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Effect of CuO–TiO_2–SiO_2 additions on the sintering behavior and mechanical properties of corundum abrasive synthesized through sol–gel method

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Abstract

The effect of ternary compound additives, CuO–TiO_2–SiO_2 on the phase transformation, densification and microstructure of corundum abrasive were investigated. The results showed that the co-doping had synergistic effects on reducing the phase transformation temperature from γ- to α-Al_2O_3, enhancing the densification and refining the microstructure of corundum abrasive particles. With the addition of CuO, TiO_2 and SiO_2 fixed at 3 wt% of the total weight of abrasive and in the molar ratio of 7:7:5, the samples sintered at 1400 °C for 0.5 h reached about 99.3% of theoretical density. The abrasives exhibited a homogeneous microstructure and excellent mechanical properties with single particle compressive strength higher than 40 N in all doped samples. The influence of each additive and their combined effects on the sintering of abrasives were also discussed in detail.

Keywords: A. Sol–gel processes; B. X-ray methods; C. Strength; Abrasive

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1. Introduction

Corundum abrasive has attracted great interest in recent years for its high hardness, good wear-resistance, high strength and long service life [1–3]. The intrinsic property of corundum is the high ionic bond strength between Al^{3+} and O^{2-}, which unfortunately results in low sinterability. In general, the sintering temperature of pure alumina is as high as 1700 °C, and abnormal grain growth is easy to occur under this temperature, which will impair the mechanical properties of alumina. These disadvantages have limited its applications to some extent. It has been confirmed that the incorporation of a small amount of additives is an effective way to lower the sintering temperature and improve the properties of alumina ceramics. The influence of sintering additives on the properties of alumina has been studied extensively, including MgO, CaO, BaO, TiO_2, CuO, La_2O_3, Nd_2O_3 and MnO_2, etc. [4–8]. These additives accelerate sintering process either by generation of solid solutions and lattice defects, or by the formation of a liquid phase, both favoring the atom diffusion which is vital to sintering. In some previous studies, CuO and TiO_2 had been co-incorporated to investigate their combined effects on the sintering of alumina. For example, Sathiyakumar and Gnanam [9] obtained 99.6% TD at a considerably lower level of 1200 °C by introducing CuO and TiO_2 into alumina. A low eutectic temperature is likely to exist in the additive system containing CuO and TiO_2, for the eutectic of Al_2O_3–CuO–Cu_2O system has been known to be 1096 °C. In addition, the co-dope of Ti^{4+} and Cu^{2+} could promote the solubility of each other due to mutual charge compensation, which may be beneficial to enhancing the sintering kinetics. Another well-known compound additive, MgO–CaO–SiO_2, is often employed as liquid phase to obtain uniform microstructure with fine grains at a low sintering temperature. Moreover, it has been verified that the addition of MgO can roughen the grain boundaries in the

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alumina doped with SiO_2 and CaO thus converting abnormal grain growth into normal growth [10].

As one of the most widely used doping elements in alumina, titanium has a controversial effect on the sintering. On the one hand, it could enhance the rate of densification; on the other hand, it is easy to induce anisotropic grain growth. But anisotropic grain growth cannot be observed when TiO_2 additions is up to 2 wt% [11]. Song and Coble attributed the development of anisotropic grains to the presence of specific concentrations of silica and either CaO, Na_2O, SrO or BaO [12]. Although it causes exaggerated grain growth, it cannot be overlooked that the contribution of TiO_2 on the densification of alumina.

Dopants forming liquid phases (e.g. in the alumina–calcia–silica system) have achieved considerable importance because they have a low eutectics that can generate liquid phases at the temperature of 1200–1300 °C, inducing an accelerated densification of alumina. During cooling, these liquids form films at both the grain boundaries and grain surface to act as a strengthen phase. Dusan Galusek et al. [13] found that there was a certain amount of intergranular silicate films in the magnesium silicate-doped alumina. These films not only strengthen grain boundary and reduce microcrack propagation rate but also provide a higher overall compliance and capacity to retard microcrack initiation.

