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Manufacture and performance of diamond-coated thermocouples

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Abstract

Diamond-coated thermocouples were prepared with the aim of measuring the temperature at the interface between a grinding wheel and workpiece material. Thermocouples with K-type, R-type and G-type alloy combinations were prepared. Continuous diamond films were grown on each type of thermocouple using hot filament chemical vapour deposition techniques. The resulting diamond films were characterised using Raman spectroscopy and scanning electron microscopy. Diamond deposition was found to be particularly successful, in terms of quality and adhesion, with the G-type thermocouple. Diamond-coated G-type thermocouples, with varying diamond thickness, were then prepared and characterised with respect to their thermoelectric voltage response. Such measurements were performed under *static* conditions, where the thermocouple joint had been allowed to reach a thermal equilibrium with its environment, and under *dynamic* conditions, where a pulse of heat was applied to the thermocouple at various intensities and frequencies.

Keywords: Deposition; Diamond; Thermocouple; Thermoelectric response

1. Introduction

Research is currently being undertaken to minimise and eventually to eliminate surface damage caused during the grinding of brittle materials, [1–3]. Heat generated at the interface between the workpiece and the grinding wheel is a significant factor in determining the extent of the surface damage, [4,5]. Temperatures at the grinding interface are commonly measured with thermocouples embedded within the brittle material and positioned close to the grinding surface. However, this approach has two distinct disadvantages: firstly, the temperature measured at the thermocouple may not correspond to that of the grinding interface and secondly, it cannot be incorporated within large-scale manufacture regimes. We are developing an alternative approach in which a thermocouple is coated with diamond and then embedded within the grinding wheel. The high wear resistance and thermal conductivity of the diamond will enable the thermocouple to be positioned at, or close to, the interface between the grinding wheel and the workpiece, where the recorded temperature should provide an accurate measure of the grinding temperature.

Diamond-coated fibres have been manufactured successfully using hot filament chemical vapour (HFCVD) techniques, [6,7]. The successful deposition of diamond onto thermocouples using HFCVD techniques relies on the following properties of the thermocouple alloys:

(1) A melting point sufficient to withstand the temperature of the deposition process.
(2) A low thermal expansivity thus minimising interfacial strains between the thermocouple alloy and the diamond.
(3) Minimal reactivity with the deposition gases so as to maintain their thermoelectric properties.

An investigation into the manufacture and performance of diamond-coated thermocouples has been undertaken and complements earlier work [8]. Diamond-coated thermocouples consisting of K-type, R-type and G-type alloy combinations were prepared, with the diamond deposition characteristics of each alloy investigated using Raman spectroscopy and scanning electron microscopy techniques. Further work was carried out to explore the reactivity of the G-type

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thermocouple alloy with the deposition gases. Finally the thermoelectric properties of the diamond-coated G-type thermocouple alloy were measured in a static thermal environment, where the thermocouple is allowed to reach thermal equilibrium with its environment, and in dynamic thermal environments where the frequency response of the thermocouple was characterised.

2. Experiments and results

2.1. Diamond-coated thermocouple fabrication and characterisation

Thermocouples with K-type ($Ni_{90}Cr_{10}$–$Ni_{95}Mn_2$-Al_2Si_1), R-type ($Pt_{87}Rh_{13}$–Pt) and G-type ($W_{74}Re_{26}$–W) alloy combinations were prepared using 125 μm

