行政院國家科學委員會專題研究計畫 成果報告

金屬/陶瓷界面工程：金屬/陶瓷界面改質
研究成果報告(精簡版)

計畫類別：個別型
計畫編號：NSC 95-2218-E-002-043-
執行期間：95年02月01日至96年07月31日
執行單位：國立臺灣大學材料科學與工程學系暨研究所

計畫主持人：段維新

計畫參與人員：博士班研究生-兼任助理：李炤佑、郭書廷、靳元良

報告附件：出席國際會議研究心得報告及發表論文
國際合作計畫研究心得報告

處理方式：本計畫可公開查詢

中華民國 96年08月01日

行政院國家科學委員會補助專題研究計畫 **v□v 成果報告**
□期中進度報告

金屬/陶瓷界面工程：金屬/陶瓷界面改質

計畫類別：v□v 個別型計畫(雙邊國際合作研究計畫)
計畫編號：NSC 95－2218－E－002－043－
執行期間：95年02月01日至96年07月31日

計畫主持人：段維新
共同主持人：Michel Dupeux
計畫參與人員： 李炤佑，郭書廷，靳元良

成果報告類型(依經費核定清單規定繳交)：v□v精簡報告　□完整報告

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□赴國外出差或研習心得報告一份
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v□v出席國際學術會議心得報告及發表之論文各一份
v□v國際合作研究計畫國外研究報告書一份

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□涉及專利或其他智慧財產權，□一年□二年後可公開查詢

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中華民國96年08月01日

行政院國家科學委員會專題研究計畫成果報告

金屬/陶瓷界面工程：金屬/陶瓷界面改質

計畫編號 ：NSC 95-2216-E-002 -043
執行期限 ：95 年 2 月 1 日 至 96 年 7 月31 日
主持人: 段維新 教授
共同主持人: Michel Dupeux 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: 李炤佑, 郭書廷, 靳元良

一. 摘要

The adhesion strength between a porous silver coating and pure barium titanate has been determined using a blister test. The porous Ag layer was made by screen printing and sintering on a flat dense barium titanate surface. Firing temperature influences the porosity of the silver film and Ag/$BaTiO_3$ interface strength. A simple graphic procedure was used to subtract the effect of the generalized plastic yielding in the Ag membrane from the total strain energy produced by the pressure application. The average value of the critical interface crack propagation energy G_{ci} at the porous Ag/$BaTiO_3$ interface ranges between 4.5 J/m^2 and 6.6 J/m^2 while the porosity decreases from 11% to 7%.

Keywords: Adhesion, Ag/barium titanate interface, porosity, interfacial crack propagation energy

本研究探討陶瓷材料與多孔金屬材料之間界面的接合，此界面的強度以Blister方法量測，材料系統為鈦酸鋇與以網版印刷製備的多孔銀電極，孔隙率的高低影響界面強度，鈦酸鋇/多孔銀的裂縫傳遞能量從4.5 J/m^2 增加至6.6 J/m^2 當孔隙率由11%減至7%.

關鍵字： 鈦酸鋇/銀, 界面強度, 裂縫傳遞能量, 孔隙率

二. 緣由及目的

Metal–ceramic interfaces play an important role in many applications, such as electronic packages, ceramic brazing, wear resistant coatings, thermal barrier coatings or micro-electromechanical systems. The metal/ceramic interfacial strength is an important parameter for all applications since it fully governs the integrity of the device of interest. Among many techniques, screen printing and sintering is a low cost method to make a metal film bonded to a ceramic substrate. It is generally used to fabricate multilayer ceramic capacitors (MLCC), sensors, device structure, etc. This technique produces a porous metal layer; the porosity of the metal film depends on the various chemical and physical characteristics of the metal paste and on its firing temperature.

Many different methods have been proposed to measure the adhesion strength of the metal–oxide interface, either as bulk bi-material or as film/substrate systems [1, 2]. These methods, however, are very specific and often their results are mutually inconsistent. The blister test is one of the few techniques which can deliver quantitative and meaningful estimation on interfacial strength. It allows reliable quantitative interfacial fracture energy measurements thanks to a stable interfacial crack growth under a constant and well known mode mixity for film/substrate systems [3, 4]. For this test, a rigid film/substrate specimen has to be prepared with a free-standing film membrane covering a hole located at the center of the specimen. During a first step of the experiment, the free-standing membrane is pressurized by liquid injection through the hole in the substrate, keeping the contour of the initial window. This "bulge" step is reported as an increasing curve when plotting the pressure p vs. the membrane maximum deflection h. The elastic bulging of the membranes can be analyzed using various models to describe their mechanical equilibrium and geometry [5-10].

