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# Two-step sintering of titania nanoceramics assisted by anatase-to-rutile phase transformation

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A remarkable suppression of grain growth was achieved by taking advantage of the anatase-to-rutile phase transformation during the final stage of a two-step sintering process. The minimum grain size obtainable via two-step sintering of a sample with full-rutile (98%) phase at the end of the first sintering step was around ~250 nm, whereas the anatase-to-rutile transformation at the end of the second step facilitated a reduction in grain size to around 100 nm.


*Keywords:* $TiO_2$; Phase transformation; Two-step sintering; Grain growth

Two-step sintering is a novel technique used to suppress the accelerated grain growth that usually occurs during the final firing stage [1]. High-temperature heating followed by structural freezing via rapid cooling to a constant temperature levels off the grain growth but does not stop the densification. Triple junctions provide drag forces that control grain-boundary migration most effectively [2]. A smaller grain size at the end of the first step thus results in a higher triple junction density that pins the grain boundaries, preventing them from further advancement.

There is, however, a critical density up to which the continued densification generally occurs. No remarkable densification is usually possible below this critical value of compactness [3]. The question arises as to whether this critical value of compactness can be decreased any further, perhaps thereby obtaining a finer grain distribution.

In order to suppress the grain growth course of action, the sintering temperature needs lowering to an extent that no interruption to the densification occurs. Applying pressure can stimulate such a process, assisting the formation of a fully dense structure even at a very low temperature [4]. As well as pressure, an incidental enhancement in atomic mobility can also increase the densification when a phase transformation has to occur due to the inevitable heating up of the sample when sintering takes place [5]. As a consequence, transformation-assisted sintering facilitates the lowering of the sintering temperature with nanophase formation retention and near theoretical-density compaction.

Several researchers have successfully applied the two-step sintering procedure to exhaust grain growth in nanoceramic specimens [1–3,6]. A few others have used transformation-assisted sintering [5,7] with the same purpose. No one has, however, designed any system of significant grain growth suppression based on simultaneous two-step sintering and phase transformation compaction. In the present study, different regimes are envisaged to reveal the role of a combined phase transformation/two-step sintering procedure on the grain growth and microstructural evolution of the titania nanoceramic exploiting the anatase-to-rutile phase transformation.

$TiO_2$ nanopowder (P25, Degussa Co., Frankfurt, Germany) with a particle size ranging from 11 to 27 nm measured via transmission electron microscopy (TEM, CM200 FEG, Philips, The Netherlands) was used. Phase analysis of as-received powder was conducted by an X-ray diffractometer (XRD, Philips X'Pert). The maximum peak intensities in the XRD pattern using Cu K$\alpha$ radiation gave about 77% anatase content and 23% rutile phase. Powder samples were uniaxially pressed under 100 MPa in a rigid die (5 mm diameter). By measuring the weight and dimensions of the pressed powders, a green compactness of ~53 ± 2% of the theoretical-density (TD) was obtained.

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