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Effect of Composition and Sintering Temperature on The Microstructure and Theoretical Density of TiO_2 Stabilized Al_2O_3 Cutting Tool.

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Abstract

Cutting tool samples of three different compositions such as 2% TiO_2 -, 5% TiO_2 - and 8% TiO_2 stabilized Al_2O_3 were made by pressing the prepared powder at a pressure of 175-180 MPa and sintered at three different temperatures of 1350^{o}C, 1400^{o}C and 1450^{o}C for 2 hours. The microstructure of the sintered cutting tool samples were studied by scanning electron microscope. The theoretical densities of the prepared cutting tool samples were also studied. It was found that as sintering temperature increases grain size increases for all three compositions of Al_2O_3 cutting tools but with higher % of TiO_2 the extent of grain coarsening decreases. With increasing sintering temperature and higher % TiO_2 content high degree of densification has been achieved. The maximum % theoretical density of 95.18% has been obtained at 1450^{o}C for 8% TiO_2 stabilized alumina cutting tool.

Keywords: *TiO_2, Nano-scale Al_2O_3, Microstructure, Pinning effect, Ceramic tool materials.*

1. Introduction

Alumina base ceramic cutting tool is one of the most wide used cutting tool [1] materials in the industrial levels. The reason behind is, it contains good physical properties such as high hardness [2] and good wear resistance [3]. It also contains good chemical properties such as high melting point and excellent chemical stability. They are more stable than high-speed steels and carbides, thus having fewer tendencies to adhere to metals during machining and less tendency to form built-up edge. This results in good surface finish and dimensional accuracy in machining steels.

These properties of alumina materials are mainly related to the density and microstructures. Highly dense and fine grained structures provide the best mechanical properties [4]. Ceramic cutting tools should have % theoretical density more than 90% for good performance. In order to achieve high density, alumina has to be sintered over 1600-1700^{o}C. But highly dense and fine grain microstructure can also be achieved if alumina is stabilized with other materials such as TiC, ZrO_2, TiB_2, SiC, NbC etc in lower temperature than conventional sintering temperature. Likewise this, an attempt has been made to produce Al_2O_3 ceramic cutting tool of highly dense and fine grained microstructure, stabilizing it with different composition of TiO_2 at lower temperatures.

2. Experimental

Samples were prepared from commercial alpha Al_2O_3 powder [~40nm; purity: 99.87%] mixed with 2, 5 & 8 weight% TiO_2 [~40nm; purity: 99.9%] by the conventional ceramic processing technique. Powders were at first weighted in electronic weighted balance. Raw materials were then ball-milled at 150 rpm for 18~20 hours in acetone medium in presence of zirconia balls. After proper mixing, extraction of the powder was done with the help of acetone. The wet powder was kept in the oven at 110-120^{o}C for 24 hours to dry out. A binder was made with PVA (Poly vinyl alcohol) and water (10gm in 100 ml) in 100^{0}C with continuous stirring. After drying out of the raw powder mix, 5~8% PVA binder was mixed with the powder before compaction. Cutting tool samples were prepared using the pressure of 175-180MPa and 2 minutes holding, using a uniaxial hydraulic or mechanical press. The prepared samples were around 13 mm in diameter and 2 mm thick. These green samples were again dried for one day at 110^{0}C. After that, the samples were taken for sintering. Sintering was done in a single stage sintering process. Samples were placed in the furnace and the temperature was raised very slowly (3^{o}C/min) in order to avoid crack formation. Binder was removed by holding the samples at 500^{o}C for 1 hr and then temperature was again raised slowly to the desired sintering temperature and held at that temperature for 2 hours for all three composition of Al_2O_3- TiO_2. For every composition of Al_2O_3 and TiO_2 (2, 5 and 8%) sintering were done for three different temperatures respectively (1350^{0}C, 1400^{0}C and 1450^{o}C). Finally upon completion of sintering process, percent theoretical densities were calculated. It was done after the measurement of the density with precision electronic balance and slide calipers. Microstructure observation along with grain sizes measurement was also done using SEM (Scanning Electron Microscope).

