Fabricating a Novel Intragranular Microstructure for Al$_2$O$_3$/GdAlO$_3$ Ceramic Composites

Shuai Sun $^{1,*}$ and Qiang Xu $^2$

$^1$ Department of Materials, North China University of Technology, Beijing 100144, China
$^2$ National Key Laboratory of Science and Technology on Materials Under Shock and Impact, Beijing Institute of Technology, Beijing 100081, China; 3120100430@bit.edu.cn

* Correspondence: sunshuai@ncut.edu.cn

Received: 3 September 2018; Accepted: 28 September 2018; Published: 1 October 2018

Abstract: In order to make the embryonic form of intragranular structure, the Al$_2$O$_3$/GdAlO$_3$ system was selected due to its excellent mechanical properties. Gd$_2$O$_3$ and Al(NO$_3$)$_3$·9H$_2$O were used as the starting materials. A co-precipitation method was used for the preparation of fine ceramics and applied to synthesize the nano-powder of GdAlO$_3$ firstly. Then, the nano-powder of GdAlO$_3$ was mixed with the precipitates by the second co-precipitation method. After drying and calcination, the compound powder with eutectic composition (77 mol % Al$^{3+}$—23 mol % Gd$^{3+}$) was fast sintered by using the spark plasma sintering technique. The results revealed that the phases of the sintered samples were Al$_2$O$_3$ and GdAlO$_3$. The phases showed a homogeneous and interlaced distribution. All the matrix grains were submicron. The sizes of the intragranular structures were between 50 nm and 150 nm. Therefore, the intragranular structure displayed a novel mixture of nanometer–submicron and submicron–submicron types. The different intragranular structures all changed the fracture modes of Al$_2$O$_3$ grains from intergranular fracture to transgranular fracture.

Keywords: ceramic nanocomposite; spark plasma sintering; Al$_2$O$_3$; GdAlO$_3$; intragranular structures

1. Introduction

Compared with traditional ceramics, ceramic nanocomposites have better properties of fracture strength, fracture toughness, creep resistance, and wear resistance [1]. Niihara thought ceramic nanocomposites could be divided into three categories: intragranular nanocomposite, intergranular nanocomposite, and nano/nano composite [2]. The intragranular microstructure is unique as one nanophase exists inside the matrix grain of the other phase. Therefore, the unique microstructure enables the nanocomposite ceramic to draw much attention in the research field.

Nowadays, the main nanocomposite ceramics in the non-rare-earth system are Al$_2$O$_3$/SiC(n), Si$_3$N$_4$/SiC(n), and ZrO$_2$/Al$_2$O$_3$(n) (n stands for the nanoparticles in the above chemical compounds) et al. [3,4]. While, for the Al$_2$O$_3$/Re$_5$Al$_3$O$_{12}$ (Re stands for the rare-earth elements) system, the Al$_2$O$_3$/Y$_3$Al$_5$O$_{12}$ (YAG) composite ceramic is mainly focused on [5,6]. Liquid-coating method, reaction-sintering process, compound-powder method, and suspension-disperse-mixing method are the main synthetic methods to fabricate the intragranular microstructures [7]. The matrix grains with micron sizes are reported in the above mentioned systems. For the Al$_2$O$_3$/GdAlO$_3$ system, excellent flexural strength and thermal stability at high temperature have been reported. Waku et al. found the Al$_2$O$_3$/GdAlO$_3$ system displayed plastic deformation at 1873 K owing to dislocation motion, as in metals [8]. Ohashi et al. investigated the microstructures and orientation relationships of the Al$_2$O$_3$/GdAlO$_3$ eutectic fibers fabricated by the micro-pulling down method [9]. Ma et al. prepared the Al$_2$O$_3$/GdAlO$_3$ directionally solidified eutectic ceramics by laser floating zone melting process and studied the effects...
of composition and solidification rate on the microstructure of growth striations [10]. However, 
$\text{Al}_2\text{O}_3/\text{GdAlO}_3$ composite ceramic with the novel intragranular microstructure remains unknown. 