As far as we know, no attempt has been made to investigate the effect of $CuO–TiO_2–SiO_2$ on the fabrication of corundum abrasive. On the basis of the general effects of additives mentioned above, a simple design with ternary compound additives $CuO–TiO_2–SiO_2$ were conducted in the present work to investigate the synergistic effect of $CuO–TiO_2–SiO_2$ on the sintering behavior and microstructural evolution in corundum abrasive. The relationship between microstructure and mechanical properties was also discussed.

2. Experimental procedure

The corundum abrasive was prepared by sol–gel process with industrial aluminum sulfate ($Al_2(SO_4)_3 \cdot 18H_2O$) as raw materials. The processing flow chart of producing precursor is shown in Fig. 1. The raw materials were dissolved in distilled water completely and filtered to attain a solution with 0.3 mol/L Al^{3+}. Then the diluted $NH_3 \cdot H_2O$ with a concentration of 1 mol/L was added into the Al^{3+} solution at a constant drop speed to adjust the value of pH in the range of 9–10 at the end of reaction. Then the solution was stirred under magnetic stirring at room temperature for 2 h to get clear sol. After filtration, the obtained gel was washed three times with deionized water to remove the residual ions, such as SO_4^{2-} and NH_4^+. Additives ($CuO–TiO_2–SiO_2$) were introduced into the gel in the form of copper nitrate $Cu(NO_3)_2 \cdot 3H_2O$ (AnalaR grade, Kewei Ltd., Tianjin, China), tetrabutyl titanate $Ti[O(CH_2)_3CH_3]_4$ (AnalaR grade, Kewei Ltd., Tianjin, China) and Tetraethylorthosilicate (TEOS) (AnalaR grade, Kewei Ltd., Tianjin, China). The total amount of additive components was adjusted to be equivalent to 3 wt% with $CuO:TiO_2:SiO_2$ molar ratio of x:y:5 (x varies between 1 and 7 and y ranges from 5 to 7). These additive components are listed in Table 1. After wet-milled in deionized water for 10 h with high-purity alumina media, the aqueous solution was dried under 80 °C for 24 h. Then the dried gels were crushed into particles with certain size. The precursor particles were sintered at 1300 °C or 1400 °C in conventional ways at a heating rate of 3 °C/min with a soaking time of 0.5 h at the peak temperature. And the sample abrasive was then obtained. As reference, abrasive without any additives was prepared through the same process.

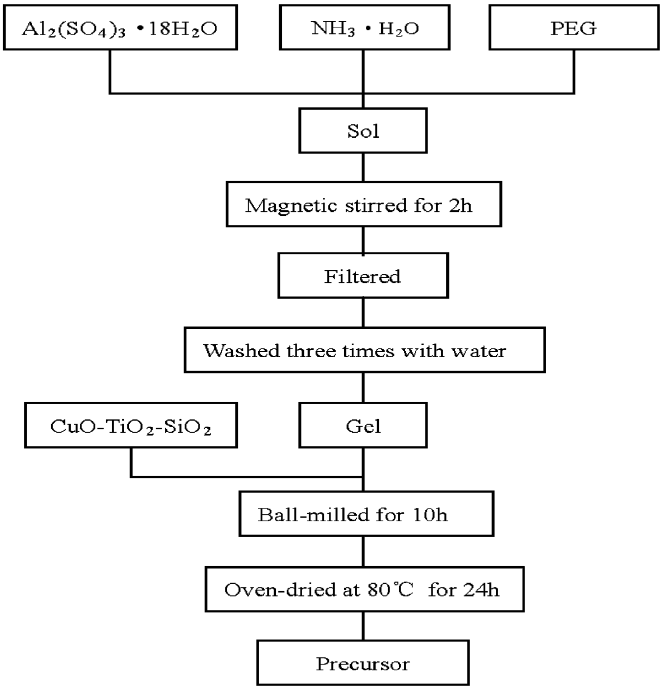

Fig. 1. Schematic flow chart of alumina precursor by sol–gel route.

