行政院國家科學委員會專題研究計畫成果報告

多相材料的微結構設計(二) 納米氧化鋯多相材料

Microstructural Design of Multiphased Materials (II) Multiphased Zirconia-matrix Nanocomposites

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一. 摘要

陶瓷的工程應用往往受制於它的脆性. 在陶瓷中加入陶瓷或金屬顆粒等一種韌化劑可改善陶瓷的機械性質. 本研究探討在氧化鋯陶瓷中同時加入奈米尺寸的金屬鎳及氧化鋁顆粒的複合材料的機械性質. 實驗結果顯示氧化鋯的強度因奈米鎳及氧化鋁顆粒的同時添加而會大幅改善.此納米氧化鋯多相材料具有應用在結構上及燃料電池方面的潛力.

關鍵詞: 奈米複合材料, 多相材料, 強度, 氧化鋯, 鎳, 氧化鋁

Abstract

The applications of ceramics for structural components are often limited by its brittleness. The addition one toughening agent, such as ceramic particles or metallic particles, can improve the mechanical properties of ceramics. The present study explores the processing-properties relationships of the zirconia-matrix composites containing both alumina particles and nano-sized nickel inclusions. Our results suggested that the strength of zirconia could be improved significantly by adding two toughening agents. In the present study, the ZrO_2- Al_2O_3-Ni nano-composites were prepared either by pressureless sintering or by spark plasma sintering (SPS) process. The nanacomposite have the potential for structural applications and as the electrode for solid oxide fuel cell.

Keywords: nanocomposite, multiphased material, strength, zirconia, nickel, alumina

二. 緣由及目的

The brittle nature of ceramics hinders their applications as structural components. One approach targets the toughness improvement through the addition of toughening reinforcement. Ceramic or metallic reinforcement is incorporated into a ceramic matrix. The reinforcement interacts with the pre-existing and/or service-induced cracks to slow down their propagation. The toughness of the brittle matrix is thus improved through such interactions. Though the strength may be sacrificed slightly by adopting this approach, the reinforcement also acts as stress concentration site. However, this approach is attractive for the reliability of ceramics during the subsequent usage can be improved significantly.

Many ceramic or metallic materials, such as zirconia[1] and nickel[2] have been used as toughening reinforcements. The presence of these toughening agents enhances the toughness of ceramics through the generation of various toughening mechanisms. [3]The propagation of cracks is hampered due to the effect of these mechanisms, resulting in an increase in the toughness of ceramics. However, the presence of a single toughening agent frequently induces more than one toughening mechanism. These mechanisms operate simultaneously to a different extent within the brittle matrix. In the previous study, we have demonstrated that the interactions between different toughening mechanisms can bring in extra toughening effect. This result has been published recently. [4] In the present study, the same approach is applied to a zirconia-matrix composite system. Similar to the previous study, two toughening agents, alumina and nickel, are incorporated into the matrix simultaneously. The mechanical properties of the composites are measured.

三. 研究步驟

Detailed procedures for the preparation of the composites containing two toughening agents can be found in the report of the previous study. A brief description is given here.

The nano-sized nickel particles were prepared by coating nickel nitrate solution onto the surface of zirconia (TZ-3Y, Tosoh Co., Japan) and alumina (TM-DAR, Taimei Chem. Co. Ltd., Tokyo, Japan) powders. This technique was developed by Prof. Yang, Fang-Ja University. The powder was also prepared by his research group. The Al_2O_3-ZrO_2–Ni green compacts were either by spark plasma sintering (SPS) at 1350C for 5 minutes or by pressureless sintering within a reducing atmosphere, carbon monoxide, at 1600℃ for 1 h. The ZrO_2-Al_2O_3 and ZrO_2-Ni specimens, for comparison, were also prepared with the same techniques. The sintered specimens were machined longitudinally with a diamond wheel. The strength was determined by a biaxial strength measurement technique. The toughness of the specimens was determined by the indentation technique. The load applied was 20 Kg. Phase identification of was performed by X-ray diffractometry (XRD). The chemical composition of the composite powders was determined by applying an induced coupled plasma (ICP) technique.

