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Triple ion beam cutting of diamond/Al composites for interface characterization

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ABSTRACT

A novel triple ion beam cutting technique was employed to prepare high-quality surfaces of diamond/Al composites for interfacial characterization, which has been unachievable so far. Near-perfect and artifact-free surfaces were obtained without mechanical pre-polishing. Hence, the as-prepared surfaces are readily available for further study and also, ready to be employed in a focus ion beam system for preferential selection of transmission electron microscopy samples. Dramatically different diamond/Al interface configurations — sub-micrometer Al_2O_3 particles and clean interfaces were unambiguously revealed.

1. Introduction

With the advent of innumerous electronic instruments, it has become imminent to develop novel materials to counter thermal expansion and to efficiently evacuate heat from electronic components for their optimal performance [1]. As a convention, composites have been preferred where the demand is for tailoring combination of properties that are not found in the individual components [2]. Recently, a diamond particle reinforced Al matrix (i.e. diamond/Al) composite has been developed for which it was demonstrated that, with a coefficient of thermal expansion lower than 10 ppm/K, thermal conductivity can be achieved as high as 670 W/m K by liquid metal infiltration [3,4] and around 600 W/m K by vacuum hot pressing (VHP) [5]. However, the heterogenic nature of the components involved in manufacturing such a composite, makes it extremely difficult to study the microstructure [6]. Conventional metallographic sample preparation methods, such as mechanical polishing, fail to equally polish the hard diamond particles and the soft Al matrix. In this regard, it is a common practice to break the sample and examine the fracture surfaces for microstructural characterization [7,8]. Hence, there is imminent need for an innovative preparation method for such a heterogeneous material.

When diamond particles are embedded in the Al matrix, the diamond/Al interface plays a crucial role in determining global thermal properties of the composite. An ideal interface should provide good adhesion as well as minimum thermal

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boundary resistance [9]. Previous reports [10,11] have mainly addressed technological development by optimizing processing conditions, mainly by improving interfacial bonding to enhance global thermal properties of the composite. Regarding the thermal boundary resistance, in fact, influence of the interface formation and diffusion on the interface conductance is still not fully understood, and most of the reports only have dealt with the interface characterization using the fracture surface technique [7,8]. Till today, none of the published works has been able to successfully obtain a flat surface for clear identification of the microstructure, especially interfacial features, except for a few works based on focus ion beam (FIB) preparation [9,12,13]. Apart from that, some indirect methods consisting of chemical [7] or electrochemical etching and cleaning [6] of the diamond particles extracted from the matrix, have been employed to reveal morphology and distribution of the interfacial reaction products. However, these methods suffer from serious drawbacks such as nanosized interfacial features can be destroyed or affected by the chemical treatment. For example, aluminum carbide (Al_4C_3) usually formed as an interface reaction product, is very reactive with either water or alcohol in the chemical or electrochemical etching solutions [14]. Hence, this paper aims at the applicability of a novel triple ion beam (TIB) cutting technique for a clean metallographic preparation of the diamond/Al composites and the application of FIB by targeting suitable interfaces prepared with TIB in order to cross check the sub-micrometer level, and to reveal the nanosized interfacial features.

2. Material and Methods

An aluminum powder (99.9% in purity) with an average particle size in the range 75–105 μm was used as material for the matrix. Synthetic diamond particles (type HWD40), with different average sizes of about 30 and 200 μm, were employed to obtain two sets of diamond/Al composites. Two component powders were taken in 60:40 (Al:diamond) volume percent ratios and were mechanically mixed before pouring into a graphite mould. Then, the mould was mounted on to a VHP unit. The VHP process involved the following steps: the graphite mould loaded with mixed powders was heated up to 673 K at the rate of 10 K/min and held for 30 min for degassing. It was further heated up to the sintering temperature of 923 K and a uniaxial pressure of 67.7 MPa was immediately applied with a holding time of 210 min (30 μm mesh particles) and 90 min (200 μm mesh particles) under the applied pressure. After cooling, disk-shaped specimens with a dimension of Ø 10 × 3 mm were pulled out of the mould. More details on fabrication and properties of the diamond/Al composites can be found elsewhere [15,16].

To highlight the efficiency of the TIB cutting, standard mechanical polishing was carried out on a tripod polisher using diamond-particle lapping polymer films, in the size range starting from 30 μm down to 0.5 μm for finishing. Alternatively, the new TIB technique was tested by using a Leica EM TIC 3X TIB slope cutter to achieve high quality surface finishing. As shown in Fig. 1, instead of one ion beam the TIC 3X uses three incident ion beams, namely J1, J2 and J3, which intersect at the center edge of the mask to form a milling sector of 100°. During milling the sample remains immobile contrary to the habitual oscillations in some other methods so that heat transfer between sample and stage is much better compared to that of an oscillated sample. The attached binoculars allow real-time observation to control milling time until a flat surface containing diamond/Al interfaces is acquired. In this case, the TIB parameters were set to 7 kV acceleration voltage and 2.6 mA gun current. An Ar gas of 99.999% purity was used for the milling process. Milling times used were around 6 and 8 h for the 30 μm and 200 μm diamond/Al composites, respectively.

The as-prepared surfaces were characterized using a HITACHI S-4700 scanning electron microscope (SEM) equipped with a field emission gun (FEG) and a Noran energy dispersive X-ray (EDX) spectrometry system. A STRATA DB 235 dual beam FIB system was employed to target and extract transmission electron microscopy (TEM) specimens containing the diamond/Al interface. A Philips CM30 instrument, operated at 300 kV and equipped with a Nanomegas 'Spinning Star' precession unit and a Noran EDX spectrometry system, was used for TEM characterization. High-resolution images were obtained using an FEI Tecnai G2 FEG microscope, operated at 200 kV.

3. Results and Discussion

Fig. 2 represents different surface qualities of the 30 μm diamond/Al composite obtained using two different methods — conventional metallographic preparation and TIB technique and observed by SEM. As can be seen from Fig. 2a, the large difference in hardness of the diamond and Al creates an un-even surface at which the final roughness remains around 5–15 μm despite repeated polishing. Mechanically, the softer Al matrix is easily polished off while the hard diamond particles remain un-altered and appear protruded from the polished surface, which causes the coarse un-even surface. A similar result has also been reported by other authors [7,12]. Hence, the main drawbacks of mechanical polishing are (i) un-even surface because of the inherent material properties, (ii) surface contamination caused mainly by use of polymer sheets leading to possible wrong conclusions on EDX analysis (along with lesser signals because of the protruding diamond particles) and (iii) un-even surfaces render FIB preparation difficult for a preferred site selection because of deleterious surface roughness and invisible interfacial features in some cases. Therefore, such a surface is not even qualitatively suitable for metallographic investigations. Comparatively, as shown in Fig. 2b, the TIB prepared sample exhibits a quite smooth surface, even revealing grain contrast in the Al matrix. It can be concluded from Fig. 2c that interfacial particles of several hundred nanometers in size are more or less homogenously distributed along the diamond/Al interfaces and protruding into the Al matrix.

Initially, it was suspected that the TIB method tested in the present study may introduce artifacts during ion milling. To check this possibility, another sample containing 200 μm diamond particles was prepared under similar conditions. The as-polished surface of this sample represented in Fig. 2f shows a quite clean interface where no interfacial particles are present except for very fine fissures. Thus, the only