Eutectic chemical composition (77 mol % $\text{Al}^{3+}$—23 mol % $\text{Gd}^{3+}$) will enable the volume fractions 
of the two phases of the $\text{Al}_2\text{O}_3/\text{GdAlO}_3$ composite ceramic to be close. Then, the fine microstructure 
can be expected due to the much higher volume fraction of the second phase (~50 vol %). However, 
the increase of the volume fraction of the second phase will suppress the formation of the intragranular 
microstructure [11,12]. To attempt to solve this contradiction, the nano-powder of GdAlO$_3$ was 
synthesized firstly. Then, the nano-powder of GdAlO$_3$ was mixed with the precursors synthesized via 
co-precipitation method.

Meanwhile, spark plasma sintering (SPS) technique that was assisted by electric field was applied 
to fabricate the expected novel microstructure. Compared with hot pressed sintering, the spark plasma 
sintering technique can achieve rapid sintering densification for the ceramic powders in a few minutes 
at lower temperatures [13,14]. It has often been used to prepare the advanced and fine ceramic 
composites. The features of the microstructures of the composite ceramic were analyzed and their 
effect to fracture mode was also explored.

2. Materials and Methods

Gadolinium oxide ($\text{Gd}_2\text{O}_3$, 99.99% in purity, Rare-Chem hi-tech co., ltd., Huizhou, China) and 
aluminium nitrate ($\text{Al(NO}_3\text{)}_3$·9$\text{H}_2\text{O}$, 99.9% in purity, Sinopharm Chemical Reagent Co., Ltd., Shanghai, 
China) were used as the starting materials. The $d_{50}$ of $\text{Al(NO}_3\text{)}_3$·9$\text{H}_2\text{O}$ powder was 10 µm and 
the $d_{50}$ of $\text{Gd}_2\text{O}_3$ powder was 3 µm. Preparation process of GdAlO$_3$ was marked as routine 1.

The process of routine 1 was as follows. $\text{Al(NO}_3\text{)}_3$·9$\text{H}_2\text{O}$ and $\text{Gd}_2\text{O}_3$ were weighed according to 1:1 
of the stoichiometric ratio for Al and Gd elements, respectively. The powder of $\text{Al(NO}_3\text{)}_3$·9$\text{H}_2\text{O}$ was 
dissolved in deionized water. The chemical reaction is listed below.

$$\text{Al(NO}_3\text{)}_3$·9$\text{H}_2\text{O} = \text{Al(NO}_3\text{)}_3 + 9\text{H}_2\text{O}$$

Then, $\text{Gd}_2\text{O}_3$ was dissolved in nitric acid to produce $\text{Gd(NO}_3\text{)}_3$.

$$\text{Gd}_2\text{O}_3 + 6\text{HNO}_3 = 2\text{Gd(NO}_3\text{)}_3 + 3\text{H}_2\text{O}$$

$\text{Al(NO}_3\text{)}_3$ and $\text{Gd(NO}_3\text{)}_3$ were mixed in the homogeneous aqueous solution. For the 
co-precipitation method, the above salt solution and ammonia were both added into distilled water 
simultaneously in the back titration while pH value was kept between 8 and 9 till the end of the 
titration. The reactions between the above nitrate solutions and the ammonia are as follows.

$$\text{Al(NO}_3\text{)}_3 + \text{NH}_3\cdot\text{H}_2\text{O} = \text{Al(OH)}_3 + \text{NH}_4\text{NO}_3$$

$$\text{Gd(NO}_3\text{)}_3 + \text{NH}_3\cdot\text{H}_2\text{O} = \text{Gd(OH)}_3 + \text{NH}_4\text{NO}_3$$

In order to avoid the aggregation of small particles, ultrasonic fibrations were used during the 
co-precipitation reaction. The power of the ultrasonic instrument (Kunshan Ultrasonic Instruments 
Co, Ltd KQ-100DB, Kunshan, China) was selected as 100 W and the ultrasonic frequency was selected 
as 40 kHz. Then, the gelatinous precipitate was filtered and washed several times with water and 
ethanol, respectively. After drying at 120 °C for 24 h, the precipitates were calcined in air for 2 h, at a 
temperature ranging from 800 to 1200 °C, at a heating rate of 10 °C/min and cooled with the furnace.

