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Study on Microstructure and Properties of Al_2O_3/(W,Ti)C Self-Lubricating Tool Material with the Addition of $Al(OH)_3$ Coated CaF_2

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Abstract

$Al(OH)_3$ coated CaF_2 powders were prepared by liquid phase coating method with heterogeneous nucleation principle. Al_2O_3/(W,Ti)C self-lubricating tool material was prepared with the addition of the $Al(OH)_3$ coated CaF_2 powders. The mechanical properties of self-lubricating tool materials with different contents of the coated powders were investigated. The results indicate that the best mechanical properties of the tool material were obtained when the volume content of $Al(OH)_3$ coated CaF_2 was 10%. Compared to its counterpart tool material with uncoated powders, hardness, fracture toughness and flexural strength were increased by 21.7%, 8% and 10.7% respectively. Scanning electron microscope (SEM) was used to observe and analyze the microstructure of tool materials. The result shows that mixed intergranular fracture and transgranular fracture is fracture modes of the tool materials. The cutting tests were carried out using the prepared ceramic cutting tools, which show good wear resistance and antifriction property.

Keywords

Heterogeneous nucleation, Self-lubricating tool, Microstructure, Mechanical properties, Cutting performance.

1. Introduction

With the rapid development of high speed machining technology, it is impossible to meet the production demand just by adding cutting fluid to reduce the cutting temperature [1-3]. The research and application of self-lubricating cutting tool provide a new selection for high speed machining. The significance of achieving self-lubricating for cutting tool material is that it can decrease friction, reduce wear and save the cooling lubricating system. In addition, in can overcome the secondary contamination caused by cutting fluid and improve the ability to protect environment effectively [4-6].

In recent years, the research on the surface coated self-lubricating ceramic cutting tools with good lubricating properties has attracted researchers' attention and has obtained some progress [7-9]. Yan et al. [10] prepared Co-based alloy/TiC/CaF_2 self-lubricating coatings on copper by the method of laser cladding. Pandey et al. [11] prepared CaF_2 thin films by electron beam evaporation technique at room temperature and in a vacuum environment. Shi et al. [12] prepared CaF_2-coated $LiMn_{1/3}Ni_{1/3}Co_{1/3}O_2$ particles used for cathode material via a wet chemical process followed by a low-temperature annealing process. Powder coating technology is introduced to the preparation of self-lubricating ceramic cutting tool. It can coat solid lubricant particles, which shows poor performance, with ceramic material, which has excellent mechanical properties. Thereby it can reduce the effect of solid lubricant on the mechanical properties of cutting tool material and maintain the self-lubricating effect of the cutting tool.

In this work, the material design concept of particles surface coating technology was introduced to the research and development of self-lubricating ceramic cutting tools. Coated ceramic self-lubricating cutting tools with self-lubricating properties and improved mechanical properties were fabricated. The combination of lubrication properties and mechanical properties was realized. Moreover, mechanical properties, cutting performance and microstructures of the cutting tool material were studied in detail.

2. Experimental Procedure

2.1 Preparation of $Al(OH)_3$ Coated CaF_2 Powders

In this study, core-shell structured $Al(OH)_3$ coated CaF_2 powders (denoted by CaF_2@$Al(OH)_3$) were prepared by heterogeneous nucleation method. The raw materials used in the experiment are listed in Table 1.

Tab. 1. Raw Materials of Experiment

Name	Chemical formula	Purity
Aluminum nitrate nonahydrate	$Al(NO_3)_3·9H_2O$	99%
Ammonia	NH_3H_2O	Analytically pure
Anhydrous ethanol	CH_3CH_2OH	Analytically pure
Calcium fluoride	CaF_2	99%

Powers preparation procedure is as follows: (1) Put a certain amount of $Al(NO_3)_3·9H_2O$ into deionized water, stirring them by a magnetic stirrer until complete dissolution. (2) Put a certain amount of milled CaF_2 powders into a beaker and mix them with deionized water. (3) CaF_2 solution was dispersed uniformly with the ultrasonic disperser and at the same time heated to a certain temperature in the water bath, adding polyethylene glycol (PEG) as surface dispersant. (4) Stir the preceding CaF_2 solution by the DF-Ⅱ digital display collector magnetic stirrer, dropping ammonia slowly until a small amount of white floe appears. (5) When dropping finished, continue stirring and insulation 30min. (6) After the reaction was completed, the solution aged 12h at room temperature. (7) The suspension was repeatedly washed with deionized water followed by anhydrous ethanol 2 to 3 times, then centrifuged by centrifuge at 2000 rpm for 10 min. To complete the process, it was dried in a vacuum oven at 100℃ for 12 h to obtain CaF_2@$Al(OH)_3$ powders.