四. 結果與討論

The chemical composition of ZrO_2-Al_2O_3, ZrO_2-Ni and ZrO_2-Al_2O_3-Ni composite powders as revealed by ICP analysis were 95%-5%, 99.%-0.9 and 94.1%-5.2%-0.7%, respectively. The content is expressed in their volume percent.
The XRD analyses detect t-ZrO_2, α-Al_2O_3 and cubic Ni in the sintered composites. The nano-sized nickel particles can be successfully prepared by the coating technique.

The relative density of all the composites prepared by pressureless sintering and SPS is higher than 97%, Fig. 1. The microstructures of the specimens are shown in Fig. 2. From Fig. 2, even though the temperature of SPS is low and time at peak temperature is short, the density is rather high. It further confirms that dense composites can be prepared by SPS at a temperature much lower than the one for conventional sintering.

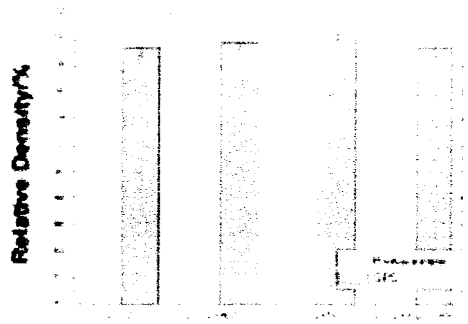

Fig. 1, The relative density of the ZrO_2-matrix composites.

The specimens shown in Fig. 2 were ground and polished, then etched thermally at 1250C for half hour. The atmosphere applied for the thermal etching processing was nitrogen. A small amount of oxygen was present in the atmosphere. The Ni particles on the polished surface were thus oxidized during thermal etching. Therefore, the bright particles were virtually nickel oxide. It size is bigger than the size of the Ni inclusions.

