

# 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.

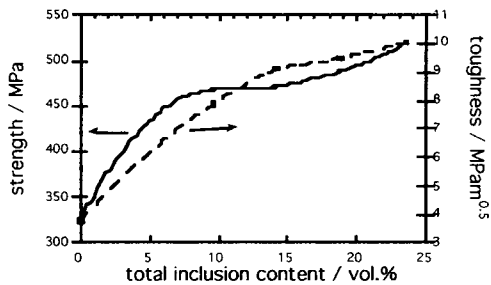


Fig. 2, The strength and toughness of Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub>-Ni composites.

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.

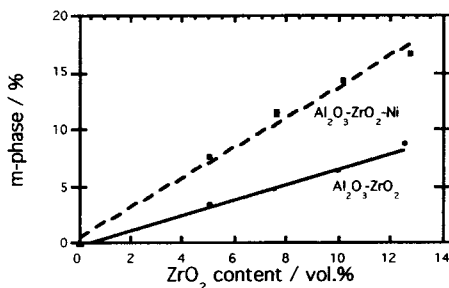


Fig. 3, The extent of transformation of ZrO<sub>2</sub> inclusions on the fracture surface.

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>

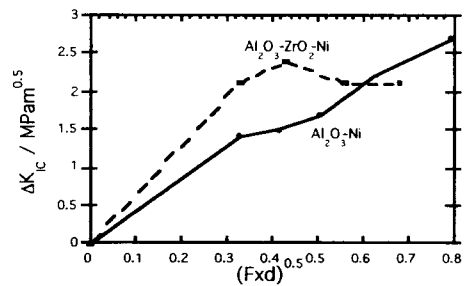


Fig. 4, The toughness increase,  $\Delta K_{IC}$ , as a function of the square root of the product of volume fraction, F, and inclusion size, d.

## 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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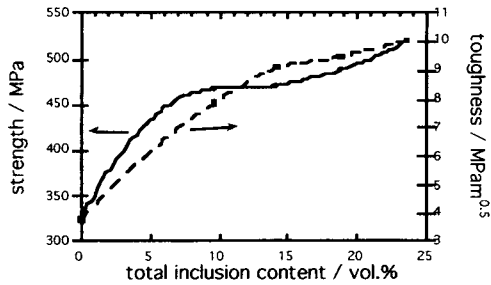


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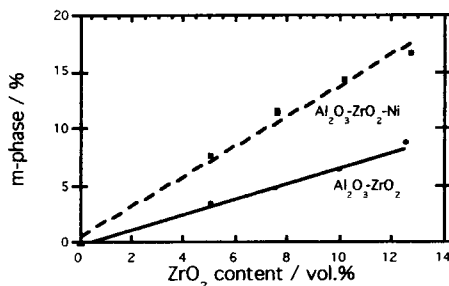


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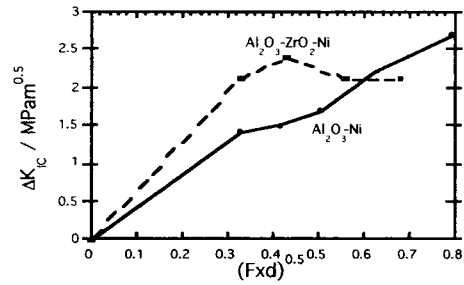


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# 訪材料複合新技術國家重點實驗室

## 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

材料複合新技術國家重點實驗室於 1987 年設立，目前有 23 位教授(8 位博士導師，15 位教授)，38 位碩士研究生，32 位博士研究生，6 位博士後研究。實驗室擁有先進的陶瓷製程設備，例如：Spark Plasma Sintering (SPS)設備，熱壓(HP)高溫爐，熱均壓(HIP)高溫爐，Self-Propagation Sintering (SHS)製粉設備，流化床製粉設備，真空燒結爐，微波反應爐，化學氣相沉積設備等。

材料複合新技術國家重點實驗室在 SHS 製程方面與俄國國家實驗室合作，在梯度材料(Functional Gradient Materials) 與日本東北大學等單位合作，在這兩個研究方向上，研究成果具世界領先水準。

## 2.2 訪問內容及心得

Spark Plasma Sintering (SPS)設備為日本 Sumitomo 公司開發製造，Spark Plasma Sintering (SPS)設備可在燒結時同時加上一個高電壓，此高電壓為交流變化，且正負向的持壓時間可以調整，以筆者所知，電壓對燒結的影響至今仍不清楚，相關研究仍極少。但材料複合新技術國家重點實驗室在這方面已有許多經驗及成果，例如：他們可將氧化鋁在 1250C 左右緻密化，他們在硼化物，氮化物，碳化物等材料的製備亦有許多經驗。

筆者到訪時，他們做了充份的準備及安排，訪問時程如附件一。

此次訪問共與如下人員見面並討論

袁潤章教授，武漢理工大學名譽校長，材料複合新技術國家重點實驗室主任

張聯盟教授，武漢理工大學副校長，材料複合新技術國家重點實驗室教授

傅正義教授，材料複合新技術國家重點實驗室教授，長江學者

劉寒星教授，武漢理工大學新材料研究所所長，材料複合新技術國家重點實驗室教授

趙修建教授，材料複合新技術國家重點實驗室教授，長江學者

潘牧教授，武漢理工大學新材料研究所副所長，材料複合新技術國家重點實驗室副教授

張東明教授，材料複合新技術國家重點實驗室副教授

曹鳳婷教授，武漢理工大學外事處主任

筆者於 11 月 9 日在材料複合新技術國家重點實驗室以[Toughening Ceramics with Two Toughening Agents]為題演講,約有教授及學生 40 多人參加,演講後並與在場學生討論,約費時兩個小時,然後由材料複合新技術國家重點實驗室主任袁校長簡介實驗室概況並參觀實驗室,最後與實驗室教授討論可實質合作項目.達成共識如下

- a. 筆者可將納米粉寄交傅正義教授,由張東明教授將納米粉以最適當條件用 SPS 製備成納米多相材料.
- b.在各自研究基礎上,開展  $Al_2O_3/Ni/ZrO_2$ ,  $TiB_2$ ,  $WC/Co$  複合材料的製備原理及分析測試.
- c.研究成果共同署名,署名第一的單位為成果產權的主要擁有者.
- d. 雙方各自向相關部門申請研究經費.