To investigate the temperature of alumina phase transformation, an X-ray diffractometer(XRD, PHILIPS X'Pert-MPD System) using CuKa radiation at a scan rate of 8°/min was used to identify the phases after the thermal treat of gels at different temperatures for 1 h.

The strength of abrasive was measured by using a single particle compressive strength tester (ZMC-II,China) after sieved by 60/80 mesh screen. For each sample, 40 particles were selected randomly from the abrasives and tested by strength tester one by one. An average strength was reported. Density was determined by the Archimede's method in deionized water. The microstructure of sintered abrasive particles was investigated by field emission scanning electron microscopy (FESEM, JEOL JSM-6700F, Japan). The average grain size was determined by using the linear intercept method from SEM micrographs.

3. Results and discussion

The relative density and average grain size of the corundum abrasive as a function of sintering temperature are

Table 1
Relative density and average grain size of different samples.

Designation of sintered sample	Additive composition ($CuO:TiO_2:SiO_2$)	Sintering temperature (°C)	Relative density (%)	Average grain size (μm)
S01	no adding	1300	95.2	–
S02	no adding	1400	97.5	0.50
S11	1:6:5	1300	98.5	0.52
S12	1:6:5	1400	98.9	0.75
S21	7:7:5	1300	99.0	0.47
S22	7:7:5	1400	99.3	0.49
S31	5:7:5	1300	98.7	0.49
S32	5:7:5	1400	99.1	0.51
S41	7:6:5	1300	98.0	0.48
S42	7:6:5	1400	98.3	0.50

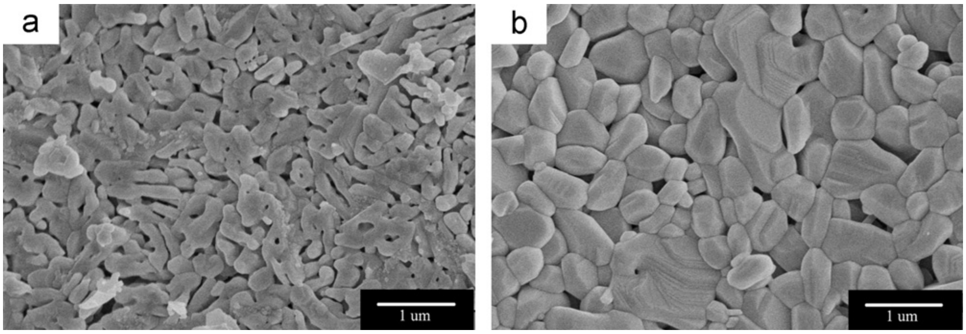

Fig. 2. SEM micrographs of the undoped samples sintered at (a) 1300 °C and (b) 1400 °C for 0.5 h.

shown in Table 1. It can be seen that the average grain size increased with the increase of temperature. The relative density of doped samples in our works were all above 98%, and it increased slightly as the sintering temperature changed from 1300 °C to 1400 °C. This phenomenon was attributed to the different grain growth that was remarkably influenced by the heating temperature. As the temperature rose up, more grains were activated to grow up and the pores were more easily removed. Consequently the samples obtained under 1400 °C exhibited a higher relative density and coarser grain size than the samples sintered at 1300 °C.

Compared with co-doped samples, however, undoped samples exhibited a relative low density under the same temperatures. This shows that the combined addition has a favorable effect in the densification process.

Figs. 2 and 3 demonstrate the SEM morphologies of corundum abrasive samples sintered at different temperatures without and with additives, respectively. As seen from the microstructures, all doped samples mainly consisted of equiaxed grains and presented a denser structure compared to the undoped ones. It was clear that the specimens with the same additive composition exhibited different grain sizes due to the difference of calcination temperature. For example, sample S11 was composed of grains with an average grain size of 0.52 μm which was smaller than the grain size in sample S12 (0.75 μm). The same phenomenon was found in other samples, which could be ascribed to the accelerated ionic mobility under higher temperature. Obvious microstructural distinctions were observed between doped and undoped samples under the same sintering conditions. Sample S01 (Fig. 2a) and S02 (Fig. 2b) appeared more porous than other samples. The grains in S01 had not grown adequately under the sintering temperatures employed in this study as they appeared vermicular which caused negative effect on the strength of abrasives. When the reference samples were sintered at 1400 °C, the grains obviously tend to be equiaxed although the porosity remained higher than doped samples.

