

Nano-Structured Carbide-Derived Carbon Films and Their Tribology

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Abstract: Carbide-derived carbon (CDC) is a form of carbon produced by reacting metal carbides, such as SiC or TiC, with halogens at temperatures high enough to produce fast kinetics, but too low to permit the re-arrangement of the carbon atoms into an equilibrium graphitic structure. The structure of CDC is derivative of the original carbide structure and contains nanoscale porosity and both sp^2 and sp^3 bonded carbon in a variety of nanoscale structures. CDC can be produced as a thin film on hard carbides to improve their tribological performance. CDC coatings are distinguished by their low friction coefficients and high wear resistance in many important industrial environments and by their resistance to spallation and delamination. The tribology of CDC coatings on SiC surfaces is described in detail.

Key words: carbon; coatings; tribology; friction; chlorination

Introduction

Carbide-derived carbon (CDC) refers to a unique structure of carbon produced when metal carbides, such as SiC, are exposed to halogens at moderately elevated temperatures. The halogens react with the metal component of the carbide to form volatile species, such as $SiCl_4$, but because CCl_4 is not thermodynamically stable at high temperatures, the carbon is left behind^[1-3]. When the process is performed at temperatures that are low in comparison to the graphitization temperature of carbon, the structure of the CDC will be a metastable combination of nanoporous amorphous carbon, nanocrystalline diamond, and nested fullerene structures^[4-7].

CDC materials have many potential applications

deriving from their unique physical and chemical properties. The applications that may be most easily exploited are in tribology, where CDC functions as a solid lubricant film. CDC layers in the thickness range from 1 to 100 μm can be grown quickly on hard carbides, such as SiC or WC, by exposure to flowing chlorine gas at temperatures in the range of 800°C to 1000°C. These carbides are widely used in wear-sensitive applications, such as bearings or pump seal rings, because of their high hardness and wear resistance. CDC coatings can be applied to the surfaces of SiC components with minimal dimensional change, and produce a substantial reduction in the friction coefficient during sliding^[8-10]. The CDC coating is much more wear resistant than graphitic carbon. The mechanical hardness of CDC is affected by structural variations, and changes gradually from a high value near the CDC/SiC interface to a lower value at the external surface. This graded hardness matches the

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hardness of the underlying SiC and reduces the susceptibility of CDC to spallation and delamination in comparison to vapor-deposited diamond or diamond-like carbon.

The tribological properties of CDC can be related to the properties of the carbide substrate and the processing conditions under which the CDC conversion layer was produced. CDC layers have been formed on commercial SiC materials widely used in applications such as pump seal rings by exposure to flowing Ar-3.5% (vol) Cl₂ gas mixtures in fused silica furnace tubes at temperatures on the order of 1000°C. The resulting CDC coatings have been characterized by optical and electron microscopy, Raman and X-ray emission spectroscopy, mechanical testing by nano-indentation, and in a pin-on-disk tribology tester. The results provide some insights into the performance of these coatings and the mechanism by which they reduce tribological losses.

1 Experimental Details

The apparatus in which the CDC layers were grown has been described elsewhere^[1,2]. It consists of a fused silica reaction tube in a resistance heated electric furnace, fitted to a gas flow and purification system set up for handling mixtures of argon, chlorine, and hydrogen. In a typical experiment, a SiC specimen is placed within the furnace, and the furnace is purged with the chlorine-containing gas mixtures before heating to the intended reaction temperature. The furnace is held at the test temperature for a fixed period of time (often on the order of one hour), and then the furnace is cooled to near room temperature in the flowing gas mixture so that the test specimen can be removed for characterization. The gas flow system is designed such that the chlorine flows only through glass or polytetrafluoroethylene (PTFE) tubing between leaving the supply cylinder and being exhausted to a fume hood to avoid corrosion losses of the gas. Halide deposits must be removed periodically from the reaction tube to avoid flow interruptions due to plugging.

CDC coatings on SiC and other carbides have been produced in varying thickness and have been characterized by optical and electron microscopy and by X-ray and Raman spectroscopy. The tribological performance of the layers has been evaluated in pin-on-disk tribology tests as well as more specialized seal tests.

Figure 1 is an optical micrograph of a CDC coating produced on sintered alpha SiC after one hour in flowing Ar-3.5% Cl₂ at 1000°C. The thickness of the layer ranges from 10 to 20 μm. The convolutions in the SiC/CDC interface result from local variations in the rate of conversion of SiC to CDC due to the differences in orientation of the SiC grains. These convolutions interlock the CDC coating with the SiC substrate and prevent delamination during tribological testing.

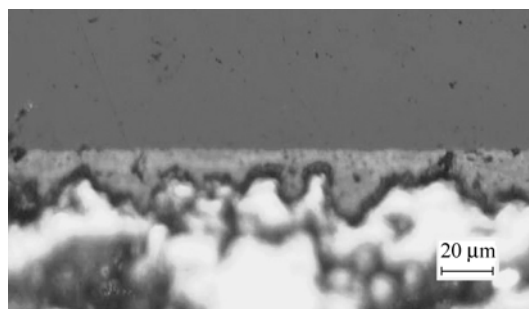


Fig. 1 Cross-sectional optical micrograph of CDC produced on sintered alpha SiC after one-hour treatment in flowing Ar-3.5% Cl₂ at 1000°C. The upper dark-grey layer is epoxy, CDC coating is in the middle, and the bottom part of the micrograph shows SiC.

The interface between the SiC and the CDC is further strengthened by a gradient in the mechanical properties of the CDC as it approaches the SiC/CDC interface. Figure 2 shows the results of nano-

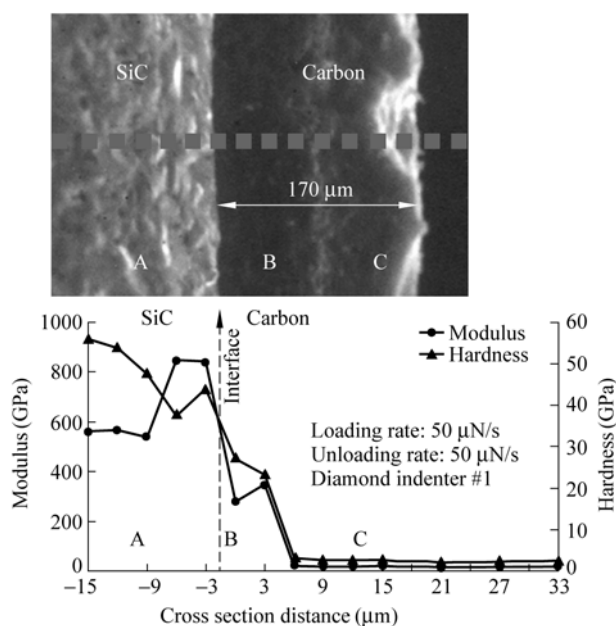


Fig. 2 Cross-sectional micrograph and results of nano-indentation tests on CDC near SiC/CDC interface on CDC produced in flowing Ar-3.5% Cl₂ at 1000°C.

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