When the pressure is high enough and the liquid injection is continued, the film starts to debond along the edges of the window. The outline of the membrane changes from its initial shape and its volume increases rapidly, thus causing a decrease of the fluid pressure and a visible change of regime on the p vs. h curve. For an elemental interfacial crack growth da, with fracture energy G_i per unit area, the balance between the increase of the fluid energy

W_{fluid} on one hand, the membrane increase of the strain energy W_{strain} plus the crack propagation energy $G_i.2pa.da$ can be written as:

$$G_i.2\pi a.da = \frac{\partial W_{fluid}}{\partial a}da - \frac{\partial W_{strain}}{\partial a}da \qquad (1)$$

Estimates of each term of equation (1) show that the p vs. h curve should then follow a decreasing hyperbolic law:

$$C . p . h = G_i \qquad (2)$$

where C is a numerical dimensionless constant between 0.618 and 0.516 according to the residual stress value in the film [11, 12].

三. 實驗步驟

2.1 Materials and specimens

A barium titanate powder (Product No. 52909, Ferro Co., USA) was used in the present study. The purity of the $BaTiO_3$ powder was higher than 99.6% and the average particle size was 1.1 μm. Discs of $BaTiO_3$ (25 mm in diameter and 2.5 mm in thickness) were prepared by die-pressing at 50 MPa. The discs were first pre-fired at 1100°C for 1 h to obtain a handling strength. A hole with 3 mm diameter was then machined into the center of the pre-fired discs by using a steel drill. The discs were then sintered at 1350°C for 2 h. The diameter of the hole shrank to 2.1 mm after firing. The relative density of $BaTiO_3$ is then > 98%. Finally the discs were ground with fine SiC particles to achieve a flat smooth surface. The final substrate surface roughness R_a is about 0.2 μm, as measured with a profilometer.

A silver paste composed of silver particles, binder and a small amount of glass particles was prepared for screen printing. A silver powder (Ag C200ED, Ferro Co., USA) with average particle size of 1.1 μm was used. The silver powder/glass frit ratio was 98.75/1.25 in weight. The powder/organic vehicle ratio was 80/20 in weight. All were mixed and milled with the help of a media mill. Screen-printing was used to apply the Ag paste onto the flat surface of the $BaTiO_3$ discs. Round adhesive tape patches 3 mm in diameter were applied on a $BaTiO_3$ plate, Ag layer was screen printed over the whole surface, then the $BaTiO_3$ plate with Ag layer and adhesive patches was fired at 500 °C for 1 h to remove the adhesive tape. Therefore, we could obtain several pre-fired silver membranes with 10 μm thickness and 3 mm diameter, which were used to cover the central hole of the discs as free-standing Ag membranes. The discs including these free-standing membranes over the holes were then screen printed with Ag electrode for several times until the desired thickness of Ag layer is achieved and fired at various temperatures, respectively 600, 700, 800 or 850°C for 1h.

2.2 Experimental apparatus

The apparatus used for the blister test consisted of a specimen holder and pressurizing stage, and an optical fringe projection profilometer. A commercial software (HOLO-3: Fringe Analysis ®) was used to analyze the results in terms of blister radius and deflection at various steps of each test. The complete setup of facilities has been described in more details in previous papers [13, 14].

For each firing temperature, some of the specimens were cold mounted and cut transversally and then prepared by standard metallographic techniques. The cross-section microstructures were observed by scanning electron microscopy (LEO 440 and JEOL 6400). Quantitative analysis of digitalized images (ImageJ 1.34s) was used to determine the porosity of the screen printed silver films.

四. 結果與討論

3.1 Microstructure observations

Figure 1 shows the cross-sections of the specimens after sintering. The $Ag/BaTiO_3$ interface appears to be relatively smooth and abrupt at the magnification we used. The thickness of the silver layer has been changed between 55 μm and 130 μm. It has been measured after sintering by micrometer, with reference to the initial substrate thickness. For each substrate and substrate with film, five points were measured and the average was taken as thickness.

