

## Effect of heat treatment on microstructure and thermophysical properties of diamond/2024 Al composites

Zi-yang XIU<sup>1</sup>, Xu WANG<sup>2</sup>, M. HUSSAIN<sup>3</sup>, Chao FENG<sup>1</sup>, Long-tao JIANG<sup>1</sup>

1. State Key Laboratory of Advanced Welding and Joining, Harbin Institute of Technology, Harbin 150001, China;
2. School of Mechanical Engineering, Liaoning Shihua University, Fushun 113001, China;
3. Department of Chemical Engineering, COMSATS Institute of Information Technology, Lahore 54000, Pakistan

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**Abstract:** 50% diamond particle (5  $\mu\text{m}$ ) reinforced 2024 aluminum matrix (diamond/2024 Al) composites were prepared by pressure infiltration method. Diamond particles were distributed uniformly without any particle clustering, and no apparent porosities or significant casting defects were observed in the composites. The diamond–Al interfaces of as-cast and annealed diamond/2024 Al composites were clean, smooth and free from interfacial reaction product. However, a large number of  $\text{Al}_2\text{Cu}$  precipitates were found at diamond–Al interface after aging treatment. Moreover, needle-shaped  $\text{Al}_2\text{MgCu}$  precipitates in Al matrix were observed after aging treatment. The coefficient of thermal expansion (CTE) of diamond/2024 Al composites was about  $8.5 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$  between 20 and 100  $^\circ\text{C}$ , which was compatible with that with chip materials. Annealing treatment showed little effect on thermal expansion behavior, and aging treatment could further decrease the CTE of the composites. The thermal conductivity of obtained diamond/2024 Al composites was about 100  $\text{W}/(\text{m}\cdot\text{K})$ , and it was slightly increased after annealing while decreased after aging treatment.

**Key words:** Al matrix composites; diamond; interface; annealing; aging; thermal properties

### 1 Introduction

Ever-growing requirements imposed on thermal management materials in semiconductor applications drive the development of new materials with enhanced thermal conductivity [1,2]. High-performance thermal management materials should have high thermal conductivity (TC) [3,4] to maximize heat dissipation and low coefficient of thermal expansion (CTE) [5,6] to minimize the thermal stress and warping, which are critical issues in packaging of electronic devices [7]. However, the traditional packaging materials such as W/Cu and Mo/Cu could not meet these requirements [8,9]. Therefore, a large effort has been made to develop new materials with improved thermal properties.

Metal-matrix composites (MMCs) with tailored properties have been widely investigated recently. Aluminum matrix composites reinforced with high fraction of SiC [10–12] or  $\text{Si}_3\text{N}_4$  particles [13,14] demonstrated compatible CTE with chip materials.

However, their heat conduction and heat spreading capacity could not meet well with the increasing requirement [15]. Diamond particles have been identified as a promising reinforcement due to their exceptional high TC and low CTE [16], typically 600–2000  $\text{W}/(\text{m}\cdot\text{K})$  and  $0.8 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$  at 25  $^\circ\text{C}$ , respectively.

Pressureless metal infiltration [17,18], spark plasma sintering [7,19,20], powder metallurgy method [21], vacuum hot pressing [22], gas pressure infiltration [23–25] and pressure infiltration method (squeeze casting) [26,27] have been reported to fabricate diamond/Al composites. JOHNSON and SONUPARLAK [17] used pressureless metal infiltration to obtain diamond/Al composites. The diamond particulates were coated with SiC prior to infiltration to prevent the formation of  $\text{Al}_4\text{C}_3$ . Moreover, TC of diamond/Al composites prepared by spark plasma sintering reached 403  $\text{W}/(\text{m}\cdot\text{K})$  [7,19,20]. HANADA et al [21] reported the processing of diamond/Al with mean particle size of 5 nm by powder metallurgy method. The addition of 1% (volume fraction) cluster diamond improved the friction coefficient of