The average grain size increased as the molar ratio of TiO_2 in the additives increased, as indicated in Table 1 and Fig. 3. The TiO_2 addition is known to extensively promote grain growth because it can be easily dissolved into the alumina matrix to form the lattice defects, which is believed to accelerate sintering rate by enhanced vacancy diffusion mechanisms. In general, doping elements have a solubility limit in alumina and segregate to grain boundaries (GBs) or precipitate when the GBs are saturated, a phenomenon that could inhibit the grain growth. The solubility of TiO_2 in Al_2O_3 was reported to vary from 800 at. ppm to 0.27 wt% at the temperature between 1250 and 1700 °C [14,15]. But the highest TiO_2 addition (S11) in our works was only 0.0165 wt%, a level far below the solubility limit, so there was no extra TiO_2 to precipitate at the grain boundaries. Under a low temperature at the beginning of sintering, Ti^{4+} dissolved into the alumina

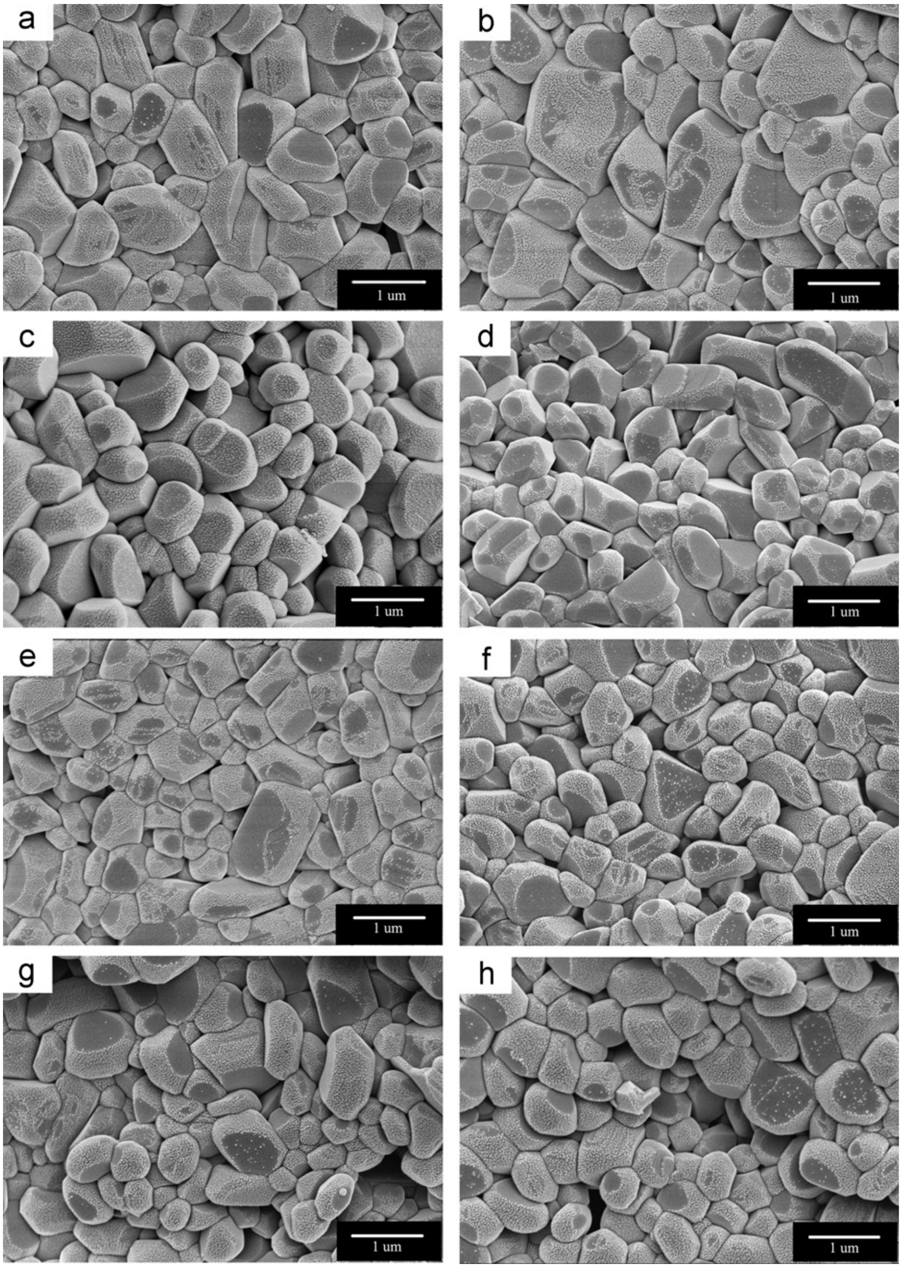

Fig. 3. SEM micrographs of the doped samples with different molar ratios: (a) S11; (b) S12; (c) S21; (d) S22; (e) S31; (f) S32; (g) S41 and (h) S42.

matrix to form the lattice defects to accelerate the sintering rate. As the temperature increased, the TiO_2 in alumina was prone to generate liquid phase, so the sintering-aid effect would mostly be achieved through a liquid-phase mechanism, especially at temperature above 1300 °C. Therefore it is possible that TiO_2 formed liquid phase to promote sintering when the heating temperature exceeded 1300 °C in present study. As a result, the grains in S11 (Fig. 3a) were coarser than grains in S41 (Fig. 3g), which was similar to the situation between S12 (Fig. 3b) and S42 (Fig. 3h).

Compared with TiO_2, the solubility limit of SiO_2 in alumina is as low as 300 ppm [16]. SiO_2 mainly forms intergranular liquid phases at the presence of other oxides under a high temperature that could enhance the mass transfer during the sintering of alumina by liquid phase sintering mechanism. After cooling, the silicates are prone to precipitate at the grain boundaries to form thin films. It has been proved that these amorphous silicate films could remarkably strengthen the grain boundary, poison the grain growth even