詳細共識內容請見附件二

此次訪問可稱的上是成果豐碩.

### 3. 第二屆中國國際高性能陶瓷研討會參加會議心得

中國國際高性能陶瓷研討會每三年舉行一次,今年因受美國 911 事件影響,美國及歐洲來參加的人較少,但仍有十幾個國家約 200 人參加此盛會.

筆者除發表一個邀請演講外(題目: Toughening Ceramics by Adding Two Toughening Agents),並擔任一場研討會的主持人(Session Chair).部份時程表如附件三.

這次大陸方面參加人員最多,且大多數學校及研究單位皆有代表參加,一般而言,大陸一流大學,如:清華大學,浙江大學,交通大學的水準很好,中國科學院的研究成果也很好.而研究領域則含蓋所有目前世界研究主要方向.與筆者研究領域相關的研究,最值得注意的是山東大學材料系的研究成果,他們發表一系列的研究成果,結果顯示他們所製備的  $Al_2O_3-FeAl$  複合材料具有優異的機械性質,他們目前已經設廠生產這種材料所製備的車削刀具,且獲得獎助.

此次會議記錄在經 peer review 後，將以專輯方式發表，筆者的論文已被接受(請見附件四)。

參加此次會議，收穫也很多。

#### 4. 綜合心得

大陸於近幾年因經濟條件改善，目前已開始注意教育及研發，經費正大幅增加中，大陸研究人員眾多，且多勤奮，固假以時日，研究水準將快速超過國內水準。兩岸合作又其必要性及迫切性。

#### 5. 附件

附件一，訪材料複合新技術國家重點實驗室時程表

附件二，與材料複合新技術國家重點實驗室合作共識內容

附件三，第二屆中國國際高性能陶瓷研討會部份時程表

附件四，筆者於第二屆中國國際高性能陶瓷研討會發表論文(已接受)

### 台湾大学段维新教授来校访问安排

时间		活动内容	地点	参加人员	备注	
11月8日	13:30	天河机场接机 CZ3076		张东明	外办派车	12:30
	18:00	学校宴请	太子酒轩 3楼208	张联盟、袁润章、傅正义、 刘韩星、赵修建、曹凤婷、 潘牧、张东明等	外办派车 接送段教授	15:40
11月9日	9:30	段维新教授讲学: 利用金属相增韧陶瓷	六楼报告厅	新材所研究人员及学生	外办派车 送至新材所	8:40
	14:30	参观国家实验室及 讨论合作事宜	401	袁润章教授、傅正义教授 刘韩星教授、潘牧教授等		
	18:00	新材所宴请	大富豪	新材所领导、外事处领导	外办派车 送至湖滨花园	
11月10日		观光	东湖、磨山、黄鹤楼		外办负责 用车、用餐、门票	9:00
11月11日	8:05	离汉		张东明	外办派车	6:00

住宿：湖滨花园酒店

外办联系人：曹凤婷    Tel: 1360-7124012  
 新材所联系人：潘牧    Tel: 135-07116428

国立台湾大学材料研究所和

武汉理工大学材料复合新技术国家重点实验室

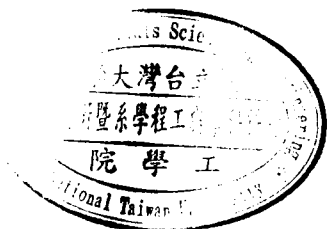
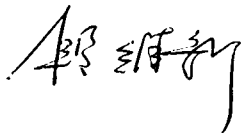
关于联合开展合作研究的协议

台湾大学材料研究所是从事复合材料研究的知名单位，在陶瓷、金属及其复合材料的研究领域开展了大量工作，取得多项研究成果；武汉理工大学材料复合新技术国家重点实验室在原位复合技术、梯度复合技术及纳米复合技术领域的研究独具特色，也拥有一批国内领先的研究成果。2001年11月8日台湾大学材料研究所段维新教授对武汉理工大学材料复合新技术国家重点实验室进行了访问，达成如下共识：

1. 同意在各自研究工作的基础上，开展  $Al_2O_3/Ni$ 、 $TiB_2$ 、 $WC-Co$  纳米材料制备原理、制备工艺及分析测试的合作研究。
2. 采取的合作方式包括：
  - (1) 互为对方提供设备条件，包括样品制备、分析测试等
  - (2) 互派研究人员或研究生
3. 研究成果共同署名，署名第一的单位为成果产权的主要拥有者。
4. 双方通过各自上级有关部门申请合作研究经费。

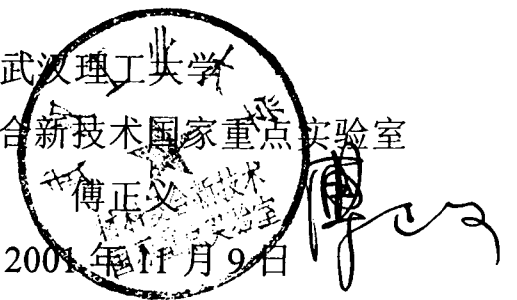
国立台湾大学  
材料研究所  
段维新

2001年11月9日



武汉理工大学  
材料复合新技术国家重点实验室

傅正义  
2001年11月9日



- 17:00 (D014O) "Thermal properties of cobalt cadmium ferrites," E. E. Assem, A. M. Abden and O. M. Hemada (Faculty of Education Kafr El-Sheikh, Egypt)
- 17:20 (D015O) "Low thermal expansion behavior of Al<sub>2</sub>TiO<sub>5</sub>-ZrTiO<sub>4</sub> ceramics having high thermal durability between 750 and 1400 h," I. J. Kim, K. S. Lee, G. Z. Cao (Hanse University, Korea)
- 17:40 (C049O) "Application, development and prospects of aftertreatment technology in conventional abrasive materials," M. N. Jiang, W. Pan and D. G. Chen (Tsinghua University, China)