3. Result and Discussions

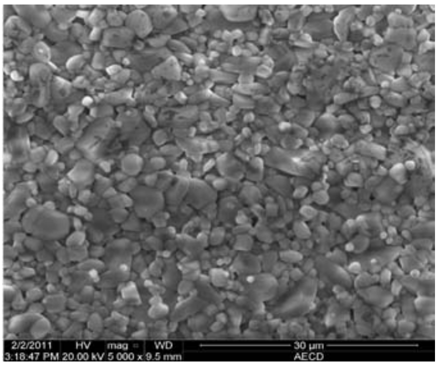


Fig.1. SEM of 2% TiO_2-98% Al_2O_3 sintered at 1350^{0}C (5000X)

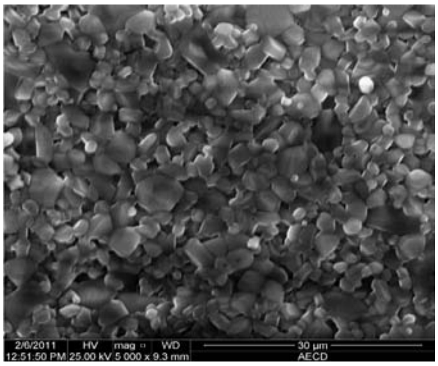


Fig.2. SEM of 2% TiO_2-98% Al_2O_3 sintered at 1400^{0}C (5000X)

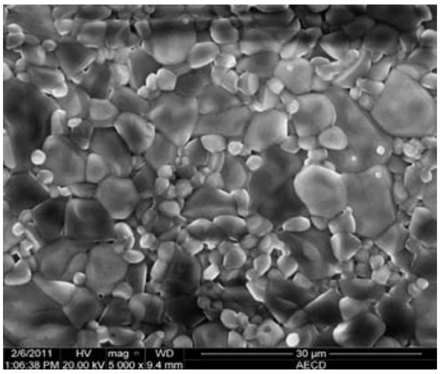


Fig.3. SEM of 2% TiO_2-98% Al_2O_3 sintered at 1450^{0}C (5000 X)

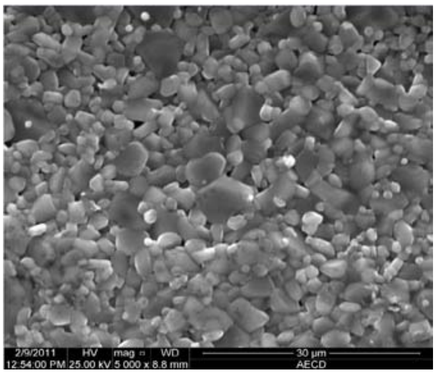
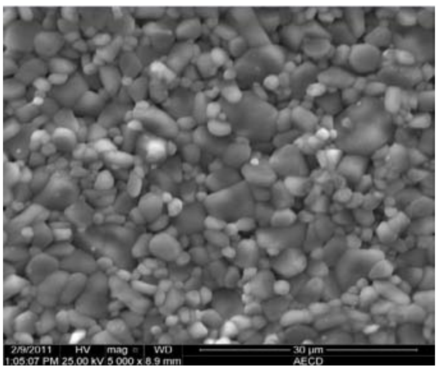


Fig.4. SEM of 5% TiO_2-95% Al_2O_3 sintered at 1350^{0}C (5000 X)



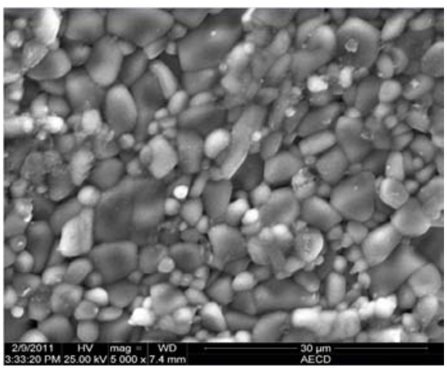
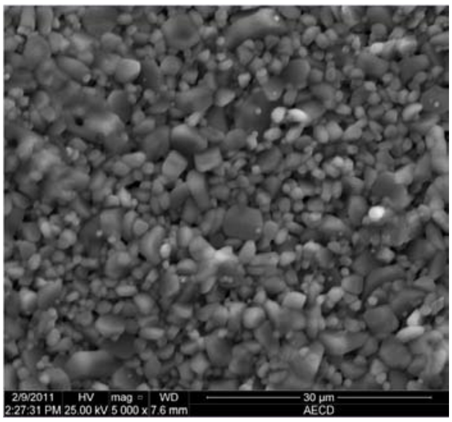