The mixture process (routine 2) was as follows. The starting materials of $\text{Al(NO}_3\text{)}_3$·9$\text{H}_2\text{O}$ and 
$\text{Gd}_2\text{O}_3$ were weighed to achieve the final eutectic ratio (77 mol % $\text{Al}^{3+}$—23 mol % $\text{Gd}^{3+}$). The amount 
of GdAlO$_3$ powder was selected to represent 2 vol % of the ceramic composite. Considering the 
dispersion of the GdAlO$_3$ powder and the precipitation reaction, the pH value was kept between 9 
and 10 [15]. At the same time, the nanoparticles of GdAlO$_3$ from routine 1 were added during the 
co-precipitation reaction for routine 2. In order to gain a well dispersion of GdAlO$_3$ nanoparticles,
the ultrasonic and mechanical agitation were used during the reaction. After drying and calcination at 1100 °C, the compound powder was loaded in the graphite mold.

According to phase diagram of Al$_2$O$_3$-Gd$_2$O$_3$ [16], the temperature for the coexistence of GdAlO$_3$ and Al$_2$O$_3$ phases is near 1600 °C. Therefore, the spark plasma sintering (SPS-3.20-MK-V, Sumitomo Coal Mining Co., Ltd., Kyoto, Japan) was conducted at 1600 °C for 3 min at a heating rate of 100 °C/min, at the pressure maintaining of 30 MPa and cooled with the furnace.

Phases of the calcined powder and the sintered sample were identified by an X-ray diffractometer (XRD, RIGAKU D/Max-rB, Tokyo, Japan) with Cu Ka radiation (0.1542 nm). The accelerating voltage was 40 kV with a tube current of 40 mA. The morphology of the calcined powder and the microstructure of the composite ceramic were examined by using a scanning electron microscope (FE-SEM, S-4800, HITACHI, Tokyo, Japan).

3. Results and Discussion

3.1. XRD Patterns of the Powder and Sintered Sample

The XRD patterns of the precursor powders of GdAlO$_3$ calcined at different temperatures are shown in Figure 1. As shown in Figure 1, there was no distinctive diffraction peak of the precursor powder calcined at 800 °C. When the calcination temperature got to 900 °C, the diffraction peaks were made up of GdAlO$_3$, α-Al$_2$O$_3$, Gd$_2$O$_3$, and Gd$_4$Al$_2$O$_9$. It can be known that hydroxide in the precursor underwent decomposition and the perovskite-type GdAlO$_3$ began to crystallize. The reaction equations are shown as follows.

$$2\text{Al(OH)}_3 = \text{Al}_2\text{O}_3 + 3\text{H}_2\text{O} \quad (5)$$
$$2\text{Gd(OH)}_3 = \text{Gd}_2\text{O}_3 + 3\text{H}_2\text{O} \quad (6)$$
$$\text{Gd}_2\text{O}_3 + \text{Al}_2\text{O}_3 = 2\text{GdAlO}_3 \quad (7)$$

Further calcining at 1000 °C led to an increase in the intensity of the GdAlO$_3$ peaks while the intensities of the diffraction peaks of α-Al$_2$O$_3$, Gd$_2$O$_3$, and Gd$_4$Al$_2$O$_9$ decreased.

$$\text{Gd}_4\text{Al}_2\text{O}_9 + 2\text{Al}_2\text{O}_3 = 4\text{GdAlO}_3 \quad (8)$$

![Figure 1. XRD patterns of the precursor powders of GdAlO$_3$ calcined at different temperatures.](image-url)
close, the transition phase of Gd₄Al₂O₉ could be easily formed in the calcination process. When the stoichiometric ratio for Al and Gd was close to the eutectic ratio, the transition phase of Gd₃Al₅O₁₂ could be easily formed in the calcination process [15].