**Morning, Nov. 13: Oral Presentations**

**Session A: Dielectric Properties (2)**

Chair: **G. Y. Meng**  
(USTC, China)

Room A

- 08:00 (A007O) "Dielectric properties of doped BaTiO<sub>3</sub> and Ba<sub>1-x</sub>Sr<sub>x</sub>TiO<sub>3</sub> ceramics under DC bias," J. Q. Zhao, L. T. Li, T. Li and Z. L. Gui (Tsinghua University, China)
- 08:20 (A008O) "Effects of Sr<sub>2</sub>Nb<sub>2</sub>O<sub>7</sub> addition on mechanical and dielectric properties of LaAlO<sub>3</sub> ceramics," X. Q. Liu and X. M. Chen (Zhejiang University, China)
- 08:40 (A009O) "Preparation of fine ceramic Ba<sub>0.65</sub>Sr<sub>0.35</sub>TiO<sub>3</sub> and their dielectric properties," W. Yang, A. M. Chang, J. W. Zhuang and B. C. Yang (Xinjiang Institute of Physics, China)
- 09:00 (A016O) "Microwave ceramics of La-Ba-Ti system for wireless technology," Z. X. Xiong, J. R. Huang and W. K. Hu (Xiamen University, China)
- 09:20 (A011O) "Dielectric characteristics of tungsten-bronze structured Sr<sub>x</sub>Ba<sub>1-x</sub>Nb<sub>2</sub>O<sub>6</sub> ceramics," M. S. Kim, J. H. Lee, J. J. Kim, H. Y. Lee and S. H. Cho (Kyungpook National University, Korea)
- 09:40 Break

**Session A: Non-Ohmic Conductors**

Chair: **S. Tian**  
(BUAA, China)

Room A

- 09:50 (A058I) "Advanced composite ceramic electrolytes for solid oxide fuel cells," B. Zhu (Royal Institute of Technology, Sweden)
- 10:15 (A059I) "A novel ceramic material La<sub>1-x</sub>Sr<sub>x</sub>InO<sub>3</sub> and single layer solid oxide fuel cell," L. Q. Chen, H. P. He and X. J. Huang (Institute of Physics, China)
- 10:40 (A060O) "The key materials for reduced temperature ceramic fuel cells," G. Y. Meng, Q. X. Fu, C. R. Xia, S. W. Zha, R. R. Peng and D. K. Peng (University of Science and Technology of China, China)
- 11:00 (A061O) "Super-ionic conductors Ca<sup>2+</sup> and La<sup>3+</sup> β-alumina," Y. R. Hong, C. J. Jin and J. L. Sun (University of Science and Technology Beijing, China)
- 11:20 (A062O) "Characterization and sensing properties of a zirconia based resistive oxygen sensor," W. Cao, O. K. Tan, W. Zhu, B. Jiang and J. S. Pan (Nanyang Technological University, Singapore)
- 11:40 (A063O) "Study on oxygen-sensing properties of LaNiO<sub>3</sub> thin film," F. Hou, T. X. Xu and M. X. Xu (Tianjin University, China)

**Session A: Piezoelectrics and Electrostrictives**

Chair: **X. H. Wu**  
(Yunnan University, China)

Room A

- 08:00 (A037O) "Piezoelectric properties of (1-x)PMN-xPT (x = 0.31, 0.33 and 0.35) single crystals," P. C. Wang, X. M. Pan, D. L. Li, Y. W. Song, H. S. Luo and Z. W. Yin (Shanghai Institute of Ceramics, China)
- 08:20 (A038O) "Co-firing behavior and interfacial structure of dielectric/ferrite composites for multilayer LC filters," M. Wang, Z. X. Yue, L. T. Li, J. Zhou and Z. L. Gui (Tsinghua University, China)
- 08:40 (C038O) "Fabrication of ceramic polymer composite by Nd: YAG laser," D. Guo, C. W. Nan, L. T. Li and Z. L. Gui (Tsinghua University, China)
- 09:00 (A039O) "Electric aging behavior and mechanism of PMMN quaternary piezoelectric ceramics," H. Y. Chen, X. B. Guo and Z. Y. Meng (Shanghai University, China)
- 09:20 (A041O) "Influence of temperature on electric fatigue of 0.3PZN-0.7PZT piezoelectric ceramics," S. Tian (Beijing University of Aeronautics and Astronautics, China)
- 09:40 (A042O) "Temperature dependence of electromechanical properties for PMS-PZ-PT ceramic doped with Sr<sup>2+</sup>," X. B. Guo, H. Y. Chen and Z. Y. Meng (Shanghai University, China)
- 10:00 Break

**Session A: PTC Effects**Chair: **C. D. Feng**

Room B

*(Shanghai Institute of Ceramics, China)*

- 10:20 (A0200) "Characteristics of grain boundaries of PTC ceramics under DC bias," Y. G. Li and S. G. Cho (*Gyeongsang National University, Korea*)
- 10:40 (A0210) "Graphite/resin/BaTiO<sub>3</sub> based PTCR composite material," Y. F. Qu, J. B. Lu and W. B. Ma (*Tianjin University, China*)
- 11:00 (A0220) "The relationship between PTCR effect and donor content in Yb-doped BaTiO<sub>3</sub> ceramics," J. Q. Qi, Z. L. Gui, Y. L. Wang and L. T. Li (*Tsinghua University, China*)
- 11:20 (A0230) "Lower the room temperature resistance of PTC type barium titanate ceramics by multilayer structure," W. B. Ma, Y. F. Qu and C. X. Fang (*Tianjin University, China*)
- 11:40 (A0240) "Study on double-donor-doped and o-site displaced BaTiO<sub>3</sub>-base PTC Ceramic materials," J. L. Ma and Y. F. Qu (*Tianjin Urban Construction Institute, China*)