at a very low concentration, and obtain a finer microstructure. But excess of liquid between the grains could also induce abnormal grain growth, thus jeopardize the mechanical properties of abrasive. And superfluous liquid phase also traps pores in and between grains that resulted in deteriorating the densification of the samples. In this respect, the abnormal grain growth occurred in the sample S11 and S12 (Fig. 3a and b) might be due to the relative high amount of SiO_2 compared to other samples.

Anisotropic grain growth is considered to be disadvantageous to the properties of abrasive. There is an incubation period before the anisotropic grain growth initiates. And the nucleation of anisotropic grains was observed to occur once a small amount of grains had reached a critical size while the other grains did not appear to be significantly coarse. As was observed in some previous studies, anisotropic grain growth occurred when TiO_2 and SiO_2 were added simultaneously. This was due to the nonuniform distribution of titanium-containing aluminosilicate glassy phase, which only wrapped the basal planes of platelets whereas the edges of platelets and boundaries displayed no amorphous interlayer [17]. Generally, the equilibrium phase diagrams could offer some information about sintering process. However, the quaternary system $CuO–TiO_2–SiO_2–Al_2O_3$ has not been well established up to now. Examining the equilibrium phase diagrams of $Al_2O_3–TiO_2–SiO_2$ and $Cu_2O–CuO–Al_2O_3$ systems revealed that the lowest eutectic temperatures were 1480 °C and 1130 °C, respectively. Therefore a lower eutectic temperature is likely to exist in the system consisted of Al_2O_3, TiO_2, SiO_2 and CuO. Considering the sintering temperature employed in our studies was as high as 1400 °C, it was very likely for the additives to generate liquid phase during heating. Meanwhile, the properties of liquid phase can be influenced by additives. Note that there were no obvious anisotropic grains in all doped samples, as illustrated in Fig. 3. In the view of the above, we presume that the introduction of CuO in the $TiO_2–SiO_2$ system could modify the distribution of liquid phase and improve the homogeneity so that the grains grow uniformly and appear equiaxed.