Al matrix significantly. Furthermore, gas pressure infiltration was also adopted to prepare diamond/Al composites. However, the long exposure time of diamond to molten aluminum led to excessive formation of  $Al_4C_3$ , which was observed to be formed with a strong crystallographic preference on the {100} faces of diamond [24]. Therefore, Ti [28] or TiN [29] coatings were used to prevent the formation of  $Al_4C_3$ . Pressure infiltration method is believed to be an effective technique because of the advantages of higher production rate, elimination of expensive equipments, feasibility of mass production and near-net products [30,31]. BEFFORT et al [26] investigated the thermal and chemical stability of micron-grade diamond powders as well as the interface microstructure in diamond/Al composites produced by pressure infiltration method. It was found that diamond powders were particularly susceptible to thermal degradation in the presence of oxygen during composite processing, which leads to the formation of an amorphous layer consisting of carbon, aluminum and oxygen.

Our research group has successfully fabricated carbon fibre [32,33] and ceramic particles [13,14] reinforced Al composites by pressure infiltration method under protective atmosphere. Moreover, it is well known that the heat treatment has significant effects on microstructure and properties of metal and metal matrix composites [34,35]. However, the effect of heat treatment on microstructure and properties of diamond/Al composites has rarely been investigated. Therefore, in the present work, 50% (volume fraction) diamond particle (5  $\mu\text{m}$ ) reinforced 2024 aluminum matrix composites were prepared by pressure infiltration method. The effect of annealing and aging on microstructure and thermo-physical properties was also discussed.

## 2 Experimental

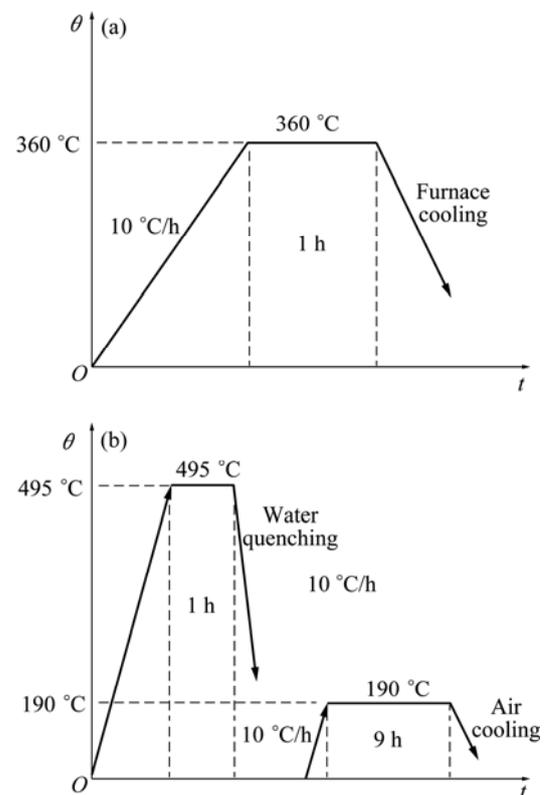
The synthetic diamond particles (SF Diamond Co., Ltd., China) with an average particle size of 5  $\mu\text{m}$  were used to reinforce 2024 Al alloy by pressure infiltration method. The basic properties of diamond particles and 2024 Al alloy are shown in Table 1. The chemical composition (mass fraction, %) of the 2024 Al alloy was 4.79% Cu, 1.49% Mg, 0.611% Mn, 0.245% Fe, 0.168%

**Table 1** Properties of diamond particles and 2024 Al alloy

Material	Density/ ( $\text{g}\cdot\text{cm}^{-3}$ )	Elastic modulus/ GPa	CTE/ ( $10^{-6}\text{ }^\circ\text{C}^{-1}$ )	TC/ ( $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ )
Diamond particles	3.52	435	1.7	900–1100
2024 Al alloy	2.82	68	21.1–24.7	134–192

Si, 0.068% Zn, 0.049% Cr, 0.046% Ti, 0.013% Ni and Al balance. A preform comprising diamond particles was prepared firstly. Preheating temperatures for the preform and pressure infiltration dies were 500 and 740–750  $^\circ\text{C}$ , respectively. The matrix alloy was melted under protection of argon and melted salts, and poured into dies at 740–750  $^\circ\text{C}$  under argon.