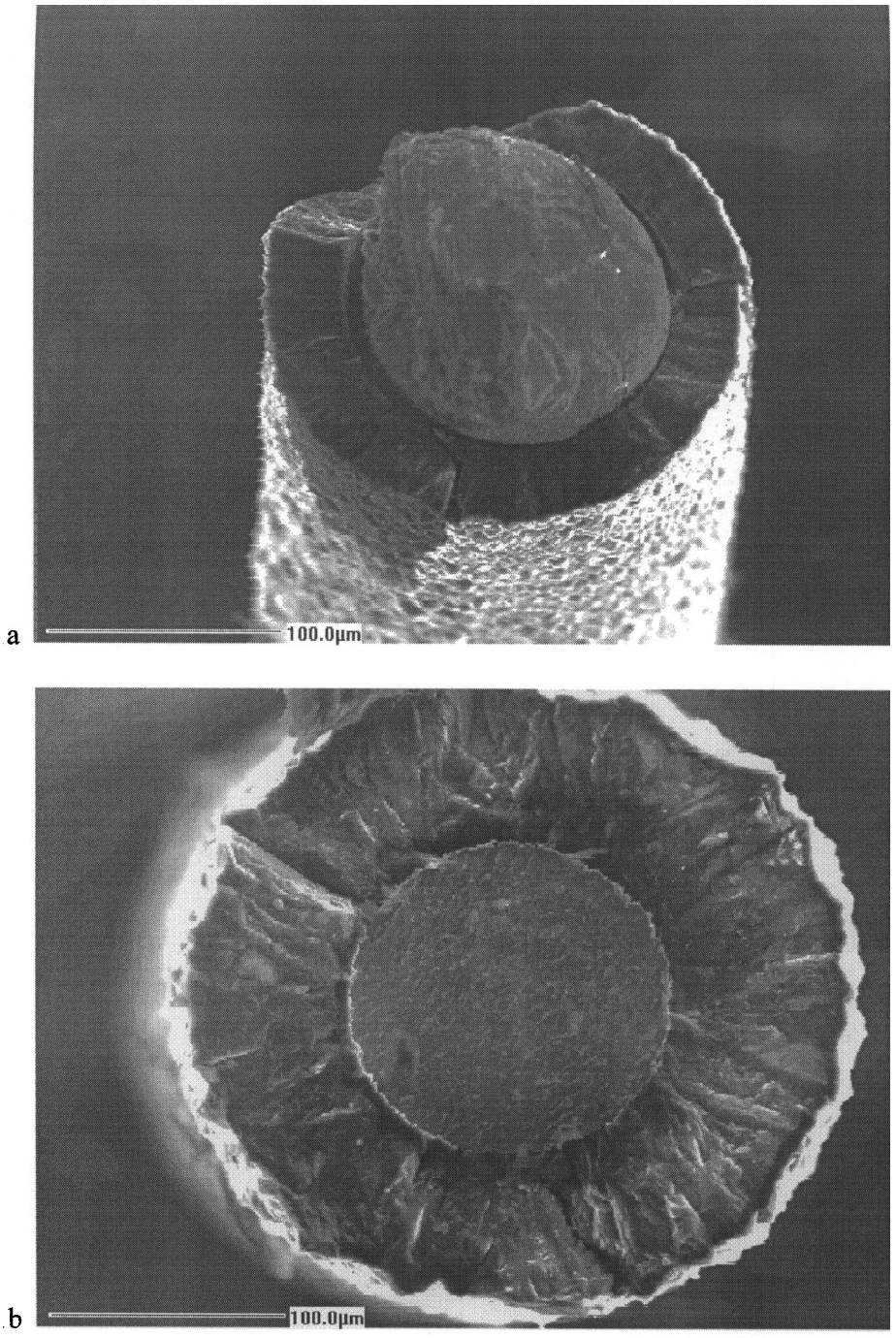

Fig. 1. (a) Fracture surface of R-type diamond-coated thermocouples showing debonding between the wire core and diamond coat. (b) Fracture surface of G-type diamond-coated thermocouple showing no debonding between the wire core and the diamond coat.

diameter wires. The wires of each thermocouple alloy combination were twisted and spot welded at an appropriate voltage to form a bead.

Diamond was deposited onto the thermocouples using HFCVD techniques, using a 0.85% methane–hydrogen gas mixture at 30 Torr (4 kPa), [6]. The temperature of the heating filament was maintained at 2200 °C, as measured with an infrared pyrometer. Radiation from this heated the thermocouples to approximately 900 °C. Deposition times for the K-type and R-type thermocouples was 30 h at a rate of 1.5 μm h^{-1}. Diamond deposition times for G-type thermocouples were varied providing diamond coating thicknesses of 20, 40, 60 and 140 μm.

To examine the coatings on each of the thermocouples, the fibres were fractured and their cross-sections examined under a scanning electron microscope at the appropriate magnification. Fig. 1 shows typical cross-sections for the diamond-coated R and G-type thermocouples. The image of the R-type thermocouple fibre shows that there is poor adhesion between the diamond coat and the wire core. In contrast, the coated G-type thermocouple appears to have good structural integrity with no evidence for debonding of the diamond from the metal core and no cracking of the diamond coat observed. This suggests that residual stresses produced during the HFCVD of diamond are not significant for this metal alloy and is consistent with previous reports of successful HFCVD of diamond on tungsten cores, [6]. Similar images of the K-type thermocouple fibres show that the coating is brittle and delaminates easily. Raman analysis shows that the coating to be non-diamond carbon.

The deposition of diamond and other carbon phases was investigated for each diamond-coated thermocouple alloy combination using Raman spectroscopy. The Raman spectrum analyser used an argon ion laser with a wavelength of 488 nm and a power output of 3 mW. Raman spectra between 800 and 1800 cm^{-1} were recorded. Fig. 2 shows the Raman spectra obtained from the K-type, R-type and G-type thermocouples after HFCVD. The intense 1332 cm^{-1} feature confirms that diamond deposition has been successful for both R-type and G-type thermocouples; the presence of a small amount of amorphous carbon is also indicated by the deviation in the Raman response from the background between 1400 and 1600 cm^{-1}. Diamond deposition for the K-type thermocouple was unsuccessful; the two broad features at 1355 and 1580 cm^{-1} are indicative of graphitic carbon.