As clearly seen from Fig. 3(b), abnormal grain growth (AGG) occurred in sample S12, which was doped with molar ratio 1:6:5 and sintered at 1400 °C. Although doped with the same composition, S11 exhibited a microstructure obviously different from S12. A uniform grain size distribution was achieved in sample S11 in spite of few relatively large grains embedded in fine-grained matrix whereas exaggerated grains with 0.8 μm in size could be found messily distributed in 0.4 μm fine-equiaxed grains matrix in S12. Usually, AGG is accompanied by pores entrapment within the grains. These pores could only be removed from the sintered body through the volume diffusion mechanism which demands substantial amount of energy. A new theory named 2D nucleation attributes the occurrence of AGG to the formation of small amounts of liquid phase and distributes nonuniformly in final stage sintering [18]. Considering the relatively high amount of TiO_2 and SiO_2 doped in sample S12 and the temperature employed, it was easy to generate certain amount of liquid phase during the sintering. These liquids were expected to play positive roles in densification at first, since they provided an additional diffusion mechanism of dissolution–precipitation and capillary forces for sintering. With the increasing temperature, the amount of liquids increased excessively and promoted the grain growth extensively. As the mass transferred too fast, the pores were easily entrapped and could not be eliminated effectively. As a result, the grains grew abnormally and the microstructure became porous, which deteriorated the properties of abrasive in further.

Relative density of all samples at different sintering temperatures is presented in Fig. 4. Despite the undoped samples, the density of doped ones increased firstly and then fell as the amount of CuO in the additives increased. Given the melting point of CuO is as low as 1026 °C, it is easy for CuO to produce liquid during the heating treatment and promotes the sintering of alumina by liquid phase sintering mechanism. However, there is a limit of the liquid content essential for accelerating the densification, once exceeded, the excessive liquid phase can cause too fast mass transfer and induce abnormal grain growth, what is disastrous to the microstructure and properties of abrasive. At the same time, the exclusion of pores in the grains is retarded by the abundant liquid between grains. Note that many pores were located both on grain boundaries and within the grains in S41 and S42, which contained the highest amount of CuO in this study, as presented in Table 1 and Fig. 3.

Fig. 5 shows the single particle compressive strength of abrasive particle samples sintered at different temperatures. The compressive strengths of the doped samples were significantly higher than that of the undoped samples at the same temperature. The undoped samples exhibited minimal compressive strengths of 18.2 N and 19.2 N at 1300 °C and 1400 °C respectively. The samples sintered at 1400 °C enjoyed a higher strength compared to that fabricated at 1300 °C. For example, the same amount of additives was added to the samples prepared, the samples sintered at 1400 °C S12 and S22 had strengths of 60.9 N and 69.9 N, compared to the samples sintered at 1300 °C S11 and S21, with values of 49.2 N and 58.8 N,

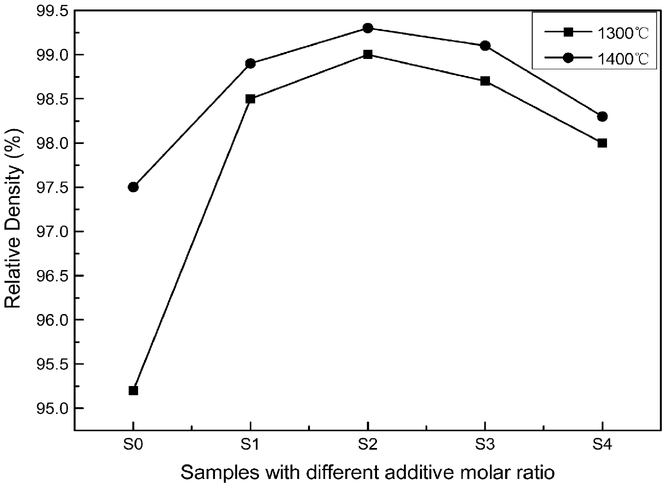

Fig. 4. Relative densities of samples with different additives molar ratios sintered at 1300 °C and 1400 °C for 0.5 h.