The schematic diagrams of annealing and aging treatment are shown in Fig. 1. The annealing samples were held at 360  $^\circ\text{C}$  for 1 h and then cooled in the furnace to room temperature. The aging samples were solution treated at 495  $^\circ\text{C}$  in  $\text{KNO}_3$  salt-bath furnace for 1 h and water quenched at room temperature. After solution treatment, specimens were aged at 190  $^\circ\text{C}$  for 8 h and cooled in the air. The as-cast samples were used for comparison.



**Fig. 1** Schematic diagrams of annealing (a) and aging (b) treatment

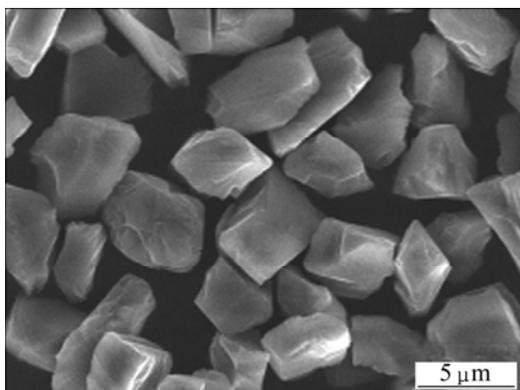
The density of diamond/2024 Al composites was measured by Archimedes' method. The morphology of diamond/2024 Al composites was observed by ZEISS 40MAT optical microscope (OM) and FEI Sirion Quanta 200 scanning electron microscope (SEM). Further microstructure observation was conducted by a Philips CM-12 transmission electron microscope (TEM) with an accelerated voltage of 100–120 kV. Thermal conductivity (TC) and specific heat capacity of the composite and matrix, with a diameter of 12.7 mm and a thickness of 2 mm, were examined on LFA 427 laser

flash apparatus (Netzsch, Germany) from 20 to 50 °C with a heating rate of 5 °C/min. CTE tests were carried out on Dilatometer 402C (Netzsch, Germany). The samples were the cylinders with a diameter of 4 mm and a length of 25 mm. During the CTE measurement, the temperature was increased from 20 to 495 °C at a heating rate of 5 °C/min. The helium atmosphere was maintained at a flow rate of 50 mL/min to ensure the equilibrium of temperature and prevent oxidation of samples.

### 3 Results and discussion

#### 3.1 Microstructure

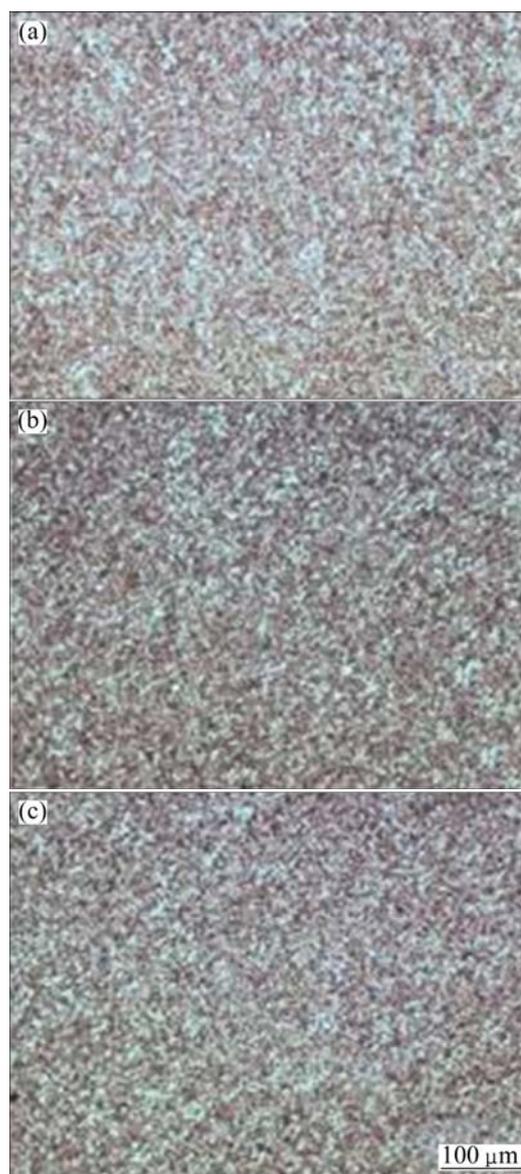
The irregular shape diamond particles were used in the present work, as shown in Fig. 2. The representative optical morphologies of the diamond/2024 Al composites in as-cast, annealed and aged condition are shown in Fig. 3. Diamond particles were distributed uniformly without any particle clustering, and no apparent porosities or significant casting defects were observed in the composites. The dense microstructure is favorable for the mechanical and thermal conductivity properties. Moreover, the dense microstructure is also beneficial to improving the dimensional stability of composite, which would prolong the service life of device.



