clearly demonstrated that reaction temperature strongly affects the growth morphology of TiC formed during interface reaction between liquid Ti and solid diamond, through enhancing the diffusion of C flux.

#### 3.3 Implications for integrity of brazed diamond grits

To create a reliable metallurgical bond with minimal thermal damage of diamond grits, it is desirable to ensure the formation of a continuous and uniform layer of TiC, but avoid the extensive formation of TiC. In order to further confirm the phase formation after brazing at variable temperatures, Raman spectrum measured on brazed diamond surface after removing Sn-Ti filler alloy was given in Fig.8. As shown in Fig.8, TiC peaks at 250, 345, 430 and 609 cm<sup>-1</sup> can be clearly observed, which is in a reasonable agreement with previous study using stoichiometric samples [26]. The small shift of TiC peaks is most likely due to varying C contents of TiC in samples brazed at different temperatures, because the Raman peaks is obtained from carbon vacancies in TiC [27]. Moreover, both diamond peak at 1331 cm<sup>-1</sup> and graphite peak at 1585 cm<sup>-1</sup> became stronger, suggesting more C atoms have precipitated as graphite. Previous research on the shear strength of brazed diamond grits decreased from (321±107) to (78±30) MPa [25]. Buhl et al.

have attributed this abrupt drop of shear strength to the linear increase of thermal stress against the increased brazing temperature [25]. From interfacial reactions prospective, the enhanced dissolution of C atoms and graphitization should also be responsible for the decrease of bonding strength of brazed diamond grits Experimental results presented in this study clearly demonstrated that reducing brazing temperature is a feasible and controllable approach to achieve preferred interface morphology of TiC during the brazing of diamond grits.

#### 4. Conclusions

In this work, the growth morphologies of TiC formed during interface reaction between diamond grits and Sn-6Ti alloys were studied at variable brazing temperatures. After brazing for 30 minutes, a continuous layer of TiC grains was formed at 600 °C, which is significantly lower than that used in the conventional brazing process. Brazing temperature had remarkable effects on the growth morphology of TiC grains by affecting the diffusion of C flux during brazing, which might affect the bonding strength and thermal damage level of brazed diamond grits. This study demonstrated that the design of a new filler alloy with a lower melting point enabled the reduction of brazing temperature, without losing the interfacial metallurgical bonds. This should be an effective approach to reduce thermal damage on brazed diamond grits, which is of great importance for the development of high quality diamond tooling and modern diamond devices.

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- Figure 8: Raman spectrum of interfacial TiC brazed at variable temperatures.



Fig. 1

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![](_page_19_Figure_1.jpeg)

Fig. 8

#### Highlights

- a) A thin and continuous layer of scallop-like nano-sized TiC grains was formed after brazing at 600 °C, which is significantly lower than that of 880 °C used in the conventional brazing process. The early stage nucleation of interfacial TiC grains was found to be completed after reaction for 30 minutes.
- b) As brazing temperature increasing from 600 to 930 °C, the morphologies of interfacial TiC grains consequently coarsened, and gradually changed from a nano-sized grains with a scallop-like morphology to a strip-like columnar TiC grains in a bilayer structure. After brazing at 1030 °C, a dense layer of columnar TiC grains were formed.
- c) The growth morphologies of interface TiC was believed to be controlled by diffusion of C flux from diamond grits, which is dependent on the brazing temperatures.

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