The XRD patterns of the sample with eutectic composition sintered by SPS is shown in Figure 2. It shows that Al₂O₃/GdAlO₃ composite ceramic is successfully fabricated and no impurity phase is found.

![XRD patterns of the sample with eutectic composition sintered by SPS](image)

**Figure 2.** XRD pattern of sample with eutectic composition sintered by SPS.

3.2. SEM Micrographs of the Powders

Figure 3a shows the SEM micrographs of the precursor powders calcined at 1100 °C. Primary nanoparticles are observed and they are slightly aggregated due to the drying and calcination processes. Figure 3b shows the SEM micrographs of compound powders prepared from routine 2.

![SEM micrographs of compound powders prepared from routine 2](image)

**Figure 3.** SEM micrographs of (a) the GdAlO₃ powder (b) the compound powder synthesized by routine 2.

3.3. Surface of Sintered Samples

The thickness of the sintered sample is about 2 mm and the diameter is about 10 mm. Figure 4 is the SEM micrograph of the polished surface of the sample sintered by SPS. It indicates that the microstructure of the ceramic composite is dense and made up of two phases. The average grain size of the microstructure is about 500 nm. According to the testing conditions, the brighter submicron phase is GdAlO₃ and the darker submicron phase is α-Al₂O₃. It reveals that the homogeneous, interlaced and fine microstructure for Al₂O₃/GdAlO₃ can be successfully prepared by the wet chemical process and the SPS technique.
3.4. Fracture Surface of Sintered Samples

Figure 5a shows the Al₂O₃ matrix grain without intragranular structure. It can be known that the two phases combines well and there is no impurity phase at the interfaces. Figure 5b shows there are several nano-particles (~50 nm) of GdAlO₃ phase in the submicron matrix grain of Al₂O₃ phase to form the novel intragranular structure of the nanometre-submicron type. Moreover, the intragranular structures are both observed in the matrix grains of Al₂O₃ phase in Figure 5c,d. The average nano-particle sizes are about 100 and 150 nm in Figure 5c,d, respectively. The grain sizes of the intragranular structures in Figure 5c,d are relatively larger than those in Figure 5b. Furthermore, the amount of the nano-particles in Figure 5b is more than those in Figure 5c,d. Thus, the intragranular structures of the submicron–submicron type in Figure 5c,d are presented.

Figure 5. SEM micrographs of fracture morphologies (a) the Al₂O₃ grain without intragranular structure (b) the particle size of intragranular structure was about 50 nm (c) the particle size of intragranular structure was about 100 nm (d) the particle size of intragranular structure was about 150 nm.
The formation of the intragranular structures is discussed as follows. The formation of the intragranular structures was associated with the chemical environment and the dispersion of GdAlO$_3$ particles. When the GdAlO$_3$ particles were dispersed and surrounded by more Al$_2$O$_3$ particles, it was beneficial to promote the diffusion and mass transfer for Al$_2$O$_3$ phase by using the spark plasma sintering process. It enabled the Al$_2$O$_3$ grains to have the relatively lower sintering temperature and faster migration velocity of crystal boundary. Then, with the enhanced growth of Al$_2$O$_3$ grains, the GdAlO$_3$ nano-particles were probably swallowed up by the Al$_2$O$_3$ grain for the case of Figure 5b. When the GdAlO$_3$ nano-particles were slightly aggregated, they were easily to form one larger grain during the sintering process for the cases of Figure 5c,d. When the GdAlO$_3$ nano-particles were surrounded by other more GdAlO$_3$ particles, the nanoparticles of GdAlO$_3$ phase tended to grow into one larger submicron grain of GdAlO$_3$ phase for the case of Figure 5a.

