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New route for processing of multilayer Al₂O₃-Co₃O₄ materials through gelcasting

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ABSTRACT

Multilayer dense ceramic materials composed of Al₂O₃ and Al₂O₃-Co₃O₄ layers have been obtained by gelcasting. The key stage in the process was the optimization of the polymerization idle time in order to ensure strong adhesion between layers without cracks and delamination in a green state and after sintering. The significant advantage of this method is occurrence of strong connections between constituent layers due to the slight migration of the slurry to the gelled bottom part of the sample, what is not observed in techniques based on lamination processes. The multilayer samples were composed of two Al₂O₃ layers and two Al₂O₃-Co₃O₄ layers arranged alternately. The rheological characterization of the slurries was done. The properties of the sintered multilayer bodies were examined in comparison to the single-layer alumina samples. Observations in SEM and light microscope were performed. The presence of the transition layer in the sintered bodies was observed.

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1. Introduction

Multilayer ceramic materials represent a wide range of materials which can be divided into two main groups. In the first one, the multilayered structure composes a thin coating on a substrate, while in the second one, the whole ceramic element is built from constituent layers, and creates the so called sandwich-type material [1,2]. There exist a few methods of the preparation of multilayer coatings on a substrate, for example CVD, PVD vacuum arc method [3], explosive joining [4] or pulsed magnetron sputtering [5]. The main shaping method used in the fabrication of multilayer or sandwich-type materials without supporting substrate is tape casting followed by tapes lamination and/or sintering [6–9]. This technique is used for example in processing of SOFC [10]. Nevertheless, much attention is paid on the development of other techniques such as: co-casting [11], particles sedimentation [12] hot-pressing [13] or combination of two or more shaping methods, for example electrophoretic deposition (EPD) coupled with gelation of polysaccharides [14]. Interesting type of the multilayer structures are functionally gradient materials for bio-medical or piezoelectric applications [14–17]. The preparation of multilayer materials by using in situ polymerization (gelcasting method) has been not reported in the literature so far, however, the combination

of tape casting and gelcasting in the preparation of calcium phosphate scaffolds was described by Sanches-Salcedo and et al. [18]. The application of radical polymerization reaction to consolidate the ceramic suspension allows to obtain homogenous green and sintered ceramic bodies of high mechanical properties and complex geometry [19]. Gelcasting can be easily coupled with other shaping techniques what gives the possibility to obtain a wide range of functional materials [20]. Therefore elaborated and described in the paper route for obtaining of multilayer samples opens up further possibilities of different materials arrangement.

The aim of the research was to elaborate the conditions of the fabrication of multilayer dense ceramics by gelcasting. The samples composed of four layer, as shown in Fig. 1, were prepared. Two Al₂O₃ layers and two Al₂O₃-Co₃O₄ layers have been arranged alternately, the thickness of each layer was 3 mm. The selection of Co₃O₄ as the second oxide in the system has been based on two arguments. The first reason was the use of oxide that could dye the slurries and subsequently the sintered bodies, thus it was possible to observe the connection between the constituent layers macroscopically. The final choice was done on the basis of phase diagrams of Al₂O₃-MeO systems in order to avoid the appearance of any new phases during sintering of the materials. The application of Co₃O₄ covers both these arguments.

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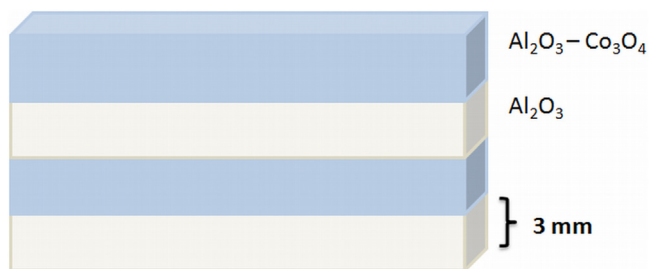


Fig. 1. The scheme of the arrangement of the layers in the multilayer ceramic samples obtained by gelcasting.

2. Experimental

2.1. Materials

The multilayer samples were obtained from α - Al_2O_3 TM-DAR (Taimei Chemicals, Japan) of the average particle size $D_{50} = 116$ nm, a specific surface area of 12.98 m²/g, and density of 3.98 g/cm³. Co_3O_4 (Sigma-Aldrich, 99.5%) of the average particle size $D_{50} = 294.9$ nm and density of 5.74 g/cm³ was used as a dyeing substance in the selected slurries. Both powders were in the form of granules as shown in Fig. 2. In the preparation of samples by gelcasting a few organic additives have been used. Diammonium hydrocitrate, DAC (puriss, POCh, Poland) was used as a dispersant, the organic monomer was glycerol monoacrylate, MG synthesized in the Authors research team. The synthesis and the properties of this monomer have been described elsewhere [21,22]. N,N,N',N' -tetramethylethylenediamine, TEMED (Sigma-Aldrich, 99%) and ammonium persulfate, APS (Sigma-Aldrich, 98%) were used as the activator and the initiator of radical polymerization, respectively. The suspensions were prepared in Milli-Q deionised water.