Diamond growth on tungsten, the main element of a G-type thermocouple, was investigated further by Auger electron analysis. Tungsten-cored diamond fibres which had different coating thicknesses, as a result of different deposition times, were fractured in air before being loaded into a scanning Auger microprobe analyser. Prior to analysis each fracture surface was sputter cleaned, at

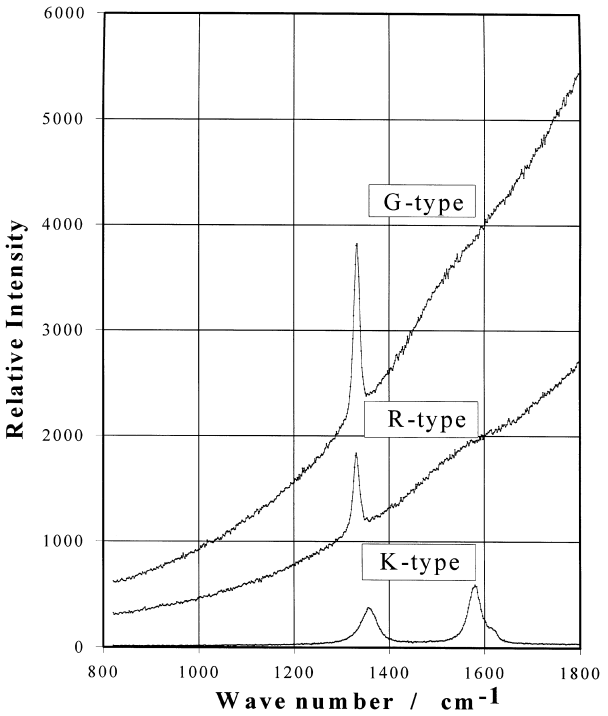

Fig. 2. Raman spectra of K-type, R-type and G-type thermocouples after removal from the HFCVD reactor.

a pressure of 10^{-10} Torr to ensure freedom from contamination. Auger analysis was carried out at a working potential of 10 keV, using a beam a current of 3×10^{7} A with a spot diameter of 0.5 μm. Auger spectra were generated between 30–600 eV. The results of the Auger analysis are shown in Fig. 3. The extent of carburisation of the tungsten core was assessed by calculating the ratio of the Auger electron intensity for tungsten carbide at 286 eV, I_{wc}, to that for tungsten at 180 eV, I_w. The ratio I_{wc}/I_w is plotted from the centre of the fibres' cores to the diamond–thermocouple interface. Fig. 3 clearly shows that the formation of tungsten carbide is limited to the interface between the tungsten wire and diamond coating. The tungsten carbide interfacial layer forms during early stages of diamond growth but its development is slowed with increasing diamond growth until it reaches a constant thickness.

2.2. *Thermoelectric response of G-type diamond-coated thermocouples*

The static thermoelectric responses of G-type thermocouples with diamond coatings between 0 and 60 μm were measured in a box furnace. Thermocouples were heated at 10 °C min^{-1} to a maximum temperature of 600 °C. The thermoelectric response of each thermocouple plotted against that of a K-type thermocouple positioned adjacent is shown in Fig. 4. The thermoelectric responses of the thermocouples are similar below 150 °C and above 450 °C. However, between these two temper-

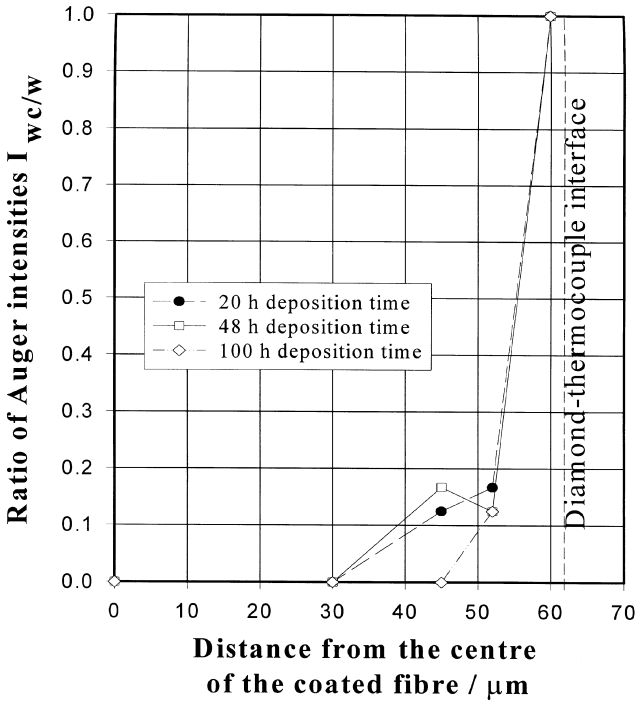

Fig. 3. Plot showing the ratio I_{wc}/I_w, deduced from Auger analysis across fracture surfaces of tungsten cored diamond-coated fibres produced using different deposition times.