**Fig. 2** SEM image of diamond particles used in the present work

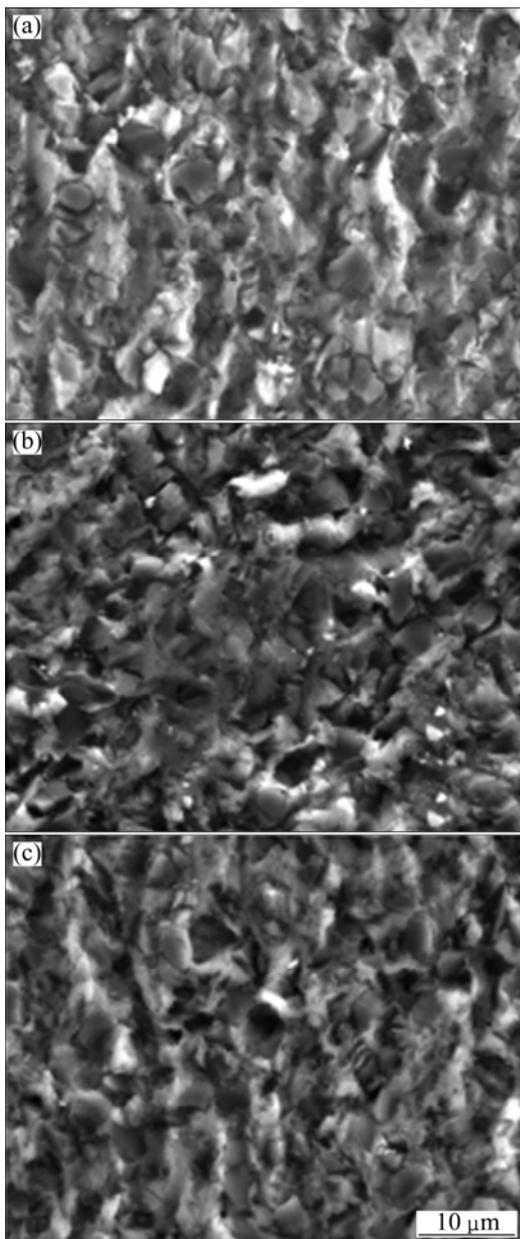
Representative SEM images of diamond/2024 Al composites in as-cast, annealed and aged condition are shown in Fig. 4. It is obvious that some diamond particles were polished away due to the large hardness difference between reinforcement and matrix.

TEM images of diamond–Al interface of as-cast, annealed and aged diamond/2024 Al composites are shown in Fig. 5. As reported by BEFFORT et al [24], amorphous interfacial C-layer and excessive  $Al_4C_3$  phases were observed in as-cast diamond/Al composites fabricated by pressure infiltration method. However, in the present work, the diamond–Al interface of as-cast