Based on the above discussion, the synthesized Al$_2$O$_3$/GdAlO$_3$ ceramic composite has the novel microstructure as shown in Figure 6a. The novel microstructure is different from the traditional intragranular microstructure and intergranular microstructure. The volume fractions for Al$_2$O$_3$ phase and GdAlO$_3$ phase are close and the well alternative distribution of the submicron matrix grains is formed. In the matrix grains of Al$_2$O$_3$ phase, nanoparticles with sizes of 50–150 nm in the intragranular microstructures are present. Up to now, the traditional intragranular microstructures of Figure 6b for Al$_2$O$_3$/YAG are mainly reported [18,19]. The common sizes of the matrix grains for the traditional intragranular microstructures are micron. The nanoparticles of YAG phase often locate in the matrix grains of Al$_2$O$_3$ phase and the larger particles of YAG phase locate at the grain boundaries of Al$_2$O$_3$ phase, as shown in Figure 6b.

![Schematic diagram of this new microstructure](image).

Figure 6. (a) Schematic diagram of this new microstructure (b) the traditional microstructure of the intragranular and intergranular types.

Generally, the fracture mode of Al$_2$O$_3$ grains is intergranular fracture [20,21]. It reveals that the intragranular structure tended to induce the transgranular fracture of Al$_2$O$_3$ grains in Figure 5d. Since the average coefficient of volume thermal expansion for GdAlO$_3$ (−31.8 × 10$^{-6}$/°C) is higher than that of Al$_2$O$_3$ (−21.9 × 10$^{-6}$/°C) [17,22], the GdAlO$_3$ nanoparticles in the intragranular structure may have larger volume contraction than that of Al$_2$O$_3$ matrix during the cooling process following the sintering densification. Thus, the GdAlO$_3$ nanoparticles pulled the Al$_2$O$_3$ matrix for the GdAlO$_3$/Al$_2$O$_3$ system at room temperature. The residual tensile stress field generated and tended to form microcracks near the interfaces of the intragranular structures. If the cracks nucleated near the intragranular structures, the stress field of the crack tip would interact with the residual tensile stress field and other microstructure defects as the external force loaded on the ceramic composite. When the shear stress value exceeded the cleavage strength of the ceramic composite, the cleavage crack would propagate along the specific crystal plane [23]. If the cracks nucleated far from the intragranular structures, the crack would be captured by the intragranular structure as it propagated [24]. Due to the interactions of residual stress and defects, the direction of crack propagation was deflected and the crack went through the Al$_2$O$_3$ matrix grain along the specific crystal plane. As described above,
the cleavages formed for the cases of Figure 5b–d. Meanwhile, Figure 5b–d also present the cleavage patterns for the Al$_2$O$_3$ matrix grains. The cleavage patterns are mainly consisted of some parallel cleavage steps that are made up of the intersections of different cleavage planes.

4. Summary

In order to prepare the intragranular structures for Al$_2$O$_3$/GdAlO$_3$ composite ceramic, GdAlO$_3$ powder was synthesized at the calcination temperature of 1100 °C for 2 h by co-precipitation method. The nanocomposite ceramic of Al$_2$O$_3$/GdAlO$_3$ with intragranular structures was successfully obtained by the chemical process and spark plasma sintering technique. The sizes of all the matrix grains were kept submicron. By the above techniques, the intragranular structure that contains GdAlO$_3$ Al$_2$O$_3$ phase and induced the transgranular fracture instead of intercrystalline fracture due to the residual stress. Furthermore, the features of cleavage in the Al$_2$O$_3$ grains display parallel cleavage steps.