Figure 1(a) shows the Ag layer fired at 600°C for 1 h. The pores within the sintered metal are interconnected and the average porosity is 11%. Figure 1(b) shows the silver film fired at 700°C. It can be observed that most of the pores are no longer interconnected and the average porosity is 10%. When the firing temperature was raised to 800°C (Figure 1(c)), the average porosity of Ag film became 8%. The shrinkage rate of Ag film from 700°C to 800°C is higher than that between 600°C and 700°C. Figure 1(d) shows the silver film fired at 850°C and the average porosity in the silver film is 7%. The size of the pore within the Ag is increased after the treatment at 850°C, though their amount is more or less the same.

For our purpose of adhesion measurement, the porosity within the $Ag/BaTiO_3$ interface is very likely to have a strong influence on the interfacial crack propagation energy, but it is very difficult to determine experimentally from optical or SEM observations. In the following, we take the interface porosity rate (defined as the ratio of the non-contacting area to the total interface area) as equal to the internal porosity in the bulk of the silver layer.

3.2 Analysis of blister test results

For many film-on-substrate systems, it is usually assumed that the top film deforms elastically. Because of its thickness, porous structure and annealed microstructure, our silver layer has a low yield stress and rather tends to deform plastically. Then the strain energy developed during the blister test is composed of the work of elastic deformation and the work of plastic deformation. They have to be separated for adhesion measurement, since only the elastic strain energy is reversible and can be released from the pressurized membrane to cause the interface crack propagation.

Experimentally, when conducting the blister test, hydraulic pressure is applied to make the metal layer blister and separate from its substrate. Then the pressure is released progressively and the p vs h curve generally falls down along a linear path and shows a residual deflection h_p, proving that the film has been plastically deformed. After the first pressurization and de-bonding, as long as the system is stable we can re-inject liquid to make blistering occur again and then release the pressure again. Therefore, each repeated pressurization can be used to determine the adhesion energy of the interface by fitting an hyperbolic curve to the debonding threshold points according to equation (2). At each pressurization, since we record successive 3-D contours of the complete blister, we can check the diameter values to determine as accurately as possible the point when the blister begins to grow and the interfacial crack begins to propagate. We defined this point as the critical point to be used for G_c determination. In many cases, we can obtain several debonding points on one specimen till the metal layer is broken or totally de-bonded. Since the Ag membrane behaviour is dominated by residual stress (due to thermal contraction mismatch between metal and ceramic after firing) rather than by high Young's modulus and stiffness, we used C = 0.516 in equation (2) for result analysis in this study [4,11].

For each re-pressurization, since the film has deformed plastically after previous pressurization, the volume underneath the new free-standing membrane can be related to its previous plastic residual deflection height h_p. The total strain energy of the pressurized film should then be changed to

$$U_{strain} = \int_0^{hp+h} p(h)dV = \int_0^{hp} p(h)dV + \int_{hp}^{h} p(h)dV \qquad (3)$$

The first term contributed by the plastic deformation should be extracted from the total apparent strain energy, to work out the elastic energy release rate which causes crack propagation. A very simple way to correct this plastic bulging of the membrane is to subtract the corresponding residual permanent deflection h_{pi} for each quasi-linear inflation-deflation elastic curve N°i at a critical point for a given specimen, as illustrated on figure 2. The critical debonding points then correspond to much smaller values of corrected deflection (h-h_{pi}) just as if the membrane had remained elastic throughout the test, and they are located on such hyperbolae that $C.p.(h-h_{pi}) = G'_i$ with much smaller and more reliable values of debonding energy.

For example, figure 3 shows the blister equation fit to the experimental data at the de-bonding point for a specimen with a silver layer fired at 850°C. The uncorrected crack propagation energy was acquired as 11 J/m^2, while the height being corrected as $h = h_{exp} - h_{pla}$, the result diminished to 4.5 J/m^2. Thanks to this very simple procedure, we can subtract the effect of the generalized plastic yielding in the Ag membrane from the total strain energy produced by the pressure application.