**Fig. 3** Optical morphologies of diamond/2024 Al composites in as-cast (a), annealed (b) and aged (c) condition

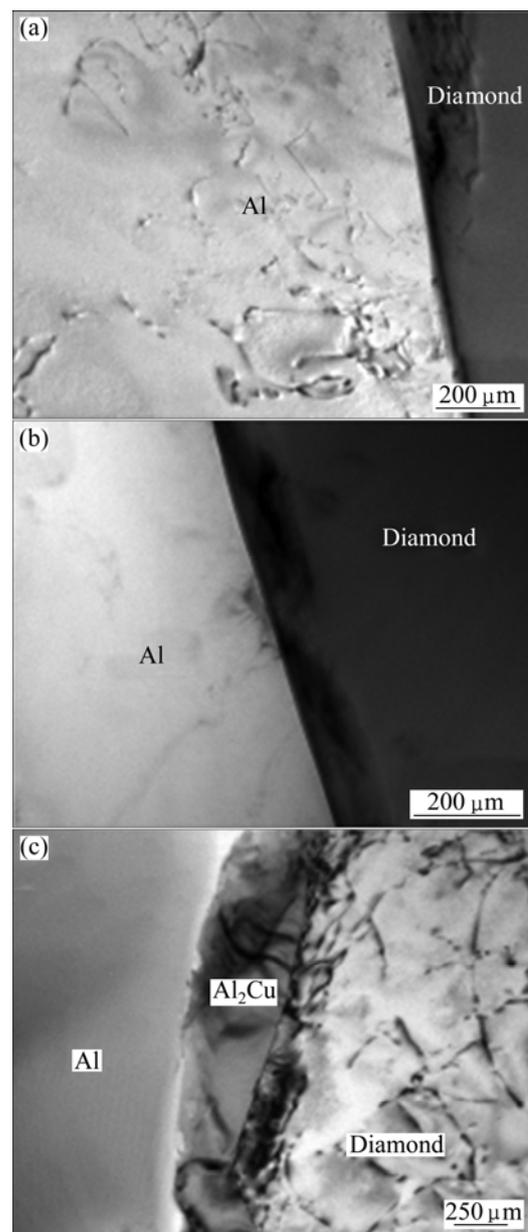
diamond/2024 Al composites was clean, smooth and free from interfacial reaction product (Fig. 5(a)). Diamond particles would undergo thermal degradation [24]. This effect is more obvious in air atmosphere [27]. Therefore, amorphous carbon and isolated Al carbides were observed in the obtained composites. However, in the present work, the diamond preform was protected by  $CO_2$ , which would inhibit its degradation. Therefore, the diamond–Al interface is clean and without amorphous layer and  $Al_4C_3$  phases in as-cast composite. No significant change was observed at diamond–Al interface in annealed composite (Fig. 5(b)). However, a large number of  $Al_2Cu$  precipitates were found at diamond–Al interface after aging treatment (Fig. 5(c)). Moreover, it was hard to observe  $Al_2Cu$  phase in Al matrix.



**Fig. 4** SEM images of diamond/2024 Al composites in as-cast (a), annealed (b) and aged (c) conditions

The diamond–Al interface provides extra energy for heterogeneous nucleation of  $\text{Al}_2\text{Cu}$ , leading to the low nucleation energy and a relatively high nucleation rate at the interface compared with that in Al matrix. Therefore,  $\text{Al}_2\text{Cu}$  phase would grow predominantly at the interface instead of in Al matrix during aging treatment.