**Session B: Ceramic-Matrix Composites (1)**Chair: **Y. F. Chen**

Room C

*(Institute of Chemical Metallurgy, China)*

- 08:00 (B033I) "Toughening ceramics by adding two toughening agents," W. H. Tuan and J. K. Guo (*National Taiwan University, Taiwan*)
- 08:25 (B035O) "Characterization of interphase structure by HREM in 3D carbon fiber/silicon carbide composites prepared by LPCVI," J. Z. Li, D. X. Li, L. T. Zhang, X. M. Meng, Y. D. Xu, L. F. Cheng and H. F. Yin (*Northwestern Polytechnic University, China*)
- 08:45 (B036O) "CVI fabrication and bending properties of SiC<sub>f</sub>/SiC composites with different interfacial configurations," Y. Shi, H. Araki, W. Yang and T. Noda (*Shanghai Institute of Ceramics, China*)
- 09:05 (B043O) "Effects of additives to BN interface layers on microstructure and properties of Si<sub>3</sub>N<sub>4</sub>/BN laminated ceramic composites," C. W. Li, Y. Huang, C. A. Wang, *et al.* (*Tsinghua University, China*)
- 09:25 (B047O) "On the ceramic fibers used in metal matrix composite," H. F. Zhao, H. M. Liu and J. F. Qian (*Xian Jiaotong University, China*)
- 09:45 Break

**Session B: Ceramic-Matrix Composites (2)**Chair: **W. H. Tuan**

Room C

*(National Taiwan University, Taiwan)*

- 09:55 (B038I) "Attempts on the new processing of Ni/Al<sub>2</sub>O<sub>3</sub> composite ceramics," Y. F. Chen, L. K. Zang, Z. J. Wu, J. H. Gong and Y. S. Xie (*Institute of Chemical Metallurgy, China*)
- 10:20 (B041O) "Electron structure research and interface design on the Al<sub>2</sub>O<sub>3</sub>/Fe<sub>3</sub>Al nano-composites," J. Li, Y. C. Liu, H. U. Gong, Y. S. Yin (*Shandong University, China*)
- 10:40 (B039O) "Investigation of fabrication processing properties of Fe<sub>3</sub>Al/Al<sub>2</sub>O<sub>3</sub> composites," H. Y. Gong, Y. S. Yin, Y. C. Liu, J. Li and J. S. Zhang (*Shandong University, China*)
- 11:00 (B040O) "The influence of Cr on fracture toughness and microstructure of Fe-Al/Al<sub>2</sub>O<sub>3</sub> composites," A. M. Li, K. N. Sun, Y. J. Zhang, Y. S. Yin and R. Liu (*Shandong University, China*)
- 11:20 (B045O) "Adjusting the purity and toughness of the Al<sub>2</sub>O<sub>3</sub>/Ti<sub>3</sub>SiC<sub>2</sub> multilayer materials by SiC coating," Q. F. Zan, Y. Huang, C. A. Wang, S. Q. Li and C. W. Li (*Tsinghua University, China*)
- 11:40 (B034O) "Effect of Na<sub>2</sub>O on the mechanical properties of MoSi<sub>2</sub>/oxide composites," S. K. Zhao, G. Wang and W. Jiang (*Shanghai Institute of Ceramics, China*)

**Session C: Polymers in Particulate Systems**Chair: **L. Gao**

Room D

*(Shanghai Institute of Ceramics, China)*

- 08:00 (C030O) "Development of new gelcasting system with low-toxicity," K. Cai, Y. Huang and J. L. Yang (*Tsinghua University, China*)
- 08:20 (C031O) "Freeform fabrication of aqueous alumina-acrylamide gelcasting suspensions," Z. Y. Yu, Y. Huang, D. J. Zhang, R. J. Zhang, X. M. Cui and S. X. Ouyang (*Tsinghua University, China*)
- 08:40 (C032O) "Surface adsorption and dispersion of BaTiO<sub>3</sub> colloids with PDAAE," B. Y. Yu and W. C. J. Wei (*National Taiwan University, Taiwan, China*)
- 09:00 (C033O) "Ion dissolution and property change of aqueous PZT suspension with PDAAE," W. T. Hsu and W. C. J. Wei (*National Taiwan University, Taiwan, China*)
- 09:20 (C034O) "Suppression of surface-exfoliation by gelcasting ceramics with mixed polymer-monomer solutions," Z. P. Xie, J. T. Ma, H. Z. Miao, Y. Huang, Y. B. Cheng (*Tsinghua University, China*)



# Toughening Ceramics by Adding Two Toughening Agents

Wei-Hsing Tuan<sup>\*,1</sup> and Jingkun Guo<sup>2</sup>

<sup>1</sup>Institute of Materials Science & Engineering, National Taiwan University, Taipei,

<sup>2</sup>Shanghai Institute of Ceramics, Chinese Academy of Science, Shanghai

**Keywords:** composite, toughening, synergy, alumina, zirconia, metal

**Abstract:** The applications of ceramics for structural components are often limited by its brittleness. The toughness of ceramics can be improved by adding one toughening agent, such as zirconia particles or metallic particles. The present study explores the toughening behavior of the composites containing two toughening agents. Our results suggest that the toughness of ceramics can also be improved significantly as two toughening agents are added simultaneously into them. In the present study, both metallic (silver or nickel) inclusions and zirconia inclusions are added into alumina. The processing design, microstructural evolution and the mechanical properties of the Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub>-Ag and Al<sub>2</sub>O<sub>3</sub>-ZrO<sub>2</sub>-Ni composites are investigated. The metallic inclusions can bridge an advancing crack and exert crack closure stresses on the crack wake. The toughness of alumina can also be enhanced by the phase transformation of zirconia. Furthermore, the presence of the metallic inclusions increases the phase transformation extent of the zirconia particles, indicating that the toughening effect provided by the zirconia and metallic inclusions can interact with each other. Therefore, a synergy effect; namely, the toughness increase for the composites containing two ceramic particles and metallic inclusions is higher than the additive of the toughness increase of the composites containing only one reinforcement, is observed.