Microstructures of the as-prepared $A1(OH)_3$ coated CaF_2 powders were observed by SEM (SUPRA^{TM}55, Carl Zeiss Group, Germany) and the composition was analyzed using energy

dispersive spectrometry (EDS, SUPRA^{TM} 55, Carl Zeiss Group, Germany).

2.2 Preparation of Self-lubricating Tool Materials

The α-Al_2O_3 powders (99.9% pure) with an average particle size of 1μm was chosen as the matrix materials. The (W,Ti)C powders (99.8% pure) with an average particle size of 1μm were used as the reinforcing phase. Powders of CaF_2@$Al(OH)_3$ have been obtained by experiment with an average particle size of 3-5μm. MgO and NiO were used as sintering additives. The volume ratio of Al_2O_3 and (W,Ti)C is 1.2:1. Using wet dispersing method to mix materials, drying and sieving powders then filling, cold press molding and hot-pressing sintering under the vacuum state to prepare coated self-lubricating cutting tool material (Al_2O_3/(W,Ti)C/(CaF_2@$Al(OH)_3$)). Sintering parameters are as follows: heating rate is 15℃/min, holding temperature is 1650℃ for 30 min, hot-pressing pressure is 25 MPa. The volume fraction of CaF_2@$Al(OH)_3$ was 5%, 10% and 15%, respectively. Microstructures of the as-prepared cutting tool material was observed by SEM and the phase compositions of cutting tool material was analyzed using X-ray diffraction (XRD, Innov-X System, USA). The sample to be tested is placed in the center of the Cu target, regulating instrument equipment until voltage is 40kV and current is 40mA. The scanning speed is set to 0.4°/min; the range of scanning angle is adjusted to 20°~80°.

2.3 Mechanical Properties Tests of Cutting Tool Materials

The as-prepared Al_2O_3/(W,Ti)C/(CaF_2@$Al(OH)_3$) ceramic disks were cut into bars with an inner-circular cutter. The bars were ground and then polished to obtain test specimens with 3mm×4 mm×30 mm in dimension. The flexural strength was determined by three-point-bending technique with a span of 20 mm at a crosshead speed of 0.5 mm/min. The hardness was measured using a Vickers hardness tester (Hv-120) with a load of 196 N and loading time of 15 s. The fracture toughness was evaluated by the indentation method using cracks generated by the Vickers indentations. Five specimens for each ceramic disk were tested and the average values of flexural strength, hardness and fracture toughness were taken.

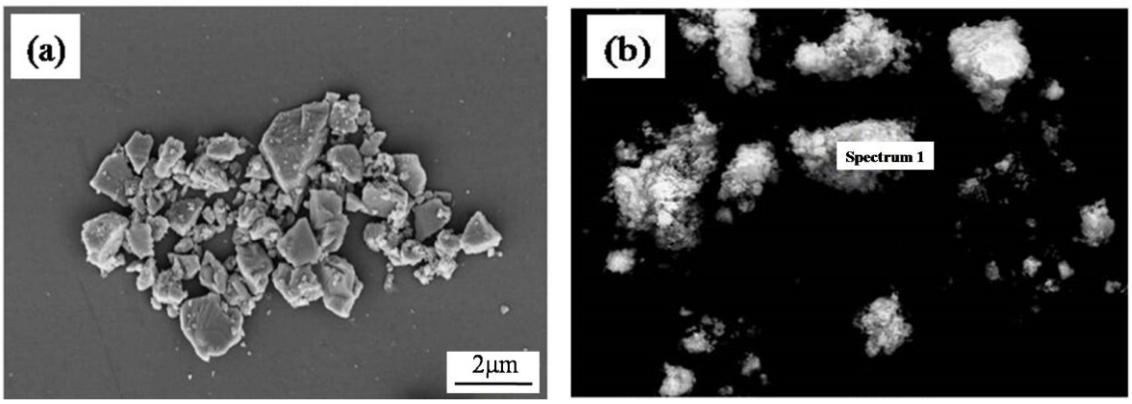
2.4 Cutting Experiment of Cutting Tools

Cutting tests were carried out on a CDE6140A lathe under dry cutting condition. The geometric parameters of Al_2O_3/(W,Ti)C/(CaF_2@$Al(OH)_3$) self-lubricating tool are as follows: rake angle γ_0=-8°; relief angle α_0=8°; inclination angle λ_S=0°; cutting edge angle κ_r=45°; chamfering width $b_{\gamma 1}$=0.2mm; chamfering angle γ_{o1}=-20°; corner radius r_ε=0.2mm. The workpiece material was 45# hardened carbon steel with hardness of HRC 25~27. The machining process was performed at a constant feed rate of 0.1 mm/r and a constant depth of cut of 0.2mm. Workpieces were machined at the cutting speed of 60 m/min, 80 m/min and 110 m/min, respectively. The wear morphologies of cutting tools was observed and analyzed by SEM.