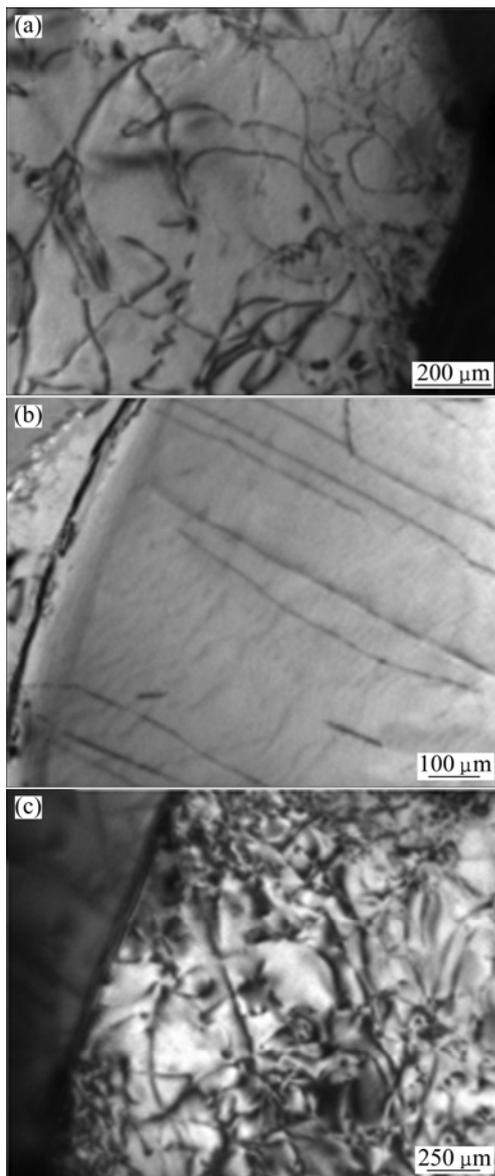
The dislocations in Al matrix of as-cast, annealed and aged diamond/2024 Al composites are shown in Fig. 6. Large CTE difference between diamond particles and Al matrix would generate enormous thermal tensile stresses on Al matrix during rapid cooling after Al infiltration, which leads to the formation of high density dislocations near the diamond–Al interface (Fig. 6(a)). The annealing treatment decreased the density of the



**Fig. 5** TEM images of diamond–Al interface of as-cast (a), annealed (b) and aged (c) diamond/2024 Al composites

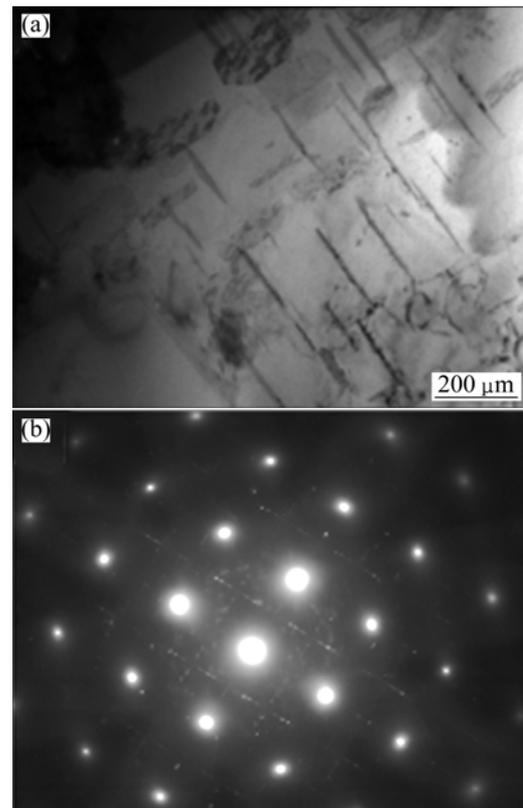
dislocations significantly due to release of residual thermal stress (Fig. 6(b)). However, more dislocations were found after aging treatment. Water quenching process in aging treatment generates larger thermal stress at diamond–Al interface, which leads to the reformation of dislocations. Moreover, apparent needle-shaped precipitates were found in Al matrix after aging treatment (Fig. 7(a)), and the weak diffraction spots in electron diffraction pattern (Fig. 7(b)) were elongated lines, which corresponds to  $\text{Al}_2\text{MgCu}$ .

Crystal defects in particle reinforcements, which have important influence on interface and properties, are significant characteristics of particle reinforced metal matrix composite. Stacking faults and dislocations have



**Fig. 6** TEM images of dislocations in Al matrix of as-cast (a), annealed (b) and aged diamond/2024 Al composites (c)

been observed in ceramic particles reinforced Al matrix composites [36,37], the high density dislocations in AlN particles even tangle with each other [38]. In the present work, few aligned linear dislocations (Fig. 8) were found in diamond particles. Generally, the crystal defects in reinforcements are generated in preparation process of particles as well as composites. The diamond particles were fabricated under high temperature and pressure, which may produce some crystal defects. Moreover, the volume fraction of particles was very high (50%), parts of particles contacted with each other during perform densification or Al infiltration under pressure. Since the morphology of the diamond particles was irregular with sharp edges (Fig. 2), some sharp edges would exert pressure to the contacted surface of other particles