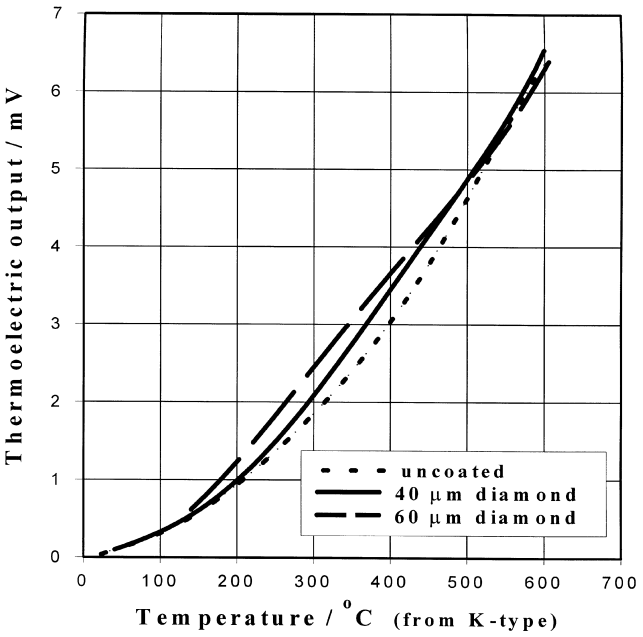

Fig. 4. The thermoelectric response of bare and diamond-coated G-type thermocouples as a function of diamond thickness measured in a static thermal environment.

atures the difference between the thermoelectric response of the coated and uncoated thermocouples varies by as much as 0.6 mV corresponding to a temperature of 100 °C. The reason for this disparity is not clear and will be investigated further in forthcoming work.

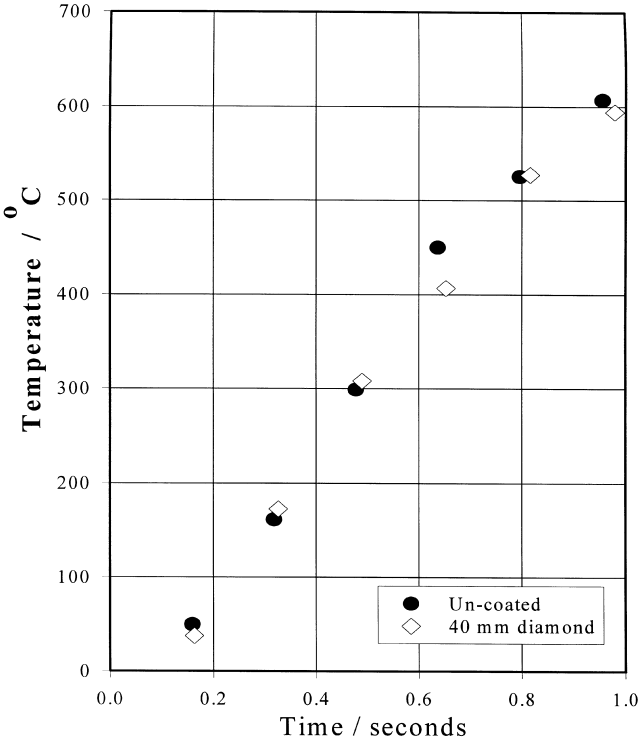

Fig. 5. The dynamic thermoelectric response of an uncoated and 40 μm thick diamond-coated G-type thermocouple with a heating rate of 650 °C s^{-1}.

The dynamic thermoelectric response of G-type thermocouples was investigated using a rapid thermal annealer with an argon atmosphere. The thermoelectric responses of an uncoated G-type thermocouple and a 40 μm diamond-coated G-type thermocouple were amplified with a half-bridge amplifier before being digitised at a 7 Hz frequency. The thermocouples were heated at 650 °C s^{-1} and the thermoelectric responses measured as a function of time. The results of this investigation, which are shown in Fig. 5, suggest that the frequency response of the thermocouple is not affected by the diamond coat.

3. Concluding remarks

Diamond-coated thermocouples have been successfully prepared for R-type and G-type thermocouple combinations. The diamond-coated G-type thermocouples exhibited good structural integrity. Debonding between the diamond coat and the thermocouple wire is evident for R-type diamond-coated thermocouples.

The reactivity of the G-type thermocouple combination to the HFCVD deposition gases was low. A tungsten carbide layer was formed by the HFCVD process but is shown to be restricted to regions close to the interface between the thermocouple wire and the diamond coating.

The thermoelectric responses of diamond-coated G-type thermocouples are shown to be unaffected by the diamond coating and independent of diamond thickness when measured in both static and dynamic thermal environments.

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