3. Results and Discussion

3.1 Microstructural Characterization of Cutting Tool Materials

SEM images of $Al(OH)_3$ coated CaF_2 in comparison with uncoated ones are shown in Fig.1. It can be seen that coated powders' surfaces become coarse, as shown in Fig.1 (b) and (c) because of $Al(OH)_3$ nucleation growths. It is found in Fig.1 (d) that many Al and O elements appear on the powders surface through EDS analysis. Combined with the coating reaction equation (1), it is considered that the surface of the powders were coated with a layer of $Al(OH)_3$.



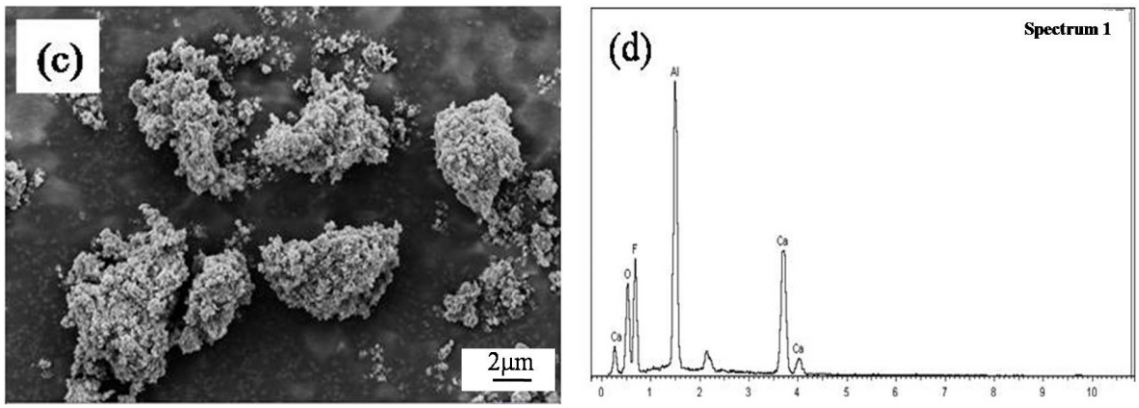


Fig.1 (a) SEM Micrographs of Raw CaF_2 Powders; (b) (c) Coated CaF_2 Powders; (d) EDS Analysis Corresponding to Spectrum 1

$$Al(NO_3)_3+3NH_3{\cdot}H_2O=3NH_4NO_3+ A1(OH)_3 \quad (1)$$

Fig.2 presents XRD image of the Al_2O_3/(W,Ti)C/(CaF_2@$Al(OH)_3$) self-lubricating tool material. It can be observed that tool materials contain CaF_2 and no derivatives are found. So all the components in the matrix were retained after sintering and CaF_2 did not react with the matrix material. During the sintering process, the coating layer of $Al(OH)_3$ decomposed into Al_2O_3, which is expressed as reaction equation (2). Therefore, no $Al(OH)_3$ was detected in Fig. (2).

$$2Al(OH)_3(s)= Al_2O_3(s)+3H_2O(g) \quad (2)$$

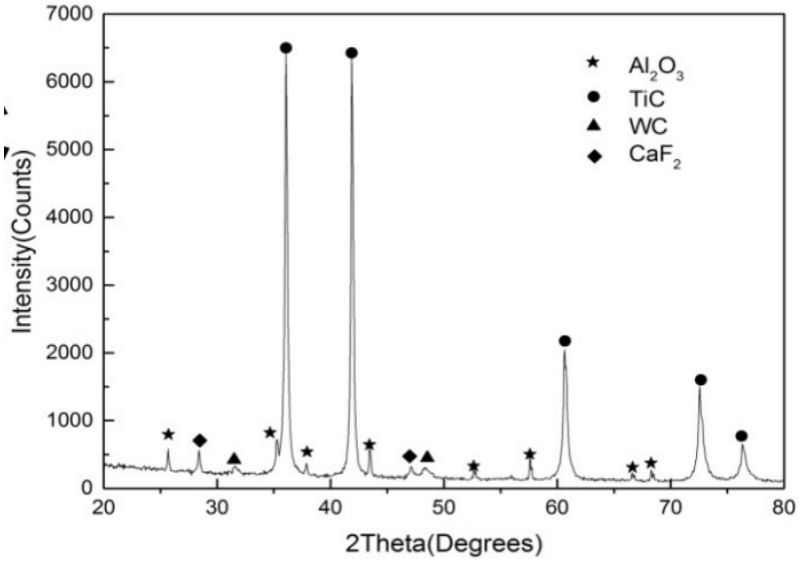


Fig.2. XRD Spectrum of Al_2O_3/(W,Ti)C/(CaF_2@$Al(OH)_3$) Self-lubricating Tool Material

