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Research article

Recovery and reutilization of high-quality boron carbide from sapphire wafer grinding-waste

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ABSTRACT

Considerable amounts of high-quality boron carbide (B_4C) are discarded as J240 sapphire-wafer grinding waste (J240-W), which can be mostly recovered and reutilized after purification for environmental protection. This paper has developed a feasibility method that simultaneously removes the alumina (Al_2O_3) and iron (Fe) impurities from J240-W with microwave-assisted acid leaching strategy. The influence factors on the Al_2O_3 leaching ratio, such as leaching temperature, sulfuric acid concentration, liquid-solid ratio and time, have been investigated and optimized. For comparison, the leaching of Al_2O_3 with conventional and ultrasound-assisted methods has also been performed. The result indicates that the Al_2O_3 leaching ratio at 80 °C with microwave assistance is 68.95%, much higher than that of conventional (23.66%) and ultrasound (53.13%) methods. Attributed to the unique heating mode of microwave, the Al_2O_3 leaching ratio can rise to 95.28% at optimum condition, while the content of purified B_4C can reach to 98.22% with the residual Al_2O_3 and Fe fall to 0.26% and 0.12%, respectively. The recovered B_4C with high purity and suitable particle size can be reutilized to manufacture W5 abrasive and W0.5 armor material, which is beneficial for environmental protection and resources reutilization.

1. Introduction

Boron carbide (B_4C) has received extensive attention in various fields due to its superior performances, such as superior hardness (Vickers hardness of 3770 kg/mm^2), low density (2.52 g/cm^3), high melting point (2450 °C), excellent wear resistance and large neutron capturing cross section (Li et al., 2010; Suri et al., 2010; Alizadeh et al., 2013). Therefore, B_4C is a good candidate for abrasive material, lightweight bulletproof armor, cutting tool, wear-resistant component, nuclear controller and shielding material (Çinar et al., 2002; Zorzia et al., 2005; Domnich et al., 2011). The demand of B_4C powder is increasing promptly due to its superior properties, for instance, the size of B_4C market is nearly 8430 tons in 2017, while the value is estimated to be 12000 tons in 2020 (Boron carbide powder report, 2017). In particular, demands of B_4C powder as abrasives for grinding of sapphire wafer (Xing, 2014) and armor material for military bulletproof armor (Savio and Madhu, 2017) are increasing rapidly in recent decades.

Noteworthily, the demand of sapphire wafer (LED substrate) is increasing promptly with the fast growth of LED industry, which promotes the wide application of B_4C abrasives in grinding process of sapphire wafer (Tang et al., 2013; Nie et al., 2018; Zhang et al., 2018). For example, B_4C powder with size J240 B_4C (30–120 μm, D50 = 60 μm) and W5 B_4C (2.0–8.0 μm, D50 = 3.5 μm) have been used as ideal abrasives for coarsely and finely grinding of sapphire wafer, respectively (Xing, 2014; Dong, 2017; Xie et al., 2017). The J240 and W5 abrasives are expensive, high-value products, of which the prices are 22 $/kg and 32 $/kg, respectively (Table 1).

In addition, due to its extreme hardness and low density, B_4C powder with size W0.5 (0.2–2.2 μm, D50 = 0.8 μm) can be applied as raw material for the preparation of light weight bulletproof armor (Wang, 2008; Hayun, 2017). The price of the W0.5 B_4C armor material is 40 $/kg, much higher than that of B_4C abrasives (Table 1). It is obvious that the value of product highly increases with the decrease of size due to the cost of producing smaller particles.

However, the B_4C production process is a high-energy consumption, high-cost and high-pollution though B_4C is widely applied as abrasives and armor material. Commercially, B4C chunk is produced by carbothermic method according to the reaction: $4H_3BO_3 + 7C = B_4C +$

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Table 1
Detail information for different kinds of B_4C product.

Application field	Classification	Particle size/μm			Price/($/kg)	Specification/%		
		D03	D50	D94		B_4C	Al_2O_3	Fe
B_4C raw material	chunk	–	–	–	10	≥95%	< 0.5%	< 0.3%
Abrasives for sapphire	J240	30	60	120	22	≥96%	< 0.5%	< 0.3%
	W5	2.0	3.5	8.0	32	≥96%	< 0.5%	< 0.3%
Armor material	W0.5	0.2	0.8	2.2	40	≥97%	< 0.3%	< 0.2%

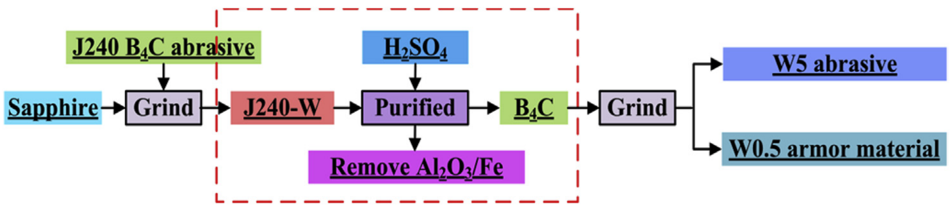

Fig. 1. Schematic flow chart for the recovering and reutilizing process of B_4C from J240-W.