The resulting critical energy release rates G_{ci} of the BaTiO$_3$/Ag interfaces for each Ag paste sintering temperature are listed in Table 1. As explained before, these values are corrected by removing the influence of generalized plastic deformation of the membrane, but they still include the contribution of the confined plasticity which is likely to occur at the crack tip during the propagation of the interfacial crack, just like cohesive crack propagation energy in any ductile homogeneous metal. The values of G_{ci} for specimens which are fired at 600°C, vary from 3.5 to 5.5 J/m^2. The values of G_{ci} for interfaces with silver fired at 700°C vary from 3 to 6 J/m^2, which is close to the result of 600°C. The inter-particle contact area for sintered powders increases while firing temperature increased from 600°C to 700°C, (Figure 1(a), 1(b)). It seems that the subsequent decrease of porosity does not affect much the interface strength at this stage. The values of G_{ci} fired at 800°C vary from 3.5 to 8 J/m^2, with an overall average value around 5.8 J/m^2. At 850°C the critical energy release rate of interface cracks ranges from 4.5 to 9 J/m^2.

3.3 Discussion

Table 1 (not shown here) also shows the thicknesses of specimens fired at 600°C, 700°C and 800°C. In same firing condition, the influence of the film thickness on interface strength seems to be smaller than the experimental dispersion of the energy values. Therefore, it will not be considered in the following discussion.

The relation between average G_{ci} and sintering temperature is shown in Figure 4. The critical energy release rate of interface cracks increases when firing temperature increases, especially in the range from 700°C to 800°C. This may be due to the changes in the pore size distribution through the metal membrane and similarly along the interface.

Figure 5 shows the critical energy release rate G_{ci} of Ag/BaTiO$_3$ interfaces versus the porosity of the Ag film. The silver paste used in this study was named as G-1. Within the range of membrane porosity from 11% to 7%, the G_{ci} of the interface between porous silver membrane and BaTiO$_3$ substrate is almost a linear function of the porosity of the membrane. It is obvious that smaller contact areas across the metal/ceramic interface will reduce the interface strength. But for a given interface porosity, the size and shape of interface cavities must also play a role. Indeed, large round-shape cavities might blunt interface cracks and rather tend to prevent their propagation, while small fine-dispersed elongated cavities should cause high interface embrittlement. Some authors have reported finite element analysis simulation of a crack growth along the interface between a porous ductile material and a rigid substrate [15, 16]. But very few experimental data for interfacial crack propagation between porous metal film and ceramic substrate are available in literature [17] – maybe because of the difficulty to characterize the interface porosity [18], as mentioned before – and even less under the form of quantitative adhesion measurement to be compared to our results. Thus the relation between the critical energy release rate of interface cracks and interface contact area is still not clear now.

Comparing the results at the same firing temperature we had reported previously [14], where the values of G_{ci} vary from 1.3 to 4.2 J/m^2 for Ag/BaTiO$_3$ interfaces, the results in the present work are much higher. The major differences between both studies are Ag powder particle size and chemical composition of Ag pastes. Both factors are likely to change the porosity of the film after firing and altogether the value of the thermodynamical interfacial work of adhesion itself. In figure 5, the values of the present work are plotted together with the previously reported adhesion strength of other silver paste (Ag 8985) to barium titanate [14], with the porosity measured using the same procedure. Both silver layer pastes contained some glass mixed with organic solvent and binder. From figure 5 we can observe that the chemical composition of the silver paste did not seem to influence much on interface adhesion. Contact area seems to play a more important role in the interface adhesion, since the same linear dependence of *Gci* on porosity appears to be valid for both Ag pastes.

Such a linear relationship is actually the simplest assumption which may be made to account for the dependence of interfacial crack propagation energy on interfacial porosity. Extrapolating the linear fit of figure 5 to a zero porosity value would lead to crack propagation energy about 10 J/m^2 for a full-contact Ag/BaTiO$_3$ interface, which is a rather reasonable value for metal/ceramic interface adhesion [19]. On the reverse, a linear extrapolation of our results to higher porosity rates would lead to null crack propagation energy far before 100% porosity, which is not physically acceptable. So, another extrapolation law should apply for porosity higher than about 15%, compatible with complete loss of adhesion only for 100% interfacial porosity. Another possible cause for this discrepancy is that our assumption taking the interfacial porosity equal to the bulk porosity in Ag is grossly wrong, which is difficult to verify, but looks unlikely as close as we could observe our metal/ceramic interfaces. More experiments with better accuracy and a wider range of Ag membrane porosity will be necessary to clear these points.