Fig.3 shows the polished surfaces of self-lubricating tool material with different contents of CaF_2@$Al(OH)_3$. The white is identified by EDS to be (W,Ti)C, and the black is Al_2O_3 and CaF_2@$Al(OH)_3$. The distribution of each component of the cutting tool material is even, and the grain arrangement is compact and the particles are wrapped with each other can be found, as seen in Fig.3.

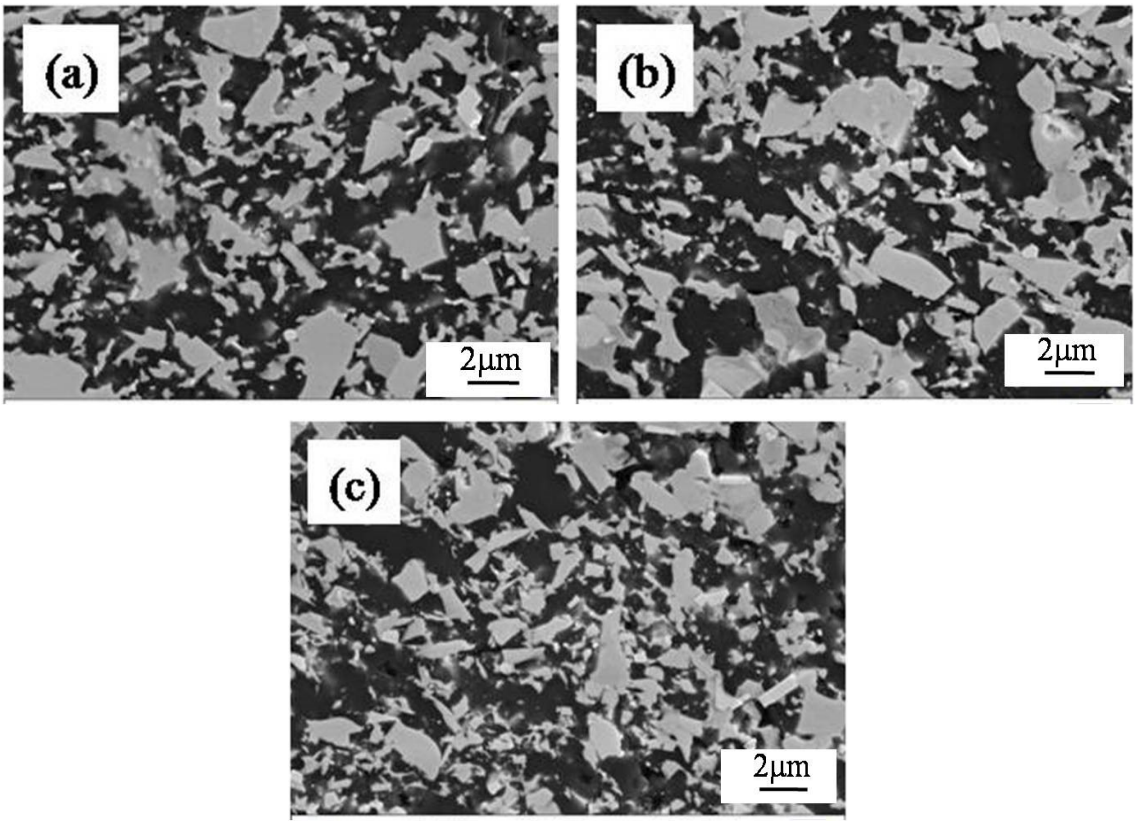


Fig.3. SEM Micrographs of Polished Surfaces of Self-lubricating Tool Material with Different Volume Contents of CaF_2@$Al(OH)_3$: (a) 5%; (b) 10%; (c) 15%