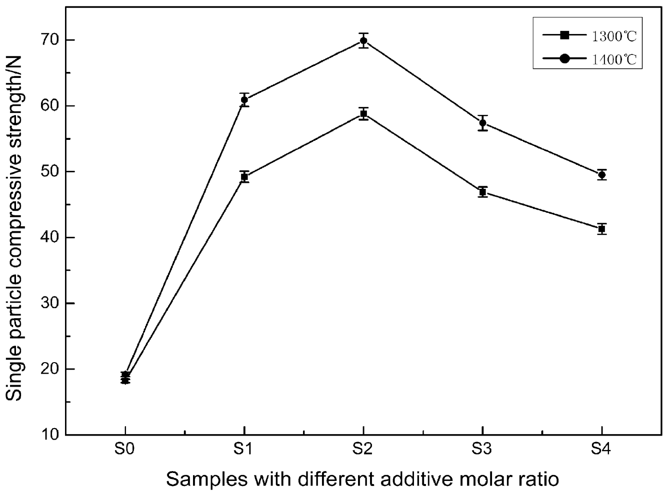

Fig. 5. The single particle compressive strength of the abrasive samples with various additives sintered at different temperatures for 0.5 h.

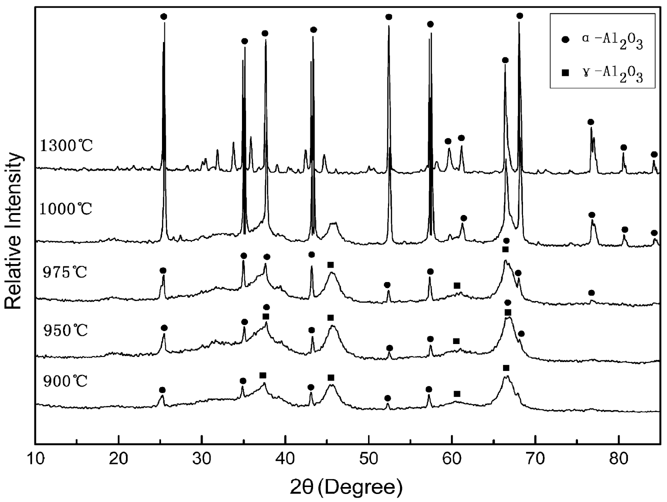

Fig. 6. XRD patterns of the samples with molar ratio (7:7:5) sintered at 900, 950, 975, 1000 and 1300 °C.

respectively. The difference appears to be a result of enhancing sintering of a higher temperature. Although increasing the sintering temperature induces a further grain growth, which is detrimental to the mechanical properties of sintered abrasive, on the other hand, it promotes the densification and brings about a denser microstructure. The combined effect in our research was an enhancement of abrasive particle strength. When the temperature stayed constant, the single particle compressive strength exhibited an increase at first and then declined as a function of different molar ratio in the additives, a similar trend to the relative density is presented in Fig. 4. From the figure, samples with CuO–TiO_2–SiO_2 molar ratio 7:7:5 (S21 and S22) demonstrated a relative highest strength of 58.8 N and 69.9 N sintered at 1300 °C and 1400 °C, respectively. Considering the finer microstructure and smaller grain size in S21 and S22 (Fig. 3c and d) compared to other samples under the same temperature, it was an evidence that under the similar heating conditions alumina abrasive particles with the smaller mean grain size, narrower grain size distribution and regularly equiaxed crystal shape exhibited higher mechanical properties. Therefore, we can conclude that the synergistic effect of CuO–TiO_2–SiO_2 achieves the maximization at a molar ratio of 7:7:5.

