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Preparation and sintering of silica-coated ultrafine diamonds-vitrified bond composite powders



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ABSTRACT

The oxidation resistance of ultrafine diamonds (UFDs) was improved by encapsulating UFDs into silica shells forming core/shell structures with a PVP-aided method. Meanwhile, the dispersion stability of the UFDs in the inorganic salt aqueous solution was also improved greatly. In addition, adopting the silica-coated UFD aqueous suspension including multi-component inorganic salts, the coated UFDs-vitrified bond composite powders with higher homogeneity were obtained by using a polyacrylamide gel method, which was used for manufacturing the vitrified-bonded UFD wheels. The porous specimens of the UFD grinding wheels were fabricated with the above composite powders at low temperature in the air and in a muffle furnace. The results suggested that the porosity, bulk density and bending strength of the specimens were 36.3%, 1.71 g/cm⁻³ and 62.9 MPa, respectively. Moreover, no obvious aggregation and degradation of UFDs were observed in the above UFD specimens. These results demonstrate a new pathway of preparing multifunctional nanostructure with a low-aggregation and high oxidation resistance that can be applied for manufacturing vitrified-bonded UFD wheels.

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1. Introduction

Ultrafine diamond (UFD) grinding wheels composed of ultrafine diamonds and a bond material have been applied increasingly in the ultra-precision grinding field for hard-brittle materials [1-3]. The primary and familiar bond materials including vitrified, resin and metal bonds are used for UFD grinding wheels [4,5]. Contrasting to resin and metal bond wheels, vitrified bond UFD wheels are the most promising ultra-precision grinding wheels for machining hard-brittle materials because of their outstanding properties, such as controlled porosity, good self-dressing capability, high machining efficiency and surface precision [6,7]. With the development of technology and science, the surface quality of hard-brittle materials has been finished to meet higher demands, for example, silicon wafers with a roughness of less than 1 nm and lower subsurface damage layer can meet the requirement for electronic applications [6,8,9]. Consequently, it is of great importance to avoid the aggregation and degradation of UFDs as they negatively affect the ultra-precision grinding performance of the UFD grinding wheels.

Recently, some researchers have reported that the modified UFDs were dispersed in aqueous or nonaqueous media as loose abrasives for lapping and polishing processes [10,11]. However, the processes involve some disadvantages, such as low machining efficiency, high machining cost and environmental pollution, which have been gradually solved by fixed-

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0263-4368/\$ - see front matter © 2013 Elsevier Ltd. All rights reserved. http://dx.doi.org/10.1016/j.ijrmhm.2013.12.001 abrasive wheels for ultra-precision grinding [12]. The fixed UFD polishing pads without aggregated UFDs have been prepared using sol–gel method by Liu [13] and Hu [14]. Although the aggregation of UFDs is settled, the polishing pads are easy to be torn apart in the polishing process because of their low strength. Besides the aggregation of UFDs, the lower oxidation resistance temperature restricted their wide application in many fields. In recent years, some coating methods for UFDs have been developed to improve their oxidation resistance [15–17]. Nevertheless, little work has been reported on a promising coating material for improving simultaneously the dispersibility and oxidation resistance of UFDs. Thus, the work has been explored and a breakthrough progress has been made in this study.

In this work, silica as one of the components of vitrified bond was used for synthesizing silica-coated UFD composites using polyvinylpyrrolidone (PVP) as a coupling agent between UFD and silica by modified Stöber method. Both the UFD dispersion and oxidation resistance were improved obviously. Moreover, the coated UFDs-vitrified bond composite powders were prepared adopting the silica-coated UFD aqueous suspension including multi-component inorganic salts by a polyacrylamide gel method. The vitrified bond UFD wheels were manufactured with the above composite powders, and their microstructures and mechanical properties were also further investigated.

2. Experimental procedure

Firstly, the silica-coated UFD aqueous suspension was prepared according to the procedure scheme illustrated in Fig. 1. Herein, all of



Fig. 1. Procedure scheme of the synthesis of silica-coated UFD composites with PVP by using a modified Stöber method.

the chemical reagents were of analytical grade. The pristine UFDs were the ultrafine powder obtained by crushing macro-particles, whose particle size was 100–200 nm. 1.20 g pristine UFDs and 0.45 g PVP were weighted and then put into 450 mL deionized water, which was ultrasonically treated for 30 min to break down agglomerates. Then the solution was stirred vigorously for 6 h at room temperature. Under continuous stirring, a moderate amount of ammonia solution (25% NH₃ in water) and 45 mL tetraethylorthosilicate (TEOS) ethanol solution (the volume ratio of TEOS and ethanol was 1:4) was successively added into the above solution. The reaction mixture was stirred for another 12 h and then the silica-coated UFD aqueous suspension was

Table 1

Chemical compositions of the vitrified bond.

Component	SiO ₂	B ₂ O ₃	Al_2O_3	R ₂ O
wt.%	57.5	18.7	10.6	13.2



Fig. 2. HRTEM images of the composites: (a) UFDs coated by SiO_2 with PVP-aided method, and (b) without PVP-aided method.

obtained. On the above basis, the coated UFDs-vitrified bond composite powders (the weight ratio of UFDs and vitrified bond was 1:3) were prepared by a polyacrylamide gel method, which was illustrated in the Refs. [18,19]. The composition of the vitrified bond was shown in Table 1. Besides silica, other components of the vitrified bond were introduced with multi-component inorganic salts.

With the coated UFDs and pristine UFDs vitrified bond composite powders, the porous UFD grinding wheels (marked as PW_{coa} and PW_{pri}) were fabricated by cold pressing and sintering under the same processing conditions. The as-prepared composite powders were pressed at 150 MPa for 30 s in a ($50 \times 5 \times 5$) mm mold. The porous UFD grinding wheels were sintered in the air and in a muffle furnace. The sintering parameters were as follows: sintered to 450 °C increasing at 4 °C/min and held for 1 h, sintered to 600 °C increasing at 2 °C/min



Fig. 3. (a) TG curves of the coated and pristine UFDs, and (b) particle size distribution curves of the coated and pristine UFDs in the inorganic salt aqueous solutions (salt concentration: 0.35 mol/L).

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