The main contribution to the incertitude for our results may be attributed to the difficulty in determining the accurate critical de-bonding points and subtracting the plastic strain contribution. Anyway, our crack propagation energy values are higher than the usual estimates of the thermodynamical work of adhesion of metal/ceramic interfaces alone, around 1 J/m^2. This difference can easily be explained by the various contributions of dissipative mechanisms which operate during interfacial crack propagation, so that strongly adherent systems (like metal/metal or metal/adhesive interfaces) may reach interfacial crack propagation energies as high as hundreds of J/m^2 [20].

五 結論

Specimens with porous Ag layer, fabricated at four sintering temperatures to produce different porosity, covering a dense BaTiO$_3$ substrate have been prepared with a special geometry appropriate for blister testing. The blister test technique allows numerical estimation of interfacial adhesion strength through determination of the interfacial crack propagation energy. A simple method is proposed to overcome experimental difficulties such as generalized plastic straining of the porous metal layer. With this procedure, the critical crack propagation energy of the interface between the Ag layer with various porosities on BaTiO$_3$ has been measured. The average value increases from 4.5 J/m^2 to 6.6 J/m^2 while the porosity decreases from 11% to 7%. The technique which has been set up here is expected to be useful for more detailed exploration of the effect of various parameters governing metal/ceramic interfacial adhesion. Experimental attempts are presently made to cross-check its results with other adhesion measurement techniques, like cross-sectional indentation, on the same metal/ceramic interfaces.

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七. 表格及圖形

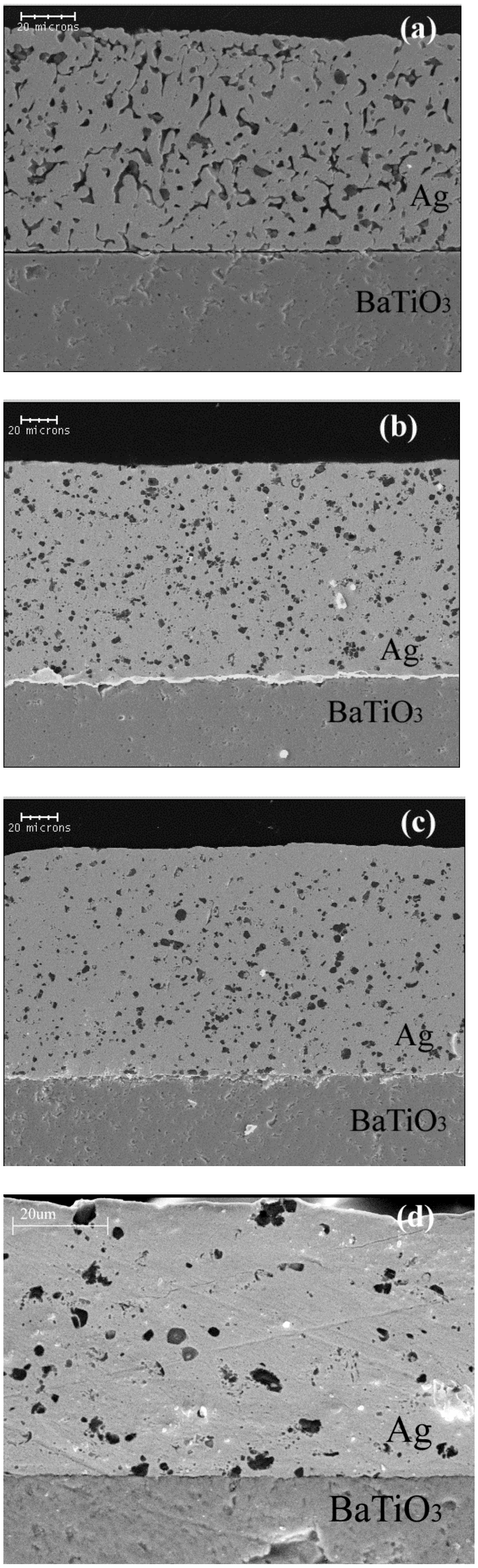

Fig. 1, Cross-section views of Ag/$BaTiO_3$ interfaces. Ag films were fired at (a) 600 ^{o}C (b) 700 ^{o}C (c) 800 ^{o}C (d) 850 ^{o}C.