Author Contributions: Conceptualization, S.S. and X.Q.; Methodology, S.S.; Investigation, S.S.; Resources, S.S. and X.Q.; Writing—Original Draft Preparation, S.S.; Writing—Review & Editing, S.S.; Funding Acquisition, S.S. and X.Q.

Funding: This research was funded by the Startup Fund of Scientific Research in North China University of Technology [110052971803/041] and National Natural Science Foundation of China [50702008]. The APC was funded by the Startup Fund of Scientific Research in North China University of Technology [110052971803/041].

Conflicts of Interest: The authors declare no conflict of interest.

References

1. Oh, S.T.; Sando, M.; Niihara, K. Mechanical and magnetic properties of Ni-Co dispersed Al$_2$O$_3$ nanocomposites. J. Mater. Sci. 2001, 36, 1817–1821. [CrossRef]
2. Niihara, K. New design concept of structural ceramics—Ceramic nanocomposites. J. Ceram. Soc. Jpn. 1991, 99, 974–982. [CrossRef]
3. Sun, X.D.; Li, J.G. Intragranular particle residual stress strengthening of Al$_2$O$_3$–SiC nanocomposites. J. Am. Ceram. Soc. 2005, 88, 1536–1543. [CrossRef]
4. Rouxel, T.; Wakai, F.; Brito, M.E.; Iwamoto, A.; Izaki, K. Intragranular crack deflection and crystallographic slip in Si$_3$N$_4$/SiC nanocomposites. J. Eur. Ceram. Soc. 1993, 11, 431–438. [CrossRef]
5. Wang, X.; Tian, Z.; Zhang, W.; Zhong, Y.; Xian, Q.; Zhang, J.; Wang, J. Mechanical properties of directionally solidified Al$_2$O$_3$/Y$_3$Al$_5$O$_12$ eutectic ceramic prepared by optical floating zone technique. J. Eur. Ceram. Soc. 2018, 38, 3610–3617. [CrossRef]
6. Wang, X.; Wang, D.; Zhang, H.; Tian, Z.; Du, K.; Wang, J.; Lou, L.; Zhang, J. Mechanism of eutectic growth in directional solidification of an Al$_2$O$_3$/Y$_3$Al$_5$O$_12$ crystal. Scr. Mater. 2016, 116, 44–48. [CrossRef]
7. Zhang, J.X.; Gao, L.Q. Study on chemical processing for Al$_2$O$_3$/SiC$_p$ nano-composites. J. Chin. Ceram. Soc. 2011, 29, 550–553.
8. Waku, Y.; Nakagawa, N.; Wakamoto, T.; Ohtsubo, H.; Shimizu, K.; Kohtoku, Y. A ductile ceramic eutectic composite with high strength at 1873 K. Nature 1997, 389, 49–52. [CrossRef]
9. Ohashi, Y.; Yasui, N.; Suzuki, T.; Watanabe, M.; Den, T.; Kamada, K.; Yokota, Y.; Yoshikawa, A. Orientation relationships of unidirectionally aligned GdAlO$_3$/Al$_2$O$_3$ eutectic fibers. J. Eur. Ceram. Soc. 2014, 34, 3849–3857. [CrossRef]
10. Ma, W.; Su, H.; Zhang, J.; Ren, Q.; Liu, H.; Wang, E.; Ren, J.; Lu, Z.; Liu, L.; Fu, H. Effects of composition and solidification rate on growth striations in laser floating zone melted Al$_2$O$_3$/GdAlO$_3$ eutectic ceramics. J. Am. Ceram. Soc. 2018, 101, 3337–3346. [CrossRef]
11. Xu, C.H.; Sun, D.M. Formation of intragranular nano-structures in micro-sized ceramic composite materials. Mater. Sci. Eng. A 2008, 1, 338–342. [CrossRef]
12. Wang, X.; Shan, Y.; Gong, H.Y.; Yu, X.G.; Xu, J.; Yin, Y.S. Formation mechanism of intragranular structure in nano-composites. Nonferr. Metall. Soc. 2004, 14, 265–269.
13. Chaim, R.; Chevallier, G.; Weibel, A.; Estournès, C. Grain growth during spark plasma and flash sintering of ceramic nanoparticles: A review. *J. Mater. Sci.* **2018**, *53*, 3087–3105. [CrossRef]

