

# 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(一) 納米多相材料

Microstructural Design of Multiphased Materials (I) Multiphased Nanocomposites

計畫編號：NSC90-2216-E-002-034

執行期限：90年8月1日至91年7月31日

主持人：段維新 教授

共同主持人：郭景坤, 楊聰仁, 傅正義 教授

執行機構：國立台灣大學材料科學及工程學研究所

計畫參與人員：連智偉, 黃永慶, 郭寶聲, 鄭至先, 黃淑敏

### 一. 摘要

陶瓷的工程應用往往受制於它的脆性。在陶瓷中加入如氧化鋯或金屬顆粒等一種韌化劑可改善陶瓷的韌性。本研究探討在陶瓷中同時加入金屬鎳及氧化鋯韌化劑的複合材料的韌化行為。本研究的實驗結果顯示氧化鋁的韌性因鎳及氧化鋯的同時添加而會大幅改善。這是因金屬的加入，氧化鋯的相變化會增加，氧化鋯的添加可改善鎳的變形能力。故使同時含氧化鋯及金屬鎳韌化劑的複合材料的韌性較只含氧化鋯的複合材料的韌性為高。

**關鍵詞：**奈米顆粒, 複合材料, 韌性, 氧化鋯, 鎳

### Abstract

The applications of ceramics for structural components are often limited by its brittleness. Adding one toughening agent, such as zirconia particles or metallic particles, can improve the toughness of ceramics. The present study explores the toughening behavior of the composites containing both zirconia particles and metallic inclusions. Our

results suggested that the toughness of ceramics could be improved significantly by adding two toughening agents. In the present study, the results on  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$ -Ni composites are demonstrated. The presence of metallic inclusions increases the phase transformation extent of the zirconia inclusions. The addition of  $\text{ZrO}_2$  particles into  $\text{Al}_2\text{O}_3$ -Ni system enhances the ductility of Ni inclusions. The toughness increase of  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$ -Ni composites is therefore higher than the sum of the toughness increase of  $\text{Al}_2\text{O}_3$ - $\text{ZrO}_2$  and of  $\text{Al}_2\text{O}_3$ -Ni composites.

**Keywords:** nano-sized particle, composite, toughness, zirconia, nickel

### 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

toughness values achieved by adding both zirconia and nickel inclusions are 519 MPa and 10 MPam<sup>0.5</sup>, respectively. These values are around 2 to 3 times those of alumina alone.

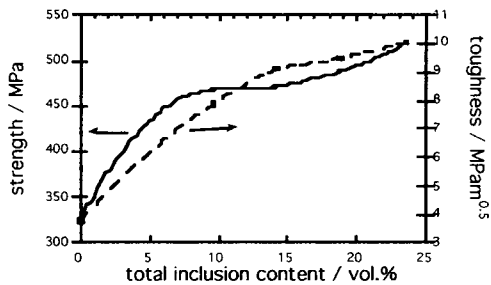


Fig. 2, The strength and toughness of Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub>-Ni composites.

Fig. 3 shows the amount of m-phase detected on the fracture surface of Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub>-Ni composites. More m-zirconia particles are detected for the Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub>-Ni composites. The elastic modulus of nickel is very close to that of zirconia.<sup>2</sup> However, the nickel is ductile; therefore, it is able to absorb more stresses from the nearby zirconia particles. More transformation is thus taken place. Fig. 3 indicates that the contribution from transformation toughening is enhanced due to the presence of nickel inclusions.

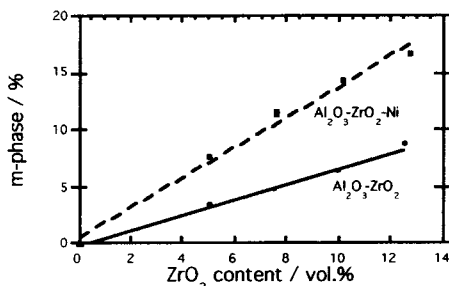


Fig. 3, The extent of transformation of ZrO<sub>2</sub> inclusions on the fracture surface.

Fig. 4 shows the toughness increase as a function of the square root of the product of volume fraction and inclusion size of Ni in the Al<sub>2</sub>O<sub>3</sub>-Ni and Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub>-Ni composites. For the Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub>-Ni composites, the contribution from transformation toughening

is removed from the total toughness increase, to evaluate the contribution from plastic deformation of Ni. There is a roughly linear relationship between  $\Delta K_{IC}$  and  $(Fxd)^{0.5}$ , indicating that the plastic deformation of Ni enhances the toughness.<sup>5</sup> Zirconia is a good oxygen conductor. The presence of zirconia particles can help the removal of oxygen from nickel inclusions. The lower oxygen solute in nickel, the higher its ductility.<sup>6</sup>

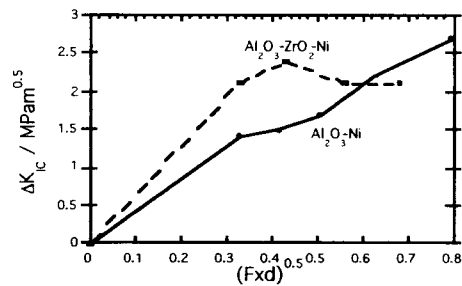


Fig. 4, The toughness increase,  $\Delta K_{IC}$ , as a function of the square root of the product of volume fraction, F, and inclusion size, d.

## 五 結論

Nano-sized nickel particles were prepared by a solution coating technique. Two toughening agents, transformable zirconia and ductile nickel, were added into alumina. Both the contributions from the transformation toughening and the plastic deformation to toughening effect are enhanced. The present study demonstrates that the toughness of brittle ceramics can be significantly improved by adding two toughening agents.