Fig.4 shows the fracture surface of the self-lubricating tool material with different contents of CaF_2@$Al(OH)_3$. It can be seen that the tool material particle is larger and the density is poor when the volume content of CaF_2@$Al(OH)_3$ is 5%, as shown in Fig.4 (a). The particle distribution of each component of the cutting tool material is uniform and more dense when the volume content of CaF_2@$Al(OH)_3$ is 10%, as shown in Fig.4 (b). Tool material particle is larger and the distribution is uneven when the volume content of CaF_2@$Al(OH)_3$ is 15%, as shown in Fig.4 (c). There is a pore in the fracture surface of the tool material, which is judged to be left after the grain was pulled out. This phenomenon is a typical intergranular fracture. At the same

time, there are some steps in the morphological appearance, which is judged to be formed after the grain fracture. This phenomenon is a typical transgranular fracture. Therefore, mixed transgranular fracture and intergranular fracture is fracture modes for the self-lubricating ceramic cutting tool materials. This model is helpful to improve the strength of the cutting tool materials.

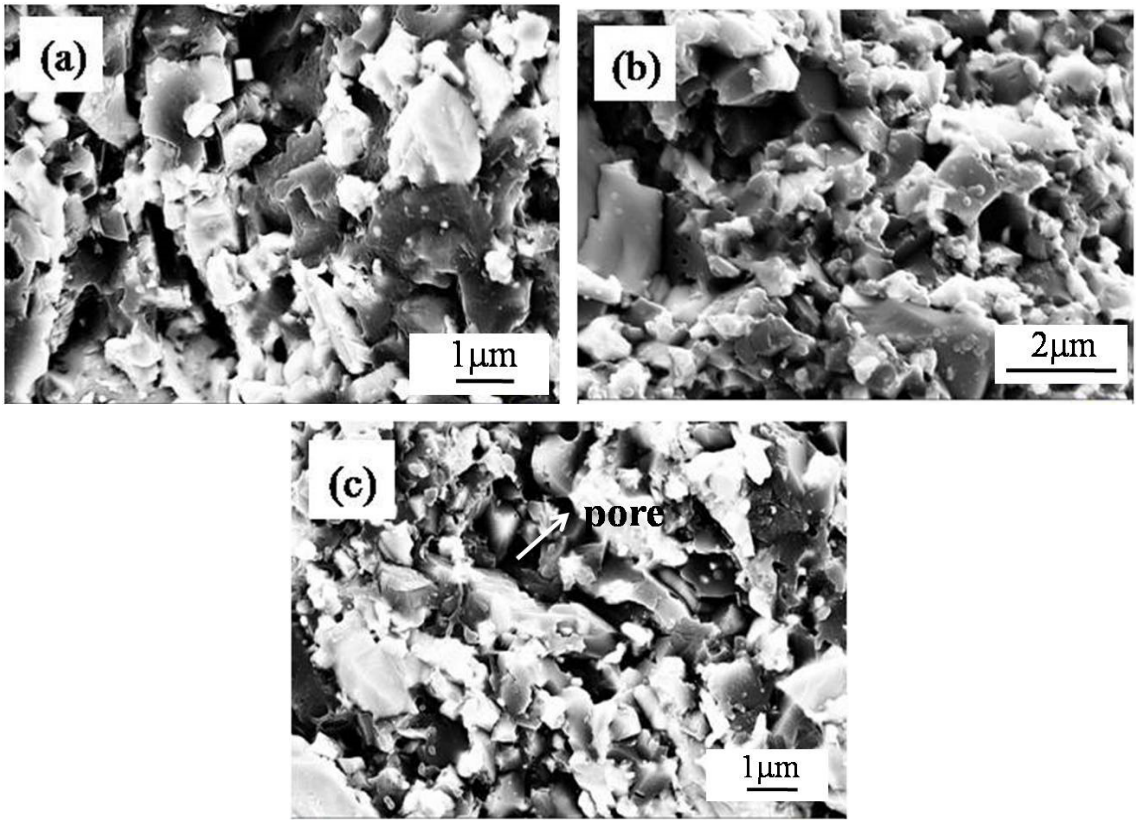


Fig.4. SEM Micrographs of Fracture Surfaces of the Self-lubricating Tool Material with Different Volume Contents of CaF_2@$Al(OH)_3$: (a) 5%; (b) 10%; (c) 15%

3.2 Mechanical Properties of Cutting Tool Materials

Fig.5 (a) shows the effect of CaF_2@$Al(OH)_3$ content on the hardness of tool material. It can be seen that with the increase of the content of CaF_2@$Al(OH)_3$, the hardness of the cutting tool material decreases gradually. The hardness of the tool material is 16.98 GPa when the volume content of CaF_2@$Al(OH)_3$ is 5%, while the hardness is 14.67 GPa with the addition of 15%, declined 15.7%, as shown in Fig.5 (a). With the increase of the content of CaF_2@$Al(OH)_3$, the fracture toughness of the cutting tool material increases first and then decreases as shown in Fig.5 (b). When the volume content is 10%, the fracture toughness is 4.12 $MPa \cdot m^{1/2}$. The flexural

strength values show a similar tendency as the fracture toughness, which can be seen in Fig.5 (c) that the flexural strength is 607 MPa when the volume content is 10%.