## Introduction

The applications of ceramics as engineering components are often limited by their brittleness. To improve the toughness of ceramics is therefore a challenging task for most ceramists. The addition of second-phase inclusions that influence the propagation of cracks has been one much-studied approach. Among the second-phase inclusions studied, zirconia particles [1,2] and metallic particles [3,4] have received great attention.

The presence of metallic inclusions can bridge the flaws by the ductile phase in the wake zone behind the crack tip [3,4]. The phase transformation of zirconia can induce stresses to prohibit the opening of crack surfaces [1,2]. The toughness of a zirconia-toughened composite,  $K_{IC,c}$ , is composed of matrix toughness,  $K_{IC,matrix}$ , and a toughness enhancement,  $\Delta K_{IC}$ , attributed to the phase transformation as

$$K_{IC,c} = K_{IC,matrix} + \Delta K_{IC} \quad (1).$$

The phase transformation of zirconia mainly takes place in a region, process zone, near an advancing crack, where the constraint applied by the rigid matrix is diminished. The toughness enhancement is also a function of matrix toughness, Eq.(1) can thus be expressed as [5],

$$K_{IC,c} = K_{IC,matrix} + C * E * F * K_{IC,matrix} \quad (2),$$

where C, E and F are a constant, elastic modulus and volume fraction, respectively. The above equation implies that the toughness of a ceramic can be significantly increased by choosing a toughened matrix. The toughened matrix can be a composite, it thus implies that the toughness of

ceramics can be significantly enhanced through the addition of two toughening agents simultaneously (one toughening agent has to be transformable  $ZrO_2$ ). In the present study, *t*- $ZrO_2$  and metallic particles, Ni or Ag, are added into an  $Al_2O_3$  matrix. The toughness of the composites are determined.

## Experimental

Alumina (TM-DR, Taimei Chem. Co. Ltd., Tokyo, Japan), nickel oxide (NiO, Johnson Matthey Co., U.S.A.) or silver oxide ( $Ag_2O$ , Johnson Matthey Co., U.K.) and zirconia (TZP,  $ZrO_2 + 3$  mol.%  $Y_2O_3$ , Hanwha Ceramics Co., Australia) powders were ball milled together in ethyl alcohol for 24 hours. The volume content of zirconia added was controlled to be the same as that of the resulting metals. The slurry of the powder mixtures was dried and subsequently sieved. Powder compacts were prepared by uniaxially pressing at 44 MPa. The sintering was carried out at 1600C for 1 hour. The heating rate and cooling rate were 5C/min. The  $Al_2O_3$ - $ZrO_2$ -Ni composites were sintered in a CO atmosphere. Nickel oxide would reduce to nickel during sintering. The  $Al_2O_3$ - $ZrO_2$ -Ag composites were sintered in air,  $Ag_2O$  would decompose to result in Ag during the heating stage [6]. Some  $Al_2O_3$ - $ZrO_2$  composites were also prepared with the same techniques for comparison purpose. The sintered specimens were machined longitudinally with a 325 grit resin-bonded diamond wheel at a cutting depth of 5  $\mu$ m/pass. The final dimensions of the specimens were 3 x 4 x 36 mm.

The strength of the specimens were determined by using the 4-point bending technique. The upper and lower spans were 10 mm and 30 mm, respectively. The fracture toughness was determined by the single-edge-notched-beam (SENB) technique. Phase identification of sintered and machined specimens was performed by X-ray diffractometry (XRD). The relative phase content of zirconia was estimated by using the method proposed by Evans et al [7]. The final density of the specimens was determined by the Archimedes method. The solubility between the materials used in the present study was low, the relative density of the sintered composites was thus estimated by using the theoretical density of 3.98 g/cm<sup>3</sup> for  $Al_2O_3$ , 6.05 g/cm<sup>3</sup> for  $ZrO_2$ , 8.90 g/cm<sup>3</sup> for Ni and 10.5 g/cm<sup>3</sup> for Ag. Nickel and silver could vaporize during sintering, the volume fraction of Ag in the specimens after sintering was determined by counting the point fraction on the polished surfaces. Microstructural characterization used scanning electron microscopy (SEM). The size of metallic inclusions and alumina matrix grains was determined by applying the line intercept technique. The interconnectivity of metallic inclusions in the alumina matrix was determined by measuring the electrical resistivity at room temperature.

## Results and Discussion

XRD analysis reveals  $\alpha$ - $Al_2O_3$ , *t*- $ZrO_2$ , Ni or Ag in the sintered  $Al_2O_3$ - $ZrO_2$ -Ni or  $Al_2O_3$ - $ZrO_2$ -Ag composites, respectively. No monoclinic  $ZrO_2$  phase was detected on the surface of the composites. The NiO and  $Ag_2O$  are fully reduced to their metallic form after sintering. Fig. 1 shows the microstructures of  $Al_2O_3/15\%ZrO_2$ ,  $Al_2O_3/(15\%ZrO_2+15\%Ni)$  and  $Al_2O_3/(15\%ZrO_2+15\%Ag)$  composites. The ceramic and metallic inclusions are distributed uniformly within the alumina matrix. The initial size of nickel and silver oxide particles is larger than that of zirconia particles. The size of metallic inclusions is thus larger than that of ceramic inclusions. The metallic and zirconia inclusions on the micrographs can thus be roughly identified by their size difference.