6CO (Alizadeh et al., 2013), which exhibits that 1 kg B_4C chunk comes from 6 kg raw material and only 40% of the chunk is high-quality for the abrasives and armor material. Besides, the B_4C chunk ($\Phi = 1$ m) needs to be intensively crushed, milled and acid leached to manufacture the B_4C powder of desired particle size. All these complex steps result in the low yield and then a large supply demand gap of high-quality B_4C powder (Suri et al., 2010). Hence, the reuse of rejected B_4C may be an effective solution to alleviate the shortage of B_4C powder (Yan et al., 2016). For instance, as shown in Fig. 1, it is promising that B_4C with larger particle size (D50 = 20–30 μm, 25 $/kg) can be recovered from J240-W (0.5 $/kg) after purification, and then reutilized to manufacture W5 abrasive (2.0–8.0 μm, 32 $/kg) and W0.5 armor material (0.2–2.2 μm, 40 $/kg) due to their high value, huge demand and also the manufacture process has the advantages of short process flow, high efficiency and low cost.

The J240-W mainly consists of nearly 90% B_4C, together with alumina (α-Al_2O_3) and metallic iron (Fe) as main impurities, which are generated during the coarsely grinding process of sapphire wafer. The dropping of Al_2O_3 scraps and tiny Fe fragments both reduce the grinding efficiency of J240 B_4C abrasive, which is then eventually discarded as J240-W (Wen et al., 2009; Ebina et al., 2012). It is estimated that approximate 5000 tons J240-W is produced in 2017, and the size will increase to 6000 tons in 2018 (Sapphire wafer waste report, 2017). However, little attention has been paid to the purification of J240-W and then the recovery of B_4C, which results in the continuous environmental pollution and serious waste of resources. Herewith, it is imperative to find a feasible, cost-efficient method to purify the J240-W and then recycle the high-quality B_4C powder.

In order to recycle the B_4C powder, the J240-W needs to be firstly purified, namely removing Al_2O_3 and Fe impurities completely. Due to its low price (0.065 $/kg) and low-pollution, sulfuric acid (H_2SO_4) has been selected to simultaneously remove Al_2O_3 and Fe impurities by acid leaching method. In this work, the acid leaching of Al_2O_3 with conventional, ultrasound-assisted and microwave-assisted methods has been performed and compared. B_4C powder has been obtained from J240-W after purification with microwave assistance, while the influence factors on Al_2O_3 leaching have also been optimized. In addition, the purified J240-W has been well characterized by scanning electron microscope (SEM) analysis, particle size analysis and B_4C content analysis. The result shows that the recovered B_4C, for its high purity (98.22%) and proper particle size (7.62–62.25 μm), can be reutilized to manufacture W5 abrasive (2.0–8.0 μm) and W0.5 armor material (0.2–2.2 μm).

2. Experimental

2.1. Materials

The J240-W was dried and then characterized with various analytical methods. The chemical composition of J240-W was analyzed and listed in Table 2. The B_4C content in the J240-W is 89.32% together with 5.51% Al_2O_3 impurity and 3.31% Fe impurity. In order to recycle the B_4C powder, Al_2O_3 and Fe impurities need to be simultaneously removed with acid leaching method.

Table 3 showed the particle size distribution of J240-W characterized by laser particle size analyzer. It depicts that the J240-W particle distributes in the range of 2.47–56.25 μm with D50 at 18.35 μm, less than the particle size of J240 abrasive (30–120 μm, D50 = 60 μm). It is evident that most B_4C abrasives are not broken during the grinding process and can be recycled to manufacture W5 abrasive (2.0–8.0 μm) and W0.5 armor material (0.2–2.2 μm).

The crystal structure of the J240-W was conducted by XRD (MPDDY2094, Netherlands). As shown in Fig. 2, the main crystal phases presented in the J240-W are well-crystallized B_4C phase and α-Al_2O_3 impurity. While Fe impurity is undetected due to the content being below the detection limit of conventional XRD. The removal method of Fe impurity is simple and mature in industry (Li et al., 2017). However, α-Al_2O_3 can hardly be removed with common regular method due to its stable and dense crystal structure (Lin et al., 2004). Hence, the focus of this paper is to investigate an efficient and low-cost removal method of α-Al_2O_3 impurity.

The morphology of the J240-W was characterized by SEM (S-4300, Rill, Japan) equipped with energy spectrum analysis (EDS) and the results were shown in Fig. 3. Fig. 3 (a) shows that there coexist irregular particles larger than 10 μm together with fine particles less than 5 μm. Fig. 3 (b) indicates that the fine particles mainly consist of B_4C particles together with Al_2O_3 and Fe as impurities. Fig. 3 (c) illustrates that the large particles are B_4C particles with trace amount of Al_2O_3 impurity. The results obviously show that fine Al_2O_3 and Fe impurities are always tightly adhered on the surface of large B_4C particles, which makes them impossible to be completely separated from B_4C through traditional mechanical separation methods, such as centrifugal separation or hydrocyclone separation.

Table 2
Chemical composition analysis of J240-W.

Constituent	B_4C	Al_2O_3	Fe	Others
Content/%	89.32	5.51	3.31	1.86