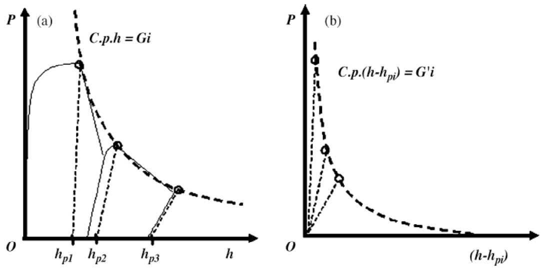

Fig. 2, Schematic of the principle of correction for the effect of the generalized plasticity of the silver membrane on the interfacial debonding energy.

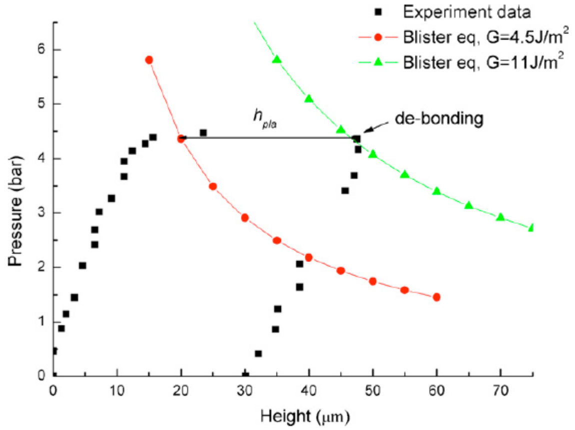

Fig. 3, Illustration of blister equation fit to experimental data for a specimen fired at 850°C. The uncorrected value G = 11 J/m^2 is adjusted on the experimental debonding point. The corrected value G' = 4.5 J/m^2 is adjusted on the corrected debonding height, according to the schematic of Fig. 2.

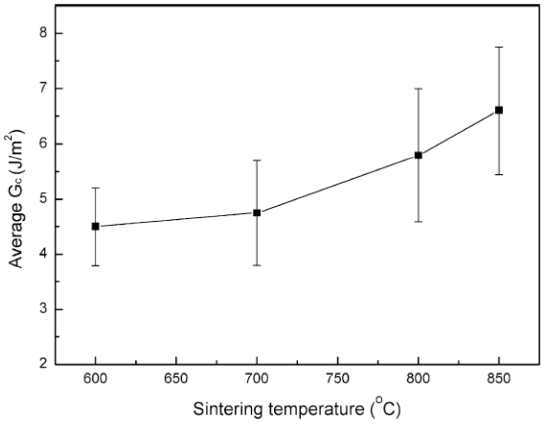

Fig. 4, Average fracture energy of interface versus sintering temperature of silver membrane.

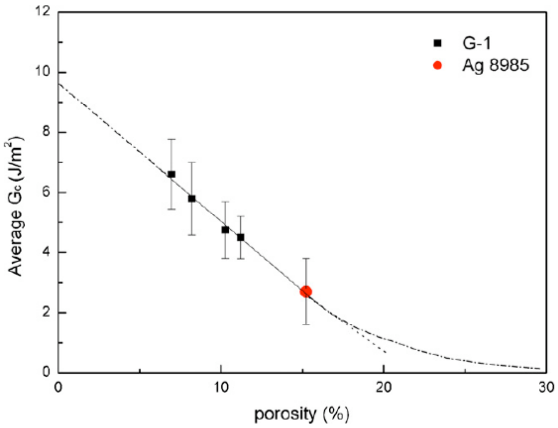

Fig. 5, Average fracture energy of interface versus average porosity of silver membrane. ■ present work; ● previous report [14].

第五屆中國先進陶瓷國際研討會

(5th China International Conference on High-Performance Ceramics, CICC-5)

I. 前言：

中國先進陶瓷國際研討會(China International Ceramic Conference, CICC)為北京清華大學所主辦之一系列的國際陶瓷會議, 本人曾參加第二屆,第三屆及第四屆, 此次參加的為第五屆. 會議從5月10日開始至5月13日在長沙舉行。

此次計有31個國家,計900餘個教授及學生參加，發表約1000篇論文，而上屆計有22個國家, 約800餘個教授及學生參加，發表約900篇論文， 而上上屆則只有約400餘個教授及學生參加，顯示此系列會議已成為世界上一個非常重要的國際陶瓷會議.

