Milling and Sintering Behaviors of the Metal-ceramic Mixed Powders

Z.X.Yang[1], S.H.Youn[1], K.H.Hwang[1,a], S.Y.Yoon[2,b], J.K.Lee[3,c], and H.Kim[4,d]

[1]Eng. Res. Inst., GyeongSang Nat'l Univ., Jinju city, S.Korea
[2]Pusan Nat'l Univ., [3]Chosun Univ., [4]Seoul Nat'l Univ., S.Korea
[a]khhwang@gsnu.ac.kr, [b]syy3@pusan.ac.kr, [c] jklee@chosun.ac.kr, [d]hwan94@snu.ac.kr

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There have been great attentions on enhancing the applicability of engineering ceramics, such as alumina or zirconia which have high strength and hardness with high chemical stabilities, by fabricating more precisely and cheaply. Preparing the ceramics outstanding powders with good sinterability is a key requirement. But for good sinterability, powders must be almost spherical, be finer than 1㎛, have narrow particle size distributions, and have no agglomeration so that chemical routes for powder synthesis can be used rather than cheap mechanical grinding method. Large sized alumina or zirconia powders are advanced in terms of price but they need to improve their sinterability. Recently, near net shape forming by reaction sintering have been applied to SiC and Si_3N_4 ceramics with hard sinterability. As pore removal by reaction and sintering between particles occurred simultaneously, large size powders need to be used and the shrinkage should be compensated so that precise forming is possible. By oxidation of Al to Al_2O_3, the volume would be expanded by about 28.6%. But it was impossible to form dense body only by full oxidation of pure Al powders due to the low melting point of Al and dense surface oxidation coatings, which required ceramic powders being mixed with Al powders. Moreover, fine Al powder is very caustic in that inert coarse Al powders must be used as raw material.

Two types of Al_2O_3 powders, hard agglomerated coarse alumina (Alcoa, A-10, 12.8μm) and soft agglomerated fine alumina (Sumitomo, AES-11C, 0.4μm) were used. And two types of Al powders, globular shape Al (Changsung, AAl-140, 24.7μm) and flaky shape Al (Yakuri, 17.9μm) were used as well. A volume ratio of Al_2O_3 to Al powders of 65:35 were mixed and comminuted in attrition mill with acetone and zirconia ball. A planetary ball mill was also used to compare the grinding efficiency. After milling particle size distribution and BET, the specific surface areas of mixed powders were measured.

After granulation of pulverized powder, granules were uniaxially pressed into rectangular bars (6mm×6mm×50mm) at 10Kg/cm^2 and then isostatically cold-pressed at 400MPa for 1 min. The green compacts were kept at 400°C-1400°C for 8 hours to oxidize Al, and then sintered at 1450°C to 1650°C for 3 hours in air. The heating rate was 5°C/min and then furnace cooled. The shrinkage and bulk density were measured using Arkimedes method and their sintered microstructures were also observed by SEM.

In the case of coarse alumina powders(A-10), strong agglomeration due to high calcination temperature was observed. But by attrition milling, it pulverized very easily

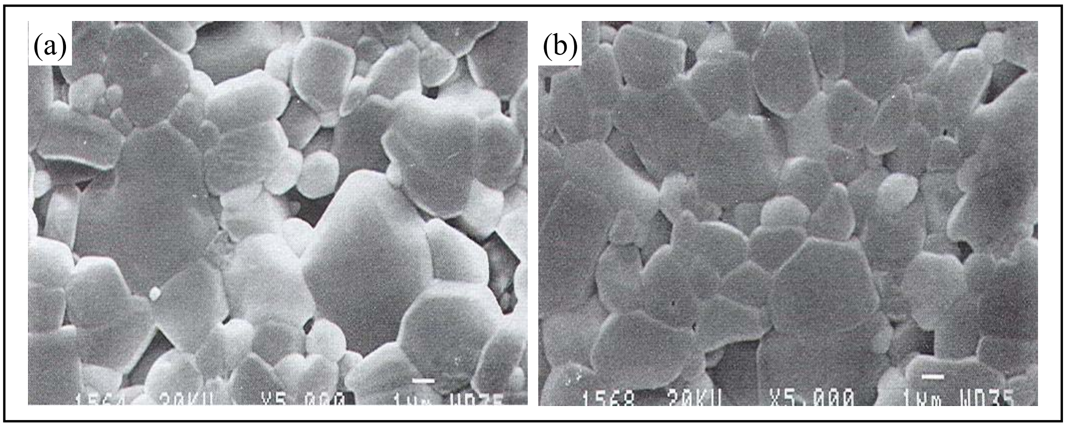

**Fig. 1. The SEM micrographs of reaction sintered Al_2O_3 fired at 1550℃ for 3hs.
(a) Coarse alumina(A-10) with 35vol.% globular Al, and (b) with flaky Al.**

to primary particles such that after 10 hours of attrition milling, it was found to become finer than the originally fine alumina powders (AES-11C). This means that attrition milling could be very effective for grinding strongly agglomerated coarse powders, and globular Al powders could be pulverized relatively easily by attrition milling despite of ductility, but flaky Al could not be grinded easily due to thin plate shape. However, when flaky Al powders were mixed with brittle Al_2O_3 powders, there was little difference in the degree of milling compared with globular Al. Also, in the case of planetary ball milling, the pulverization did not occur even after 1 hour elapsed.

The relative densities of reaction sintered Al_2O_3 ceramics increased by the milling time due to pulverization. But as shown in Fig.1 (a), by using the flaky Al, the densification could be increased much more than globular Al because the thin plate of pulverized flaky Al could be surrounded Al_2O_3 powders more homogeneously. So, the microstructures of reaction sintered Al_2O_3 ceramics using flaky Al showed higher densification. Furthermore, in the case of reaction sintering, the bulk density of fine Al_2O_3 (AES-11C) powder compacts showed lower values than the coarse (A-10) powder compacts, as shown in Fig.1 (b). This is probably due to their low grinding efficiency.

So the conclusion can be made that

1) Powder mixtures of flaky shape Al with coarse alumina was much more effectively comminuted by the attrition milling than the mixtures of globular shape Al. Coarse alumina was much more useful than fine alumina in pulverizing and grinding the ductile Al particles.

2) Since the mixed powder of coarse alumina with flaky Al was much more effectively comminuted than the globular Al, a sintered body of more than 97% theoretical density was achieved, but the low Al contents led to relatively higher shrinkage of about 8%.

3) Since the coarse alumina particles were much more useful in cutting and pulverizing the ductile Al particles, the coarse alumina powder was also useful in reaction-sintering.

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