## 六. 參考文獻

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Many ceramic or metallic materials, such as zirconia<sup>1</sup> and nickel<sup>2</sup> have been used as toughening reinforcements. The presence of these toughening agents enhances the toughness of ceramics through the generation of various toughening mechanisms. 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 present study, it will be demonstrated that the coupling between different toughening mechanisms plays a key role on the development of tough composites.

### 三. 研究步驟

Detailed procedures for the preparation of the composites containing two toughening agents can be found elsewhere.<sup>3</sup> A brief description is given here.

The nano-sized nickel particles were prepared by coating nickel nitrate solution onto the surface of an alumina powder (TM-DAR, Taimei Chem. Co. Ltd., Tokyo, Japan). This technique was developed by Prof. Yang, Fang-Ja University. The powder was also prepared by his research group. The micro-sized nickel particles were also prepared by mixing the with nickel oxide (NiO, Johnson Matthey Co., U.S.A.), and zirconia (TZP,  $ZrO_2 + 3 \text{ mol.}\% Y_2O_3$ , Hanwha Ceramics Co., Australia) powders by ball milling in ethyl alcohol for 24 hours. The grinding media used were zirconia balls. The green compacts were sintered within a reducing atmosphere, carbon monoxide, at  $1600^\circ\text{C}$  for 1 h. The  $Al_2O_3$ - $ZrO_2$  and  $Al_2O_3$ -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 the 4-point bending technique, the toughness by the single-edge-notched-beam (SENB) technique.

The rate of loading was 0.5 mm/minute. Phase identification of was performed by X-ray diffractometry (XRD). The relative phase content of zirconia was estimated by using the method proposed by Evans et al.<sup>4</sup>

### 四. 結果與討論

XRD analyses detect  $\alpha$ - $Al_2O_3$ , t- $ZrO_2$  and cubic Ni in the sintered composites. The nano-sized nickel particles can be successfully prepared by the coating technique, Fig. 1. The size of the nickel particles is around 10 nm.



Fig. 1, TEM micrograph of a Ni-coated  $Al_2O_3$  powder. Nano-sized Ni particles are found on the surface of alumina.

The relative density of the composites is higher than 98%. It demonstrate the composites containing micro-sized nickel and zirconia particles can be prepared by using a pressureless sintering technique. The sintering of nano-composites is currently carried out in our laboratory.

Fig.2 shows the strength and toughness of the  $Al_2O_3$ - $ZrO_2$ -Ni composites as a function of total inclusion content. Both the strength and toughness of alumina are enhanced. The optimal strength and

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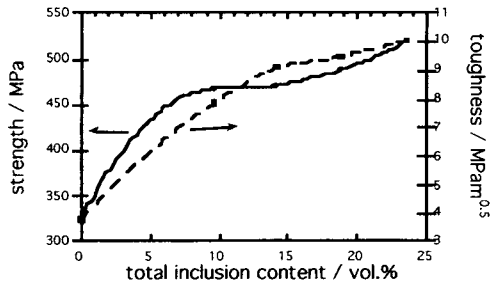


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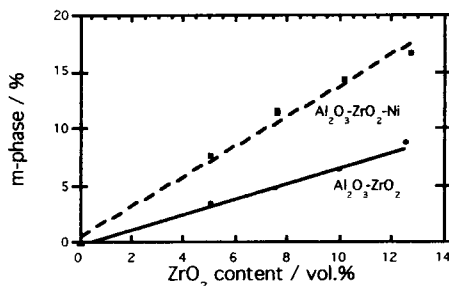


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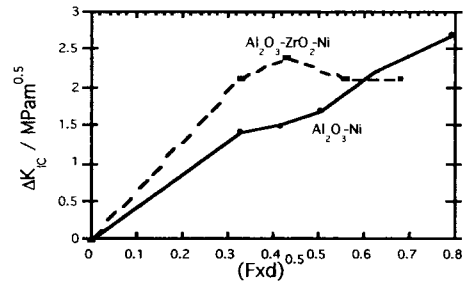


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# 訪材料複合新技術國家重點實驗室

## 心得報告

段維新

國立台灣大學材料科學及工程學系

### 1. 緣起

筆者所主持的國科會研究計畫[多相材料的微結構設計(一)納米多相材料](NSC90-2216-E002-034) 邀中國科學院上海矽酸鹽研究所的郭景坤院士及武漢理工大學材料複合新技術國家重點實驗室傅正義教授及逢甲大學的楊聰仁教授擔任共同主持人，此計畫結合四個實驗室的專長及資源 – 例如楊聰仁教授在納米粉的製備，筆者及郭景坤院士在陶瓷基複合材料多年的製程設計及微結構設計經驗及實績，傅正義教授所負責的 Spark Plasma Sintering (SPS)設備為中國第一台(目前中國已有四台，分別在武漢的材料複合新技術國家重點實驗室，上海的中國科學院上海矽酸鹽研究所，北京的清華大學材料系及北京工業大學)，傅教授具充分的製備納米材料及納米複合材料的經驗，本計劃希能透過多個實驗室的資源及經驗，共同開發納米多相材料。

筆者於 90 年 8 月 5 日至 8 月 27 日先以自費的方式訪上海的上海矽酸鹽研究所，與上海矽酸鹽研究所的郭景坤院士及其他教授就新材料開發及資源分享達成初步了解。

筆者又於 90 年 11 月 8 日至 11 月 11 日訪問位於武漢理工大學校內的材料複合新技術國家重點實驗室，並順道於 90 年 11 月 11 日至 11 月 16 日參加在昆明舉行的第二屆中國國際高性能陶瓷研討會，現將訪問及參加會議的心得，報告如下

### 2. 訪問國家重點實驗室

#### 2.1 材料複合新技術國家重點實驗室