**Fig. 7** Al<sub>2</sub>MgCu precipitates in Al matrix of diamond/2024 Al composites after aging treatment: (a) TEM image; (b) Electron diffraction pattern



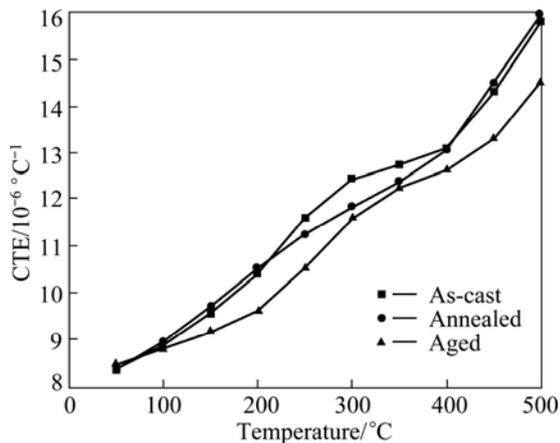
**Fig. 8** TEM image of few linear dislocations in diamond particles

inevitably, which would generate dislocations. Furthermore, a large CTE difference between diamond particles and Al matrix would generate enormous thermal compressive stresses on diamond particles, which would also contribute to the formation of dislocations in the diamond particles.

### 3.2 Thermophysical properties

The CTE comparison of diamond/2024 Al in as-cast, annealed and aged condition is shown in Fig. 9. Regardless of heat treatment, the CTEs of diamond/

2024 Al composite were reduced greatly to nearly half that of the 2024 Al matrix [13] with high addition of diamond particles. It is well known that the thermal expansion of metal matrix composite is determined by the thermal expansion of metal matrix and the restriction of reinforcement through interfaces. High content of diamond particles imposes large restriction on surrounding aluminum matrix, which leads to the low CTE of the diamond/2024 Al composite. Moreover, annealing treatment has little effect on thermal expansion behavior. Aging treatment could further decrease the CTE of the composite. The formation of  $\text{Al}_2\text{Cu}$  phases at diamond–Al interface could improve the interfacial bonding and restriction of diamond particles on the surrounding Al matrix, which leads to the decrease of CTE. Furthermore, formation of  $\text{Al}_2\text{MgCu}$  precipitates in Al matrix could also contribute to the decrease of CTE. The CTE of diamond/2024 Al composite was about  $8.5 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$  between 20 and 100  $^\circ\text{C}$ , which is compatible with that of chip materials.



**Fig. 9** CTE comparison of as-cast, annealed and aged diamond/2024 Al composites

The thermal diffusivity ( $\alpha$ ) and conductivity ( $\lambda$ ) of diamond/2024 Al composites in as-cast, annealed and aged condition are shown in Table 2. The thermal conductivity of diamond/Al composite is expected to exceed 600 W/(m·K). However, the measured thermal conductivity of the present composite was about 100 W/(m·K), which was far lower than the theoretical value. Although larger particles were adopted (average particle size of 100  $\mu\text{m}$ ), thermal conductivity of diamond/Al, which was also fabricated by pressure infiltration method, was about 130 W/(m·K) [14]. The annealing treatment improved the thermal diffusivity and conductivity while aging treatment showed an opposite effect. There are two ways for heat transfer in diamond/2024 Al composite: free electron in Al matrix and phonon in diamond particles. Both of their movements would be scattered by interface. Therefore, heat conduction in diamond/2024 Al

composite is depended on Al matrix, diamond particles and their interface. The annealing treatment would release the residual thermal stress in Al matrix generated in fabrication, which is beneficial to heat conduction, and lead to improvement of the thermal conductivity. Moreover, the density of the dislocations decreased after annealing treatment, which also contributed to the increase of TC. However, after aging treatment, more scattering interfaces for electron and phonon were introduced into the composite, such as  $\text{Al}_2\text{Cu}$  phase at diamond–Al interface (Fig. 5(c)),  $\text{Al}_2\text{MgCu}$  precipitates in Al matrix (Fig. 7(a)) and higher density dislocations introduced more interfaces into the composite, which would scatter the movement of electron and phonon, and decrease the thermal conductivity. Therefore, thermal conductivity is slightly decreased after aging treatment.