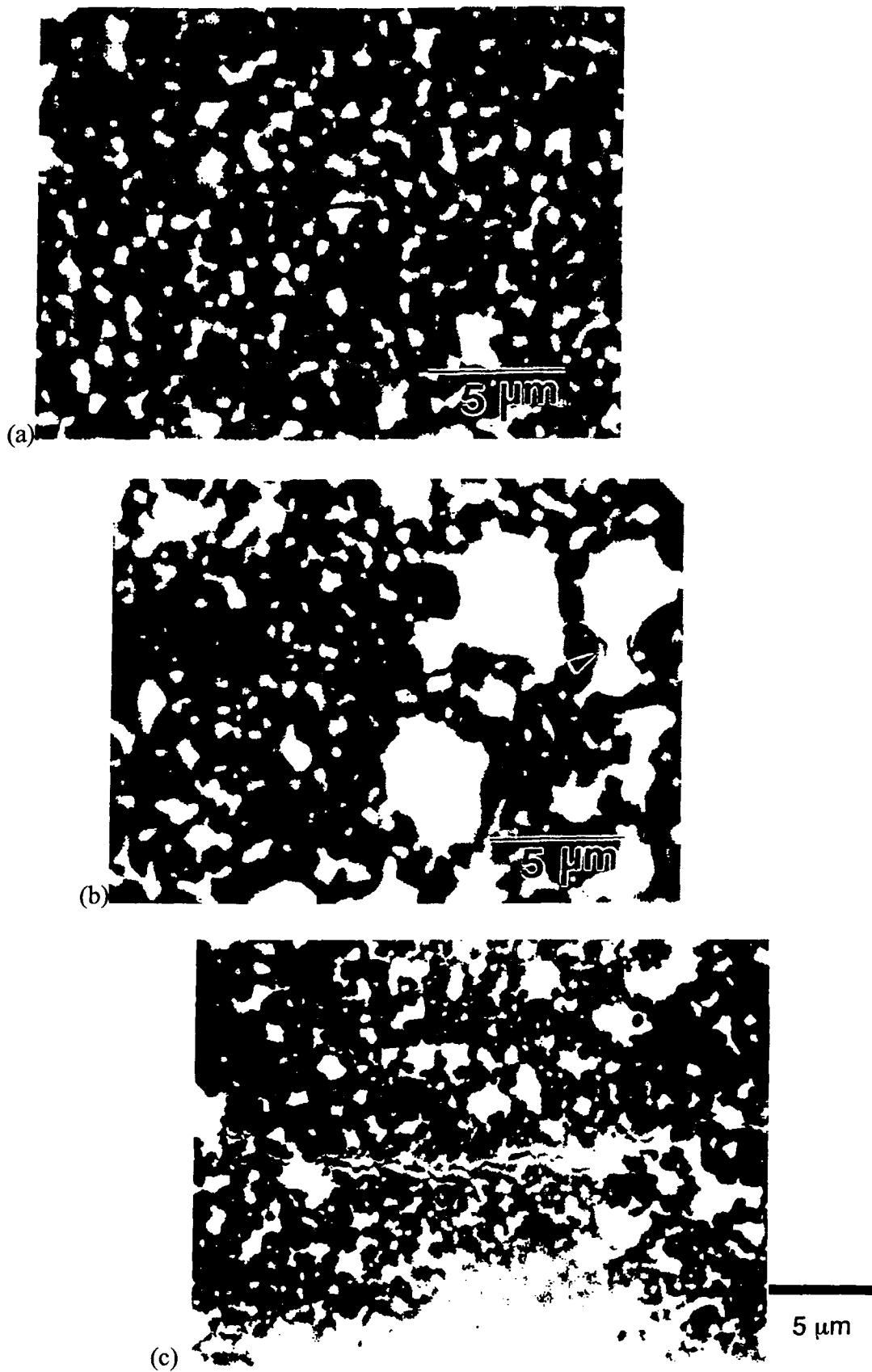


Fig. 1, The microstructures of  $\text{Al}_2\text{O}_3\text{-ZrO}_2$ ,  $\text{Al}_2\text{O}_3/(\text{ZrO}_2+\text{Ni})$  and  $\text{Al}_2\text{O}_3/(\text{ZrO}_2+\text{Ag})$  composites.

Fig. 2 shows the relative density of the composites as a function of total inclusion content. There is a little solubility of zirconia in alumina, the presence of Zr solutes prohibits the densification of alumina [8]. The presence of the metallic inclusions prohibits the densification of composites. It may result from the poor wetting of the metallic melt on alumina [9,10]. The density of the composites thus decreases with the increase of total inclusion content.

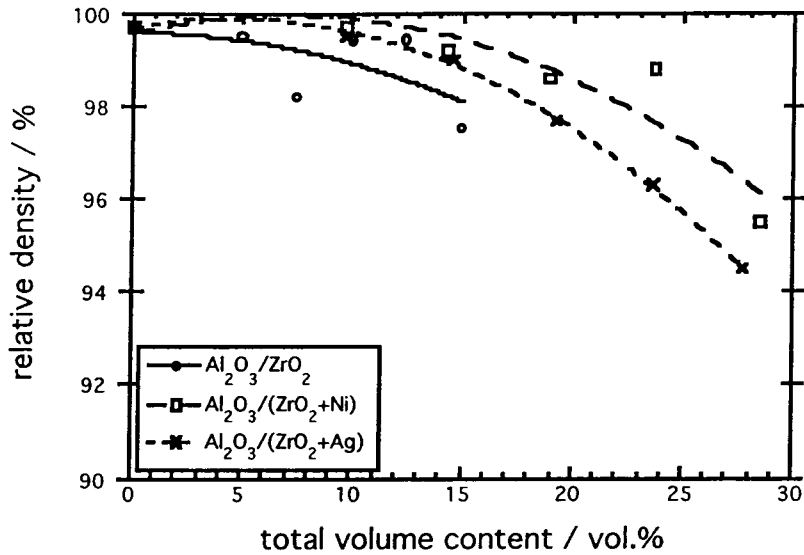


Fig. 2, Relative density of composites as function of total inclusion content.