Fig.5. SEM of 5% TiO_2-95% Al_2O_3 sintered at 1400^{0}C (5000X)

Fig.6. SEM of 5% TiO_2-95% Al_2O_3 sintered at 1450^{0}C (5000X)



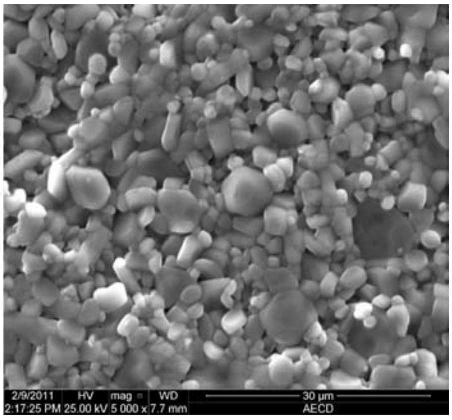
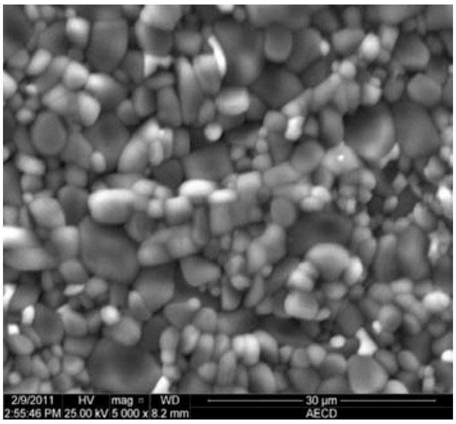


Fig.7. SEM of 8% TiO_2-92% Al_2O_3 sintered at 1350^{0}C (5000X)

Fig.8. SEM of 8% TiO_2-92% Al_2O_3 sintered at 1400^{0}C (5000X)



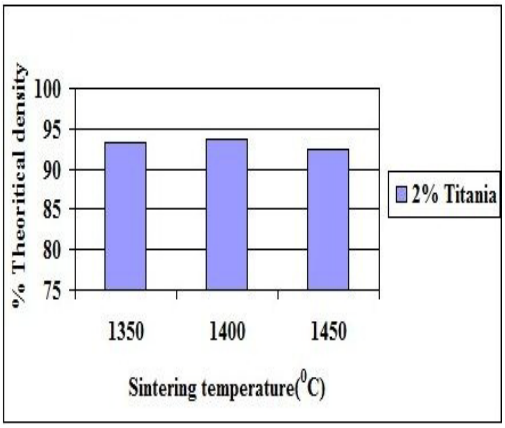
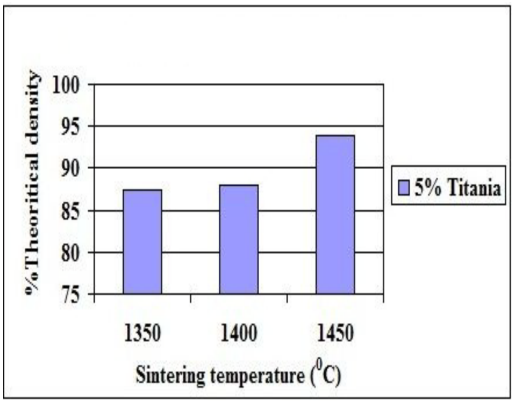


Fig.9. SEM of 8% TiO_2-92% Al_2O_3 sintered at 1450^{0}C (5000X)

Fig.10. Sintering temperature vs. % theoretical density of 2% TiO_2 stabilized Al_2O_3