In summary, the best mechanical properties of the tool material can be achieved when the volume content of CaF_2@$Al(OH)_3$ is 10%, which the hardness, fracture toughness and flexural strength are 16.03 GPa, 4.12 MPa·$m^{1/2}$ and 607 MPa, respectively.

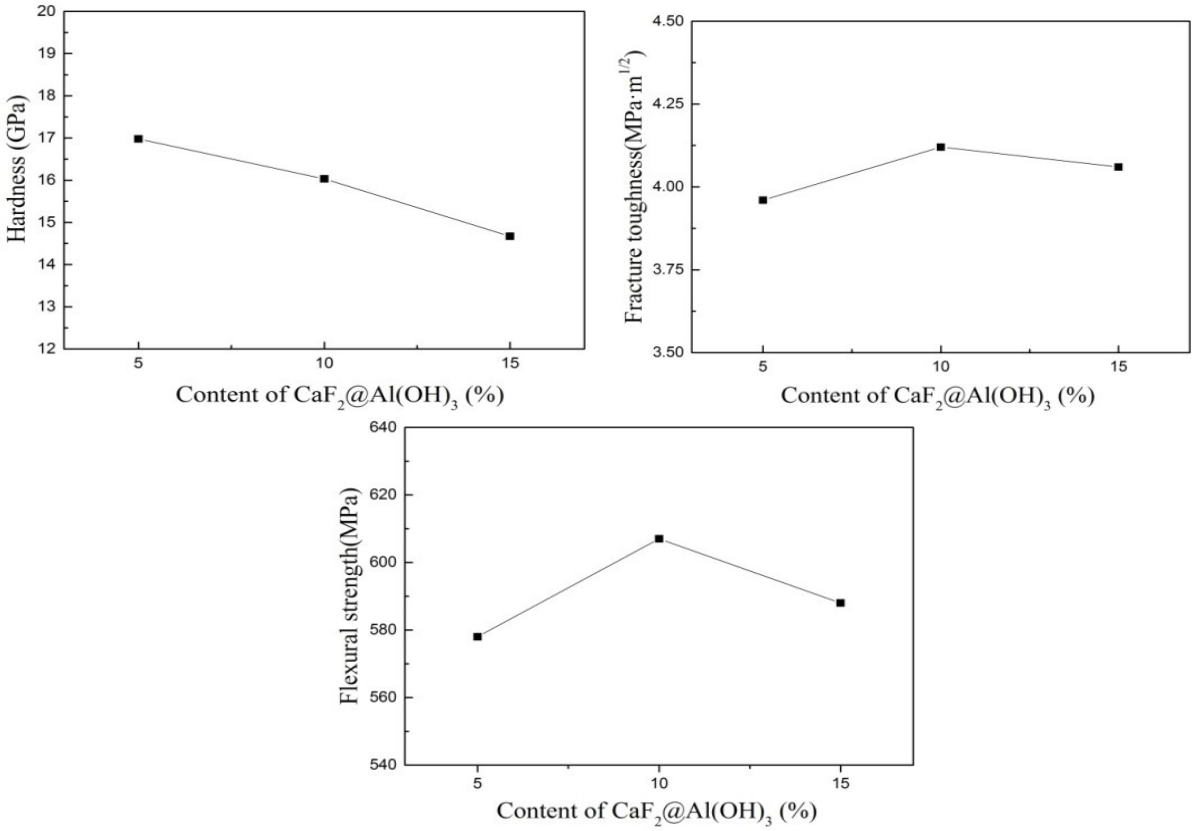


Fig.5. The Effect of CaF_2@$Al(OH)_3$ Content on Mechanical Properties of Tool Materials

Mechanical properties of the self-lubricating ceramic cutting tool materials with the addition of $Al(OH)_3$ coated CaF_2 and uncoated CaF_2 are listed in Table 2 for comparison. The hardness, fracture toughness and flexural strength of the Al_2O_3/(W,Ti)C/(CaF_2@$Al(OH)_3$) material were increased by about 21.7%, 8% and 10.7%, respectively, as compared with the corresponding uncoated CaF_2 material. The similar results can be obtained according to other studies. Chen et al. [13] prepared Al_2O_3/TiC(h-BN@SiC) self-lubricating ceramic tool materials. When h-BN@SiC powders was 5 vol.%, the tool material's hardness and fracture toughness are 12.8% and 44.4% higher than that of Al_2O_3/TiC/h-BN tool material.