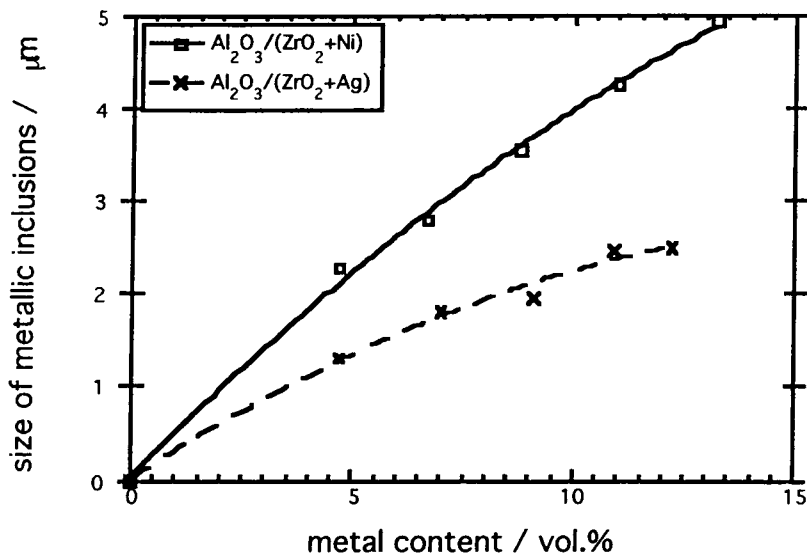


Fig. 3, Size of metallic inclusions as a function of metal content.

In the composites containing two toughening agents, the added volume fraction of Ni or Ag was the same as that of  $ZrO_2$  in the beginning. Ni or Ag vaporizes during sintering; the maximum metallic content is thus slightly lower than 15vol.%. The electrical resistivity of the metal-containing composites is higher than  $10^{14} \Omega\cdot m$ , indicating that the metallic particles are isolated from each other within the alumina matrix. Fig. 3 shows the size of metallic inclusions within alumina matrix as a function of metal content. The figure indicates that the coalescence of metallic inclusions can take place during sintering despite the metallic particles are separated from each other. Fig. 4 shows the grain size of alumina matrix, the matrix grains is significantly reduced in their size as the zirconia and metallic inclusions are incorporated. The presence of the inclusions, either zirconia or metals, prohibits the movement of alumina grain boundaries. The microstructures of composites is thus refined.

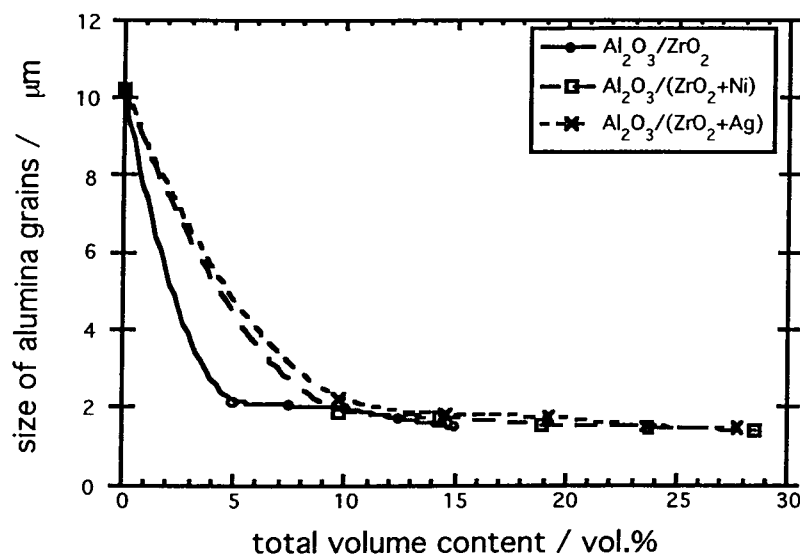


Fig. 4, Grain size of alumina matrix as function of total inclusion content.

Fig. 5 shows the strength of composites as a function of total inclusion content. The strength of alumina is enhanced by adding both ceramic and metallic inclusions. The microstructure of composites is refined due to the addition of zirconia and metallic inclusions. The strength enhancement can thus attribute to the microstructural refinement.

Fig. 6 shows the toughness of composites as a function of total inclusion content. The toughness of the composites containing two toughening agents is higher than that of  $Al_2O_3-ZrO_2$  composites. Furthermore, the toughness of  $Al_2O_3-ZrO_2-Ni$  composites is higher than that of  $Al_2O_3-ZrO_2-Ag$  composites.

The metals are ductile, relative to ceramics, in nature. They deform plastically during the opening of crack surfaces, as demonstrate in Fig. 1(b). The strain energy induced by the plastic deformation raises the toughness of the metal-containing composites. The strain energy of the constrained metallic particles in rigid matrix is proportional to their size [11]. The high toughness of the nickel-containing composites may attribute partly to the larger size of the nickel inclusions.