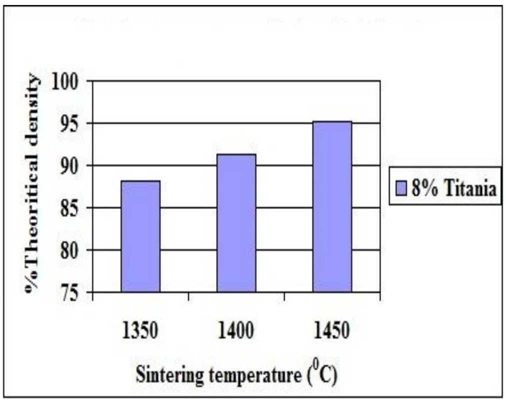


Fig.11. Sintering temperature vs. % theoretical density of 5% TiO_2 stabilized Al_2O_3

Fig.12. Sintering temperature vs. % theoretical density of 8% TiO_2 stabilized Al_2O_3

From the microstructural study it was found that grain structures of the Al_2O_3 cutting tool samples were different due to the effect of different % TiO_2 and variable sintering temperature. Usually, grain coarsening or exaggerated grain growth occurs in Al_2O_3 with increase in sintering temperature. But in the presence of little amount of TiO_2, sufficient pinning [5] effect can be generated which can successfully inhibit grain coarsening to a useful degree. TiO_2 act as grain growth inhibitor and stops Al_2O_3 grain growth. So increase in %TiO_2 in Al_2O_3 has a prompt effect on Al_2O_3 grain structures. It is quite evident from the microstructures that are shown here. From figures 1-3 it was showed that, with increase in sintering temperature from 1350-1450^{0}C, gradual grain coarsening of Al_2O_3 occurred in a normal way for 2%TiO_2. The grain sizes were 2.1µm, 2.35µm and 3.81µm respectively. So 2% TiO_2 cutting tool samples showed very negligible pinning effect on Al_2O_3. From figures 4-6 it was observed, that the grain sizes of Al_2O_3 were 2.42µm, 2.97µm and 2.98µm for 5% TiO_2 stabilized Al_2O_3. Though the grain coarsening occurred here as well but compared to the grain coarsening of 2% TiO_2, rate of grain coarsening of 5% TiO_2 composition was less and slow. In a similar way, (figures 7-9) for 8% TiO_2 stabilized $Al_2O_{3,}$ the grain sizes of Al_2O_3 were found 2.39µm, 2.82µm and 2.84µm respectively. In this case the grain coarsening rate was much more slower compared to 2% and 5% TiO_2 stabilized Al_2O_3. So very fine grain microstructure was achieved as the % TiO_2 increased up to 8%.
With the increase of % TiO_2 and temperature, higher degree of densification was achieved in Al_2O_3. From figure 10 it was found that at low %TiO_2 (2%), the density at first started to increase then drops at higher temperature. With the increase in temperature though compaction of the TiO_2 - Al_2O_3 samples occurs but due to grain coarsening as it was showed in figures 1-3 the density drops at higher temperature. As percentage of TiO_2 is very small it could not provide sufficient pinning effect during densification process. But it was observed in figures 11 and 12 the density gradually increased as the %TiO_2 increases from 5% to 8% with increasing temperature. TiO_2 provides pinning effect which inhibits grain coarsening of Al_2O_3 at higher sintering temperature and helps to achieve high degree of densification [6-7]. It was seen that 8% TiO_2 stabilized Al_2O_3 cutting tools sintered at 1450^{0}C showed maximum theoretical density of 95.18%.It was also found that from the figures 10-12 an average % theoretical density about 91.5% was achieved.

4. Conclusion

The main purpose of TiO_2 addition in Al_2O_3 ceramic cutting tool is to achieve high degree of densification at relatively lower temperature. The average theoretical density for all the composition is 91.5% and maximum theoretical density of 95.18% was observed for 8% TiO_2 stabilized Al_2O_3 at 1450^{0}C temperature. The grain coarsening inhibition and densification were occurred due to the pining effect of TiO_2 in Al_2O_3. So with the addition of the higher percent of TiO_2 in Al_2O_3, highly dense and fine grained Al_2O_3 can be produce at relatively lower temperature.

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