Tab. 2. Mechanical Properties of the Ceramic with Coated CaF_2 Powders Compared with Uncoated CaF_2 Powders

Property index	Adding coated CaF_2 powders	Adding uncoated CaF_2 powders	Increment (%)
Hardness (GPa)	16.03	13.17	21.7
Fracture toughness ($MPa \cdot m^{1/2}$)	4.12	3.81	8
Flexural strength (MPa)	607	549	10.7

3.3 Cutting Performances of Cutting Tool

Fig.6 shows the relationship between cutting tool life and cutting speed when cutting 45# steel. The depth of cut and feed rate are constant, and the standard of tool blunt VB is 0.3mm. With the increase of cutting speed, tool life increases first and then decreases as shown in Fig.6. Because the solid lubricant cannot be dragged to form lubricating film when the cutting speed is lower (v=60m/min), it is bad for tool cutting. The result is the tool wear becomes faster and the tool life becomes shorter. When the cutting speed is higher (v=80m/min), solid lubricant is more conducive to form lubricating film. It can effectively improve the wear resistance of the tool and improve the tool life.

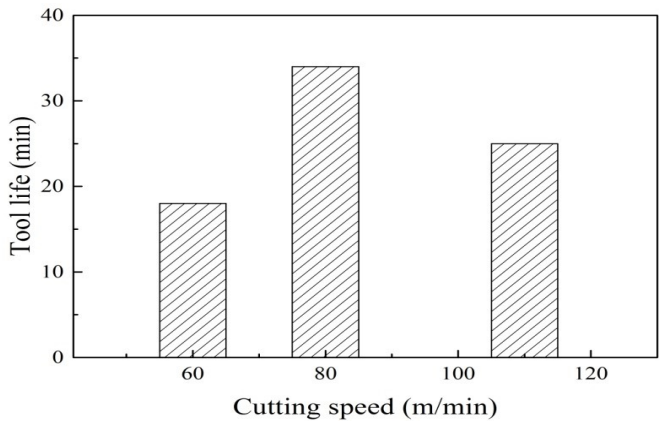


Fig.6 The Relationship between Tool Life and Cutting Speed

Fig.7 (a) presents SEM micrographs of rake face wear of self-lubricating tool when cutting 45# steel. It is obviously seen that a furrow appeared on the rake face, which belongs to abrasive wear. But the wear area is small, which indicates that the tool has better wear resistance and adding coated solid lubricant to tool material can improve the cutting performances. Fig.7 (b) presents SEM micrographs of flank wear of self-lubricating tool when cutting 45# steel. It can be seen that the area of mechanical ploughing wear appeared on the flank face and the wear mechanism is abrasive wear.

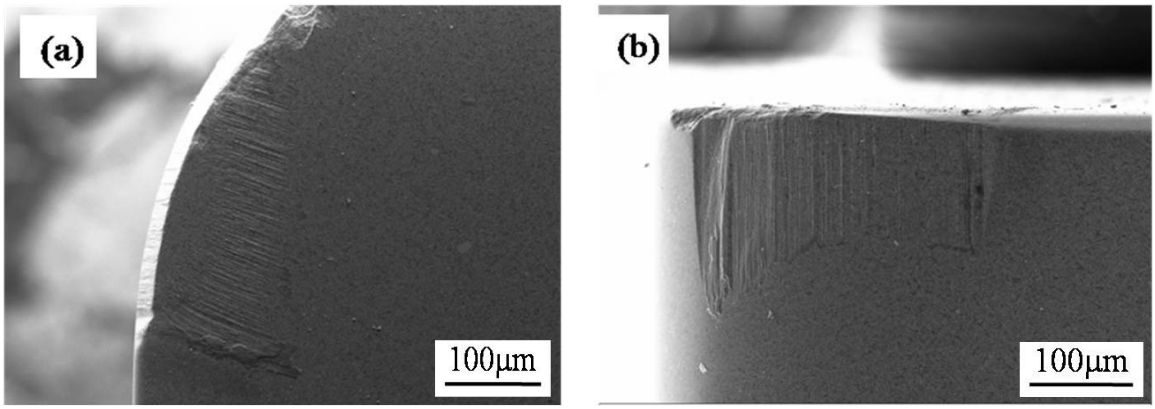
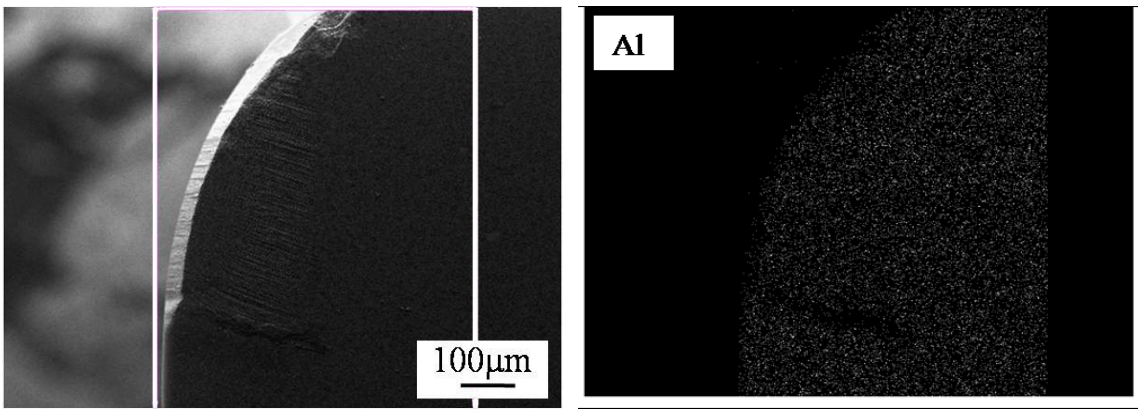