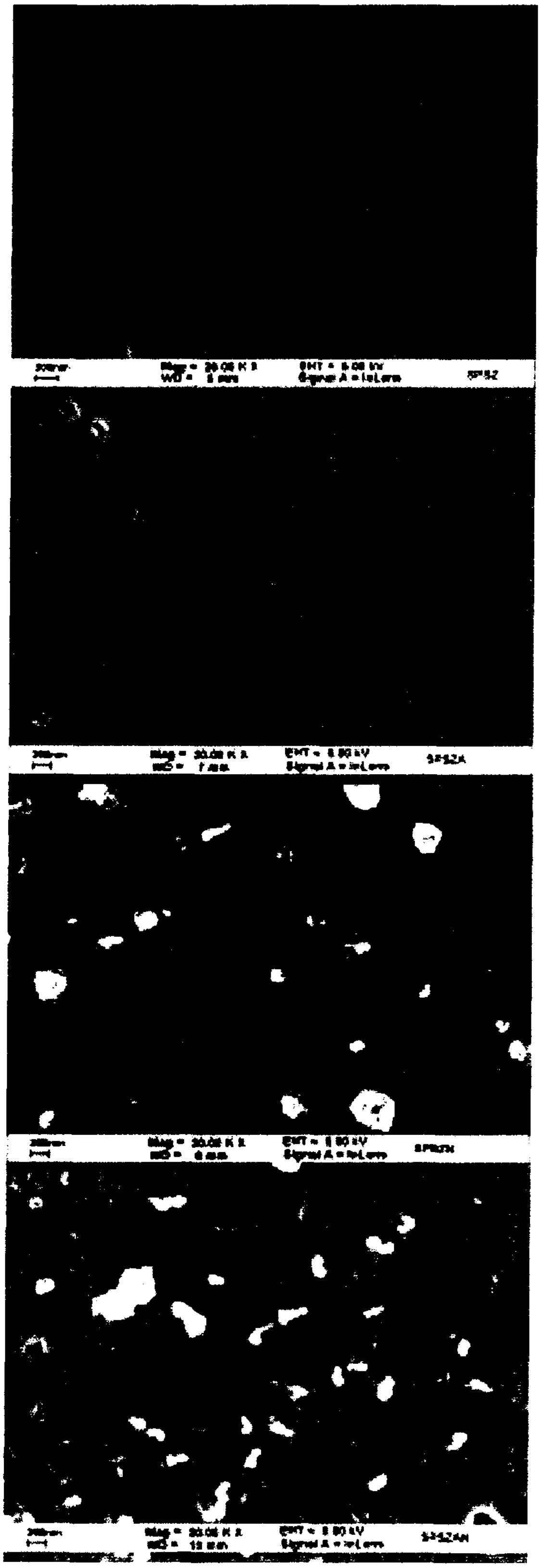

Fig. 2, SEM micrographs of the (a) ZrO_2 (b) ZrO_2-Al_2O_3, (c) ZrO_2-Ni and (d) ZrO_2-Al_2O_3-Ni specimens prepared by spark plasma sintering at 1350C for 5 minutes.

The size of ZrO_2 matrix grains in the composites is shown in Fig. 3. The presence of Al_2O_3 and Ni inclusions can all enhance the size of ZrO_2 matrix grains. The size of ZrO_2 grains in the SPS specimens is smaller than those in the pressureless sintered specimens. The strength and toughness of the composites are shown in Fig. 4 and Fig. 5, respectively.

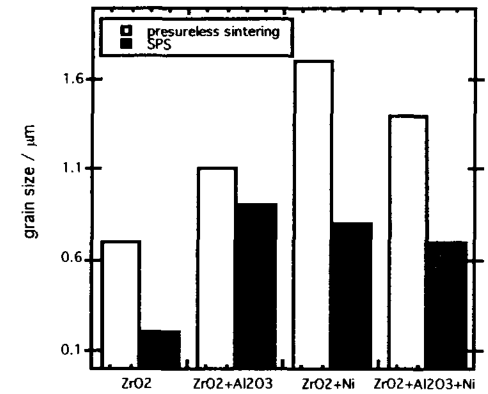

Fig. 3, Size of matrix ZrO_2 grains in the specimens.

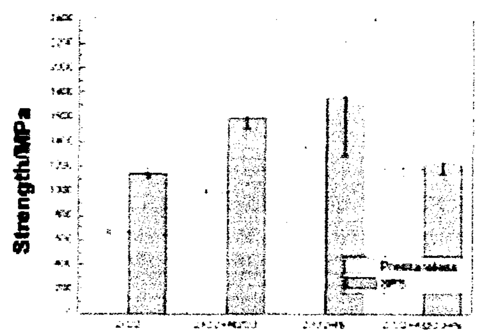

Fig. 4, Strength of the ZrO_2 matrix composites prepared by pressureless sintering and SPS.

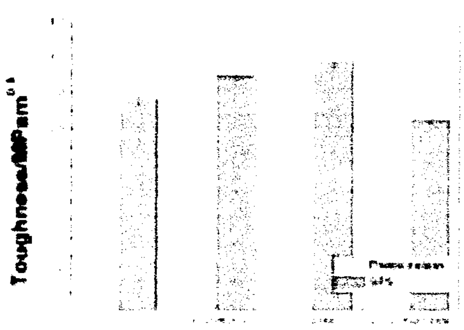

Fig. 5, Toughness of the ZrO_2 matrix composites prepared by pressureless sintering and SPS.

Though the size of ZrO_2 matrix grains in the composite is larger than that in the pure ZrO_2 specimen, the strength of the composite is higher than that of pure ZrO_2. It is also noted that the strength of the SPS specimens is higher than the presureless sintered specimens. Furthermore, the strength of the ZrO_2-Ni composite reaches a value as high as 1600MPa, though the Ni content in the composite is only 0.9 vol.%.

The toughness of all the composites is more or less the same. It may due to that the inclusion content is very low, the toughing mechanisms are not active enough to result in significant toughness increase.[5]

五 結論

Nano-sized nickel particles were prepared by a solution coating technique. Dense ZrO_2-Al_2O_3-Ni composites were prepared by presureless sintering and spark plasma sintering. The strength of the composite is higher than that of pure zirconia, though the toughness is more or less the same.

六. 參考文獻

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