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Suppression of grain growth in sub-micrometer alumina via two-step sintering method

Z. Razavi Hesabi [a,*], M. Haghighatzadeh [a], Mehdi Mazaheri [a,*], Dusan Galusek [b], S.K. Sadrnezhaad [a,c]

[a] *Materials and Energy Research Center (MERC), P.O. Box 14155-4777, Tehran, Iran*
[b] *Vitrum Laugaricio-Joint Glass Centre of IIC SAS, TnU AD and RONA, j.s.c., Trencin, Slovak Republic*
[c] *Center of Excellence for Production of Advanced Materials, Department of Materials Science and Engineering, Sharif University of Technology, P.O. Box 11365-9466, Tehran, Iran*

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Abstract

Two-step sintering (TSS) was applied to suppress the accelerated grain growth of sub-micron (∼150 nm) alumina powder. The application of an optimum TSS regime led to a remarkable decrease of grain size down to ∼500 nm; while the grain size of the full-dense structures produced by conventional sintering ranged between 1 and 2 μm. To find how important the temperatures at sintering steps might be, several TSS regimes were conducted. The results showed that the temperatures at both sintering steps play vital roles in densification and grain growth of the alumina compacts. Based on the results, the optimum regime consisted of heating the green bodies up to 1250 °C (first step) and then holding at 1150 °C for more than 60 h (second step). This yielded the finest microstructure with no deterioration of the densification. Heating at 1300 °C (first step) and then at 1200 °C (second step) was not a successful procedure. Lowering the temperature of the second step down to 1100 °C resulted in exhaustion of the densification at 88% -theoretical density. A nearly full-dense structure with an average grain size of 850 nm was obtained when the temperature of the second step was increased to 1150 °C. Empirical results show that not only the first step temperature has to be high enough to reach a structure containing unstable pores, but the second sintering temperature must also be high enough to activate the densification mechanism without grain growth. This means that a considerable densification at the first step does not imply enough second-step densification.

Keywords: Al_2O_3; Sintering; Grain growth

1. Introduction

Large increase in strength and hardness is frequently documented for ultra-fine ceramic/metallic structures; a phenomenon that is explained in part by small grain sizes using the well-known Hall–Petch relationship.[1–4] Difficulties are associated with producing ultra-fine bulk structures[5–7] due to the fact that fine structures with high interface density must store relatively high energies. As a consequence, grain refining processes have attracted much attention recently.[8,9] To achieve this goal, two principal ways are applied: decreasing the grain size from microscale down to ultra-fine range (<300 nm) and inhibiting the growth of ultra-fine grains during processing. The former has been usually used to produce ultra-fine metallic materials, while the latter has most often been conducted to obtain ultra-fine ceramics. Langdon et al.[10,11] have, for instance, refined the structure of aluminum alloys via severe plastic deformation through equal channel angular pressing (ECAP). Large plasticity of metals and alloys -allows them to be severely deformed, while the brittle nature of ceramics restricts the applicability of this method. In order to obtain an ultra-fine ceramic structure, one can use nanocrystalline powder. Accelerated grain growth during final stage of sintering usually results, however, in coarsening of the structure. Although the higher surface area of powders provides higher driving force and promotes densification leading to a decrease of interfacial energy, it increases their tendency to form agglomerates.[12,13] Dry pressing of the agglomerated powders leads to the formation of a green body with

* Corresponding authors. Tel.: +98 9121365786/+41 21 693 3389; fax: +41 21 693 4470.
E-mail addresses: zohreh.razavi@gmail.com (Z. Razavi Hesabi), mehdi.mazaheri@epfl.ch, mmazaheri@gmail.com (M. Mazaheri).

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