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学位論文題目	Fabrication of Nanocomposites in Alumina-Zirconia System by Using Centrifugal Slip Casting Method （遠心鋳造法によるアルミナージルコニア系ナノ複合材の作製）
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学位論文の内容の要旨

Continued requirements from industry for higher performance materials initiate the endeavors of using nano-particle reinforcement in advanced ceramics. Zirconia toughened alumina ceramics (ZTA) are important ceramic materials. However, using the mechanisms of nano-particle reinforcement has not been proved in this system. The objective of present work is to fabricate nano-composite in Al_2O_3-ZrO_2 system by using the centrifugal slip casting method.

Application of centrifugal method in advanced ceramics processing has been quite recent and more intensive research works are required for wider use of the method. Centrifugal consolidation of two alumina powders, which have distinct characteristics with respect to particle size, size distribution, purity, etc., was studied, to get more general information concerning rheology-consolidation relations. Present work clearly revealed that the characteristics of the starting powders have significant effects on the consolidation behaviors in centrifugal consolidation. The coarse powder showed a larger maximum packing density than that of a powder with finer particle size, higher purity and narrow particle size distribution. With the increase of the solid loadings, the packing density of the fine powder continually increased, whereas that of the coarse powder increased to a maximum value at 56 vol% solid content and then decreased. It is necessary to find an optimum solid concentration with respect to maximum packing density, avoidance of segregation and easiness for processing. Mass segregation was avoided in 56 and 44 vol% suspensions for the coarse and fine powders, respectively. Density gradient along the height of the green compacts linearly decreased from the bottom to the top of the cakes, with the fine powder showing smaller density gradient. The slight density gradient along the height of the consolidated cakes was found to be inherent for centrifugal slip casting and the magnitude of which varied with the starting powders. The effects of the slight density

gradient were found to be insignificant for most ceramics processing.

Available works mainly concentrated on centrifugal slip casting preparation of monolithic ceramics. Present work reports a first attempt in fabricating uniform ZTA ceramics by using the centrifugal slip casting method. Suspensions with 30, 40 and 45 vol% solids loadings were prepared and used to fabricate Al_2O_3-5vol%ZrO_2(3Y-TZP) ceramics. Slight variation in the green density was observed along the height of a specimen as well as between the samples prepared from slurries of different solid content. It was found that the segregation mainly results from the differential settling of aggregates in the starting ZrO_2 powder. If coarse aggregates or undesirable inclusions were absent in the starting powders, slurry of 30~45 vol% solid content would be quite suitable for preparing quite uniform green compacts. Present work proved that centrifugal slip casting is also effective in the fabrication of uniform composite if suspensions of appropriate solids concentration and starting powders of adequate particle size were adopted.

The microstructural evolution and grain growth behaviors for the fine Al_2O_3-ZrO_2 powders were investigated to establish processing conditions leading to nano-sized microstructures for ZTA ceramics. Two kinds of nanocomposites have been fabricated by varying the processing conditions. The nano-nano composites have been obtained for the samples with higher ZrO_2 content sintered at 1400℃ for 1 h. The composites with 5 and 10 vol% ZrO_2 content fired at temperatures over 1550℃ revealed microstructural features of a hybrid-type nanocomposite. It was found that the amount of intragranular particles is firing condition dependent. The zirconia composition for avoiding breakaway grain growth was higher than 5 vo% that suggested in a previous work. Grain growth kinetics for the fine powders could be described using conventional power law grain growth relationship, however, fitting of grain size data to the equation gave higher value of grain-growth exponent.

The concept of nano-FGMs was introduced. Centrifugal slip casting method was applied for the fabrication of alumina-zirconia composites with continuously graded structures. It was possible to tailor the graded structures by varying zirconia powder size and content, for dilute suspensions with a total solids content of 20 vol%. A three-region composition profile was observed for the graded materials, with the middle region showing a nearly linear variation of composition. It is interesting that this region occupied a larger part of the component. Because of the segregation of particles of different sizes, nano-scaled microstructures were obtained in the top layer of the specimen. Bending test showed that the graded composites had higher strength than that of uniform composites. The specimens fractured with the lower zirconia content region being the crack initiate plane showed higher bending strength than that of from higher zirconia content region. Increased fracture toughness could be one of the reasons for the fracture strength improvement. Present work indicated that graded structures in alumina-zirconia system could be positively used to strengthen the composite.

論 文 審 査 の 結 果 の 要 旨

酸化物系の構造用セラミックスの代表例としてはアルミナとジルコニアが挙げられるが、これらをナノ複合化することによりさらなる強靭化が期待されている。しかしながらナノ複合化を実現するためには、微細な初期粉末のみならず焼結性の向上が不可欠である。このためには遠心鋳造法が有効であることが示されているが、複合材では偏析が起こりやすい方法であるので注意を要する。

本論文では、遠心鋳造法によりアルミナージルコニア系の良好なナノ複合セラミックスの作製を目的とし、遠心鋳造法における各種条件と成形体の特性との関係を明らかにしている。

まず､遠心鋳造法において､粒度や異種相の偏析の度合いとスラリー濃度の関係が明らかにされた.本研究で用いた初期粉末はアルミナ微細粒とジルコニア微細粒であるが、ジルコニア粉末の凝集体も含まれている。高濃度のスラリー（40%以上）では粒径・成分・凝集体の偏析は殆ど見られなかったが､30%スラリーではジルコニア凝集体が成形体の最下部に顕著に沈降偏析することが見いだされた。これより、適当な濃度のスラリーを用いることにより、凝集体や他の介在物等を成形体の最下部に沈降させて除去するプロセスも可能であることが指摘されている。

また、沈降法により予め凝集体を除いたジルコニア粉末をアルミナ粉末と混合してスラリーを準備すれば、比較的低温で焼結でき、両相とも微細なナノーナノ複合セラミックスが作製できた。また、同様の粉末で、ジルコニアの量を 5vol%程度に押さえ、高温で焼結すると、ジルコニアの微細粒がアルミナの粒界および粒内に分散したナノ粒子分散型の複合セラミックスが作製できた。

さらに、遠心鋳造法における低濃度スラリーによる偏析を積極的に利用して傾斜材料の作製も試みられている。最上部と最下部を除いた大部分ではジルコニア／アルミナ濃度は直線的に変化し、上部ほどアルミナ濃度が高くなった。この成形体より試験片を採取して曲げ試験を行った結果、アルミナリッチ側が引っ張り荷重を受ける方向で顕著な強度増加がみられたが、逆方向曲げにおいても非傾斜材より若干強度が高くなることが示されている。

これら一連の成果は、遠心鋳造法がナノ複合セラミックスの作製に有効であることを示すものであり、本論文は博士（工学）の学位論文に値するものと認める。