2.2. Preparation of multilayer samples

Multilayer ceramic samples have been prepared according to the process shown in Fig. 3. The dispersant, the monomer and the activator were added to the solvent (water) and then the alumina powder. The slurries were placed in alumina containers and mixed using a planetary ball mill (PM100, Retsch) for 30 min with a speed

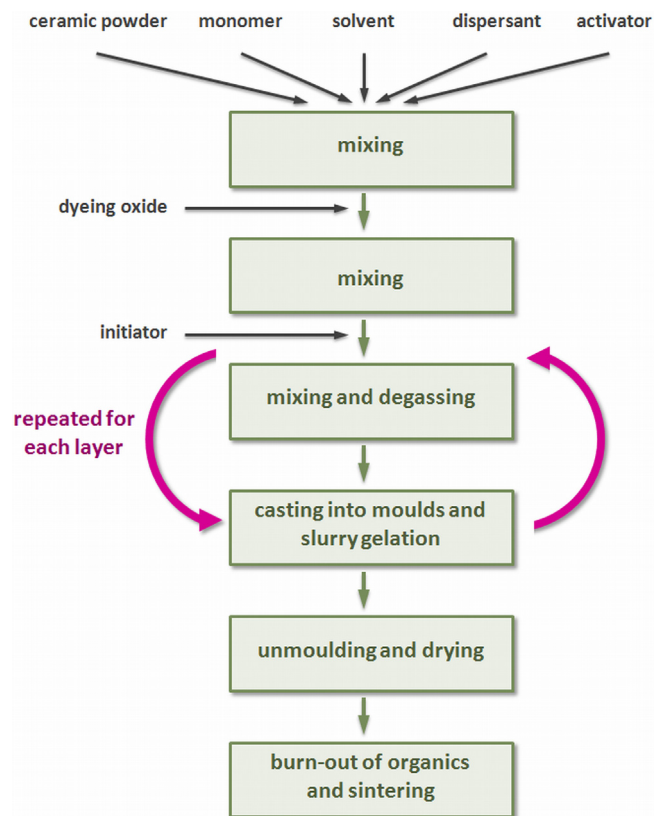


Fig. 3. Scheme of the preparation of multilayer ceramics by gelcasting.

of 300 rpm. In case of selected slurries Co_3O_4 was added as dyeing substance and the suspension was again mixed for 30 min. Slurries were degassed in a Planetary Centrifugal Mixer THINKY ARE-250 for 2 min at a speed of 1800 rpm. Then, the initiator of polymerization was added and the slurry was again mixed in the THINKY ARE-250 for 30 s at a speed of 800 rpm and degassed for 30 s at a speed of 1800 rpm.

At first, the slurry consisting of pure alumina was prepared and casted to the mould. After slurry gelation but prior to its drying the

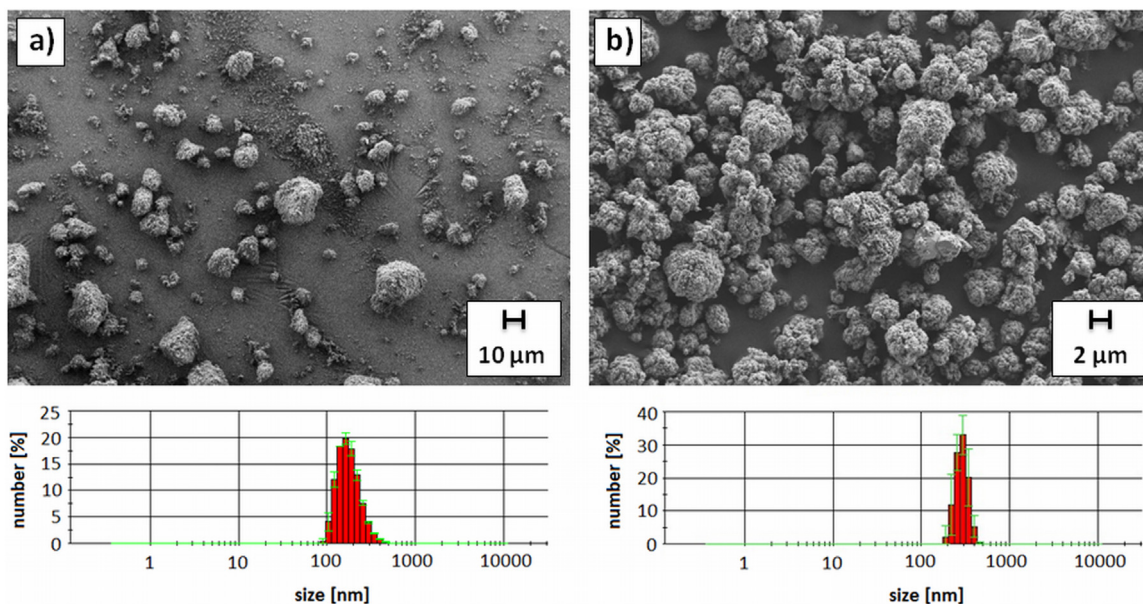


Fig. 2. SEM images and particle size distribution from DLS of Al_2O_3 (a) and Co_3O_4 (b) powders.

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