Fig.7. SEM Micrographs of Cutting Tool Wear: (a) Rake Face Wear; (b) Flank Face Wear

Fig.8 presents EDS spectrum analysis of the elemental distribution of the self-lubricating tool rake face and the elements of Al, Fe and Ca are listed respectively. It can be seen that there are a large amount of Fe elements in the wear area of the tool surface, which is caused by iron bonding with the tool surface. The distribution of Al and Ca elements and solid lubricant is homogeneous, which can be in favor to form a uniform lubricating film on rake face in cutting process.



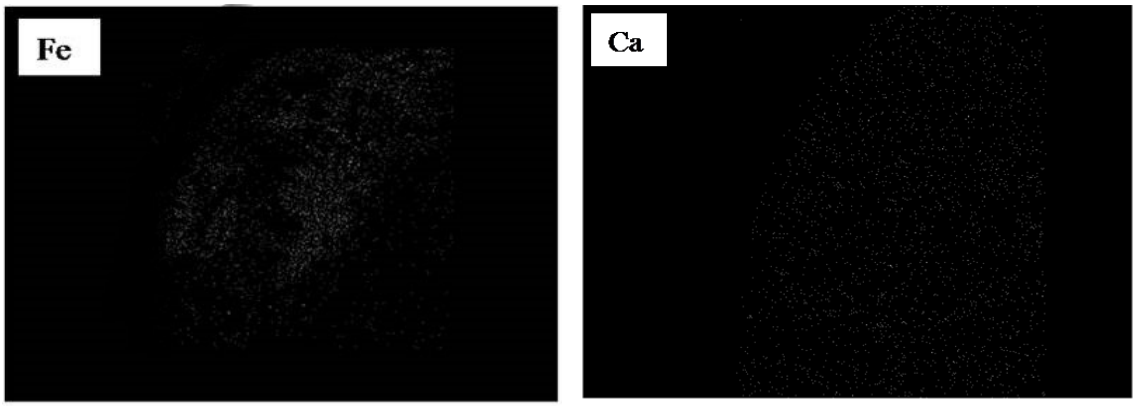


Fig.8. EDS Image of Element Distribution of Tool Rake Face

Conclusions

A type of self-lubricating ceramic cutting tool material was developed by adding $Al(OH)_3$ coated CaF_2 powders. The main conclusions of this study are as follows:

(1) $Al(OH)_3$ coated CaF_2 powders were prepared successfully and the mechanical properties of the tool material can be improved by adding moderate $Al(OH)_3$ coated CaF_2 powders. The best mechanical properties can be obtained when the volume content of CaF_2@$Al(OH)_3$ is 10%, the hardness, fracture toughness and flexural strength are 16.03 GPa, 4.12 $MPa \cdot m^{1/2}$ and 607 MPa, respectively.

(2) The microstructure of cutting the tool material was observed and analyzed by SEM. The result indicates that the fracture mode of the self-lubricating tool is intergranular fracture and transgranular fracture.

(3) Cutting test shows that the Al_2O_3/(W,Ti)C self-lubricating tool with $Al(OH)_3$ coated CaF_2 powders has good wear resistance and antifriction property.

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