14. Liu, D.; Gao, Y.; Liu, J.; Liu, F.; Li, K.; Su, H.; Wang, Y.; An, L. Preparation of Al$_2$O$_3$-Y$_3$Al$_5$O$_{12}$-ZrO$_2$ eutectic ceramic by flash sintering. *Scr. Mater.* **2016**, *114*, 108–111. [CrossRef]

15. Sun, S.; Xu, Q. Effect of calcination temperature on phase transformation and microstructure of Al$_2$O$_3$/GdAlO$_3$ compound powder prepared by Co-precipitation method. *Key Eng. Mater.* **2011**, *512*, 535–538. [CrossRef]

16. Harada, Y.; Ayabe, K.; Uekawa, N.; Kojima, T.; Kakegawa, K.; Kim, S.J. Formation of GdAlO$_3$-Al$_2$O$_3$ composite having fine pseudo-eutectic microstructure. *J. Eur. Ceram. Soc.* **2008**, *28*, 2941–2946. [CrossRef]

17. Chaudhury, S.; Parida, S.C.; Pillai, K.T.; Mudher, K.S. High-temperature X-ray diffraction and specific heat studies on GdAlO$_3$, Gd$_3$Al$_5$O$_{12}$ and Gd$_4$Al$_2$O$_9$. *J. Solid State Chem.* **2007**, *180*, 2393–2399. [CrossRef]

18. Wang, H.Z.; Gao, L.; Li, W.Q.; Kawaoka, H.; Niihara, K. Preparation and Microstructure of Al$_2$O$_3$-YAG Composites. *J. Inorg. Mater.* **2001**, *16*, 169–172.

19. Paneto, F.J.; Pereira, J.L.; Lima, J.O.; Jesus, E.J.; Silva, L.A.; Lima, E.S.; Cabral, R.F.; Santos, C. Effect of porosity on hardness of Al$_2$O$_3$-Y$_3$Al$_5$O$_{12}$ ceramic composite. *Int. J. Refract. Met. Hard Mater.* **2015**, *48*, 365–368. [CrossRef]

20. Wang, H.Z.; Gao, L.; Guo, J.K. Effect of nanoscale SiC particles on the microstructure of Al$_2$O$_3$ ceramics. *Ceram. Int.* **2000**, *26*, 391–396. [CrossRef]

21. Palmero, P.; Pulci, G.; Marra, F.; Valente, T.; Montanaro, L. Al$_2$O$_3$/ZrO$_2$/Y$_3$Al$_5$O$_{12}$ Composites: A High-Temperature Mechanical Characterization. *Materials* **2015**, *8*, 611–624. [CrossRef] [PubMed]

22. Chu, G.; Zhai, X.J.; Fu, Y.; Lu, Z.J.; Bi, S.W. Lattice thermal expansion coefficients of combustion synthesized alpha-Al$_2$O$_3$ nanoparticles. *J. Inorg. Mater.* **2005**, *20*, 755–758.

23. Matsuo, H.; Mitsuhara, M.; Ikeda, K.; Hata, S.; Nakashima, H. Electron microscopy analysis for crack propagation behavior of alumina. *Int. J. Fatigue* **2010**, *32*, 592–598. [CrossRef]

24. Xu, Y.R.; Zangvil, A.; Kerber, A. SiC nanoparticle-reinforced Al$_2$O$_3$ matrix composites: Role of intra- and intergranular particles. *J. Eur. Ceram. Soc.* **1997**, *17*, 921–928. [CrossRef]