To investigate the effect of CuO–TiO_2–SiO_2 system on the transformation temperature of metastable alumina to α-alumina, XRD was performed at different temperatures with the fixed molar ratio of 7:7:5. The results are presented in Fig. 6. Note that the dried gel calcined at 900 °C had partly converted to α-Al_2O_3, although the main phase remained γ-Al_2O_3. The degree of crystallinity of α-Al_2O_3 was improved with increasing temperature. Only a small amount of γ-Al_2O_3 had been detected after calcined at 975 °C, coexisting with the major phase α-Al_2O_3. The complete transformation of γ-Al_2O_3 to α-Al_2O_3 took place at 1000 °C, about 250 °C lower than the alumina sintered without additives as reported previously [19]. This indicated that CuO–TiO_2–SiO_2 proved to be effective in enhancing the transformation of transition alumina. The reasons come from various aspects. It has been known that the transformation of boehmite to α-Al_2O_3 experiences a successive transformation between a series of metastable alumina phase, which was described by Baca et al. [20] as a topotactic sequence. The transition from γ- to α-Al_2O_3 is reported to be a nucleation and growth type of reaction. And the process is a reconstructive transformation where cubic close packing oxygen ions rearrange into a hexagonal close packed structure to form thermally stable α-Al_2O_3 [21]. For it includes the movement of oxygen ions with a relatively big ionic radius, a remarkable amount of energy is necessary. Without any additives, this transformation would not take place until the temperature is increased to 1250 °C, as proposed in previous literatures. It is confirmed that the temperature of γ-Al_2O_3 to α-Al_2O_3 transition can be influenced by some cation additives and transition metals and Ti^{4+} has been reported to promote the transformation [22]. The additives employed in this study, such as Ti^{4+} and Cu^{2+}, are easy to dissolve into the alumina lattice and increase oxygen vacancies, thus enhance the ion diffusion and accelerate the alumina transformation. For the electrovalence of Ti^{4+} and Cu^{2+}, one is higher and the other is lower than Al^{3+}, they could increase the solubility of each other due to mutual charge compensation, leading to a further promotion in ion diffusion. Moreover, ball milling with pure corundum mediums also have a positive effect on the phase transformation, because during milling, the alumina hydroxide in the powder can be transformed into a single phase of α-Al_2O_3 at a temperature of about 900 °C and the wear debris from milling mediums could provide nucleation sites for the dry gel [23].

Since it is very difficult to generate new crystalline phase in a matrix which differs largely from the new one in crystal structure, the nucleation energy expended in the transformation from γ-Al_2O_3 to α-Al_2O_3 is considerably high. When a certain amount of second phase existed, the first stage of phase transformation would be encouraged through heterogeneous nucleation mechanism. It was notable that $CuAl_2O_4$ phase which had a spinel structure was suggested to occur before α-alumina formation during the calcinations [24]. This spinel phase acted as nucleation sites for the γ–α-alumina transformation and led to an increase in the transformation kinetics and a significant reduction of the transformation temperature.

4. Conclusions

During the sintering of alumina abrasive, the co-doping of $CuO–TiO_2–SiO_2$ played a positive role in the phase transformation, densification and microstructure evolution. The doped samples exhibited a denser and uniform microstructure than undoped ones. With the total weight of addition fixed at 3 wt% of the abrasives, approximately 99% and 99.3% of TD were obtained in samples that was co-doped with CuO, TiO_2 and SiO_2 at molar ratio of 7:7:5 when sintered at 1300 °C and 1400 °C, respectively. The temperature of phase transformation from γ- to α-alumina was decreased in contrast to pure alumina by 250 °C to about 1000 °C. All the doped samples displayed excellent mechanical properties with single particle compressive strength higher than 40 N and the highest strength was 69.9 N sintered at 1400 °C with the molar ratio of 7:7:5. The ternary compound additives facilitated sintering process through solid solution at low temperature and formed liquids to further promote sintering at a relatively high temperature. In addition, excessive amount of liquid phase may induce abnormal grain growth which deteriorates the microstructure and mechanical properties of abrasives.

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