XRD analysis detects only tetragonal  $ZrO_2$  phase on the surface of the sintered  $Al_2O_3-ZrO_2$ ,  $Al_2O_3-ZrO_2-Ni$  and  $Al_2O_3-ZrO_2-Ag$  composites. Fig. 7 shows the percentage of phase transformation of  $ZrO_2$  on the fracture surface as a function of zirconia content. On the fracture surface, some t- $ZrO_2$  particles in the composites containing two toughening agents are transformed to m-phase. Fig. 7 demonstrates the same trend as the results shown in Fig. 6. It indicates that the toughness enhancement of the composites is contributed partly by transformation toughening. The presence of metals lowers the elastic modulus of composites, less constraint is thus imposed on  $ZrO_2$  particles. More phase transformation in the metal-containing composites takes place during fracturing process, higher toughness is therefore resulted. Furthermore, the amount of monoclinic phase is higher on the fracture surface of  $Al_2O_3-ZrO_2-Ni$  composite, the toughness of the composite is thus high.

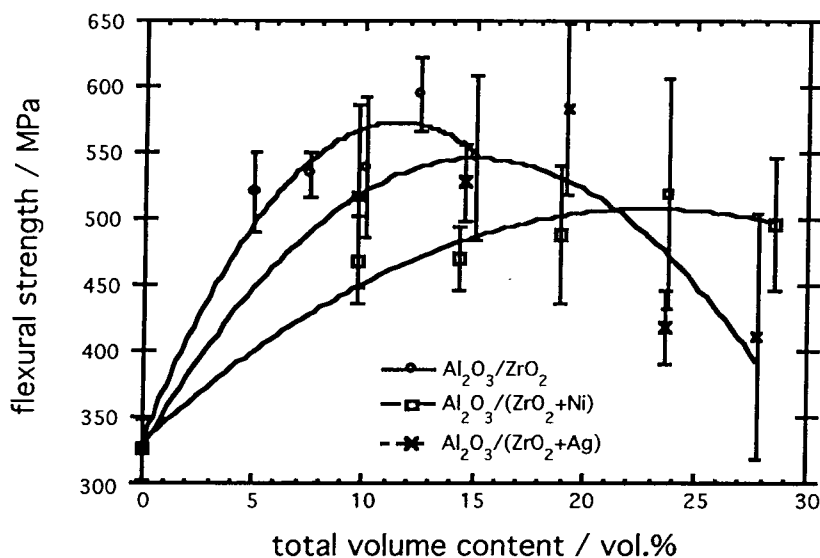


Fig. 5, Strength as composites as function of total inclusion content.

## Conclusions

Both zirconia and metallic particles were added into an alumina matrix. The strength and toughness of the composites containing two toughening agents are higher than that of matrix alone and of  $Al_2O_3-ZrO_2$  composites. The strengthening effect is contributed by microstructural refinement. More tetragonal zirconia particles are transformed into monoclinic phase due to the presence of metals. The toughness of the composites containing both zirconia and metals is therefore high. Furthermore, as the metal content is lower than 15 vol.%, as the cases investigated in the present study, the electrical resistivity of the composites is high. It suggests that the composites can be applied as electrical insulators; nevertheless, with better strength and toughness.

## References

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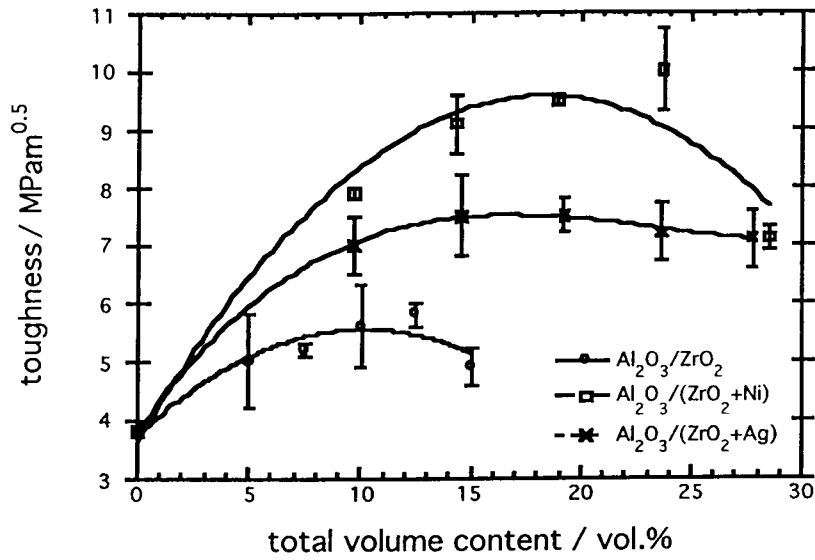


Fig. 6, Toughness of composites as function of total inclusion content.

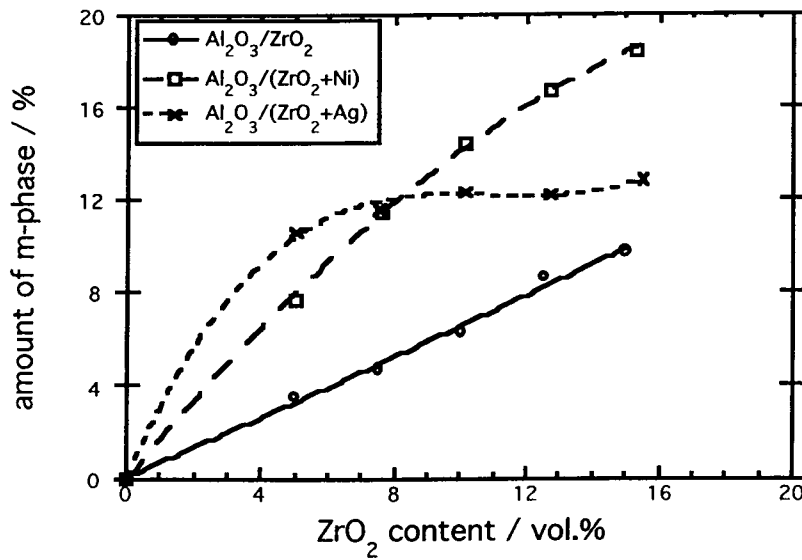


Fig. 7, Percentage of phase transformation of zirconia as function of zirconia content.

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