**Table 2** Thermal diffusivity and conductivity of diamond/2024 Al composites after different treatments

Condition	Thermal diffusivity/ ( $\text{m}^2 \cdot \text{s}^{-1}$ )	Thermal conductivity/ ( $\text{W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$ )
As-cast	42.6	106.7
Annealed	43.7	109.3
Aged	41.0	102.3

## 4 Conclusions

1) 50% (volume fraction) diamond particle (5  $\mu\text{m}$ ) reinforced 2024 aluminum matrix (diamond/2024 Al) composites were prepared by pressure infiltration method. Diamond particles are distributed uniformly without any particle clustering, and no apparent porosities or significant casting defects are observed in the composites.

2) The diamond–Al interfaces of as-cast and annealed diamond/2024 Al composites are clean, smooth and free from interfacial reaction product. However, a large number of  $\text{Al}_2\text{Cu}$  precipitates are found at diamond–Al interface after aging treatment. High density dislocations are observed in the as-cast composite. The density of dislocations is decreased and increased after annealing and aging treatment, respectively.

3) The CTE of diamond/2024 Al composites is about  $8.5 \times 10^{-6} \text{ }^\circ\text{C}^{-1}$  between 20 and 100  $^\circ\text{C}$ , which is compatible with that of chip materials. Annealing treatment has little effect on thermal expansion behavior, and aging treatment can further decrease the CTE of the composite.

4) The thermal conductivity of obtained diamond/2024 Al composites is about 100 W/(m·K). Due to the release of residual stress and decrease of dislocations, thermal conductivity of diamond/2024 Al composites slightly increases after annealing treatment. However,

thermal conductivity of diamond/2024 Al composites slightly decreases the formation of Al<sub>2</sub>Cu at diamond–Al interface and Al<sub>2</sub>MgCu precipitates in Al matrix.

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## 热处理对金刚石/2024Al 复合材料 微观组织和热物理性能的影响

修子扬<sup>1</sup>, 王旭<sup>2</sup>, M. HUSSAIN<sup>3</sup>, 冯超<sup>1</sup>, 姜龙涛<sup>1</sup>

1. 哈尔滨工业大学 先进焊接与连接国家重点实验室, 哈尔滨 150001;

2. 辽宁石油化工大学 机电学院, 抚顺 113001;

3. Department of Chemical Engineering, COMSATS Institute of Information Technology, Lahore 54000, Pakistan

**摘要:** 采用挤压铸造法制备粒径为 5 μm、体积分数为 50% 的金刚石/2024Al 复合材料。退火处理后对其金相组织界面反应、界面结合情况以及金刚石颗粒的内部缺陷进行观察与分析, 并对其热物理性能进行测试与研究。结果表明, 金刚石/2024Al 复合材料的组织致密, 无明显的气孔、夹杂等缺陷; 颗粒为不规则多边形, 有棱角, 分布比较均匀。透射电镜观察表明, 部分金刚石颗粒内部有位错和层错存在, 而 2024Al 基体中的位错密度较大, 金刚石/2024Al 界面处有较多的界面反应物生成, 可能为 Al<sub>2</sub>Cu。复合材料在 20~100 °C 温度区间内的平均热膨胀系数为  $8.5 \times 10^{-6} \text{ } ^\circ\text{C}^{-1}$ , 退火处理的复合材料其热膨胀系数有一定程度的降低; 随着温度的升高, 复合材料的平均热膨胀系数也呈现增加的趋势。复合材料的热导率约为 100 W/(m·K), 退火处理能够提高复合材料的热导率。

**关键词:** 铝基复合材料; 金刚石; 界面; 退火; 时效; 热物理性能

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