此次人數較上屆多出一百人, 但國外參與人數雖並未明顯增加, 但大陸內許多學校及研究單位的參加人數則大幅增加, 顯示北京清華大學在舉辦國際會議上的號召力及組織能力已大幅進步. 此次大陸除幾個重點大學及研究單位：清華大學、天津大學、哈爾濱工業大學、浙江大學、中國科學院皆有許多人參加. 一些較小的學校, 如技術學院等也有許多年輕教授參加.

會議在長沙市區的一個新的五星級旅館舉行, 該旅館雖在會議硬體上有完整資源, 但在國際會議經驗上仍待加強. 會議分Plenary Lecture, 口頭報告及海報展示三類, 此次plenary Lectures 在第一天上午發表, Plenary Lecture的講者, 皆具相當高的知名度, Plenary Lecture的講者也使出渾身解數, 內容精彩, 口頭報告分則在四個會議室與poster展示同步舉行, 此屆會議的口頭報告只約一百篇. 海報展示則高達九百篇, 每半天一批, 議程緊湊, 此次會議的議題如下:

Session A: Dielectric, Piezoelectric and Ferroelectric Materials
Session B: Ceramics for Fuel Cells and Batteries
Session C: Photonics Materials and Optical Properties
Session D: Advanced Electronic Materials
Session E: Engineering Ceramics and Composites
Session F: Bioceramics and Biocomposites
Session G: Ceramic Coatings
Session H: Glasses and Glass-Ceramics
Session I: Ceramics in Environmental Applications
Session J: Ceramics-Related Basic Sciences
Session Z: Others

值得注意的是上一屆的Session I 是 Nanostructured materials and nanotechnology, 而此屆取消奈米的Session, 而加入環境材料, 指出環境議題的重要性已被大陸接受, 而奈米材料已進入各領域的應用面上. 除以上十一個Session, 大會還同時組織三個系列的國際陶瓷會議, 此三個系列的國際陶瓷會議為

International symposium on ultra-high temperature ceramics
The third international workshop on layered and graded materials

International symposium on lead-free piezoelectric ceramics

這四個系列的國際陶瓷會議的召開, 顯示大陸對這系列的陶瓷領域的重視及企圖心.

筆者是此系列先進陶瓷國際會議的國際籌備委員會的委員之一, 筆者於12日擔任其中一場次的主持人,也於同日場次發表邀請演講(Invited Speech), 題目為多功能多相材料(Multi-functional multi-phase materials). 報告時會議室完全爆滿,許多人須站著聽完, 報告完後討論熱烈.

II. 心得

茲將本屆會議筆者認為最重要的四篇Plenary Lecture報告, 簡要說明於後:

1. 第一場由德國Max-Planck Institute的金屬所所長M. Ruehle 教授以”Structure and composition of grain boundaries in ceramics at the atomic level”為題發表演講, Ruehle教授在TEM方面是世界上最成名的教授之一, 他對許多陶瓷氧化物及氮化物皆有極深入的觀察, 他們且持續在Science上發表論文, Ruehle 教授大略敘訴他們實驗室的成果, 他指出grain boundary的觀察及解析是對預測材料性質最重要的關鍵. 演講中所引用的其中一個成果, 為SrTiO3的結構解析, 回國後經搜尋本校的電子期刊 Science, 附錄於後.

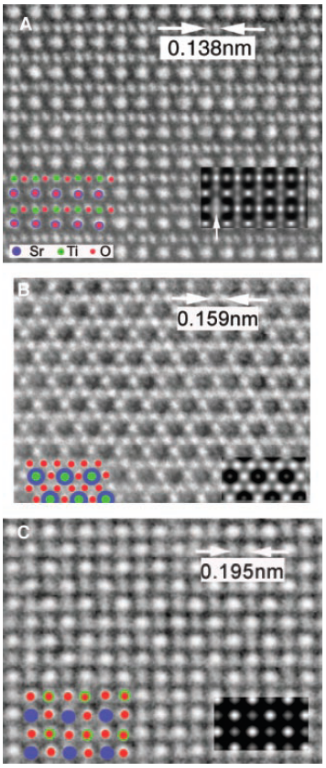

Fig. 1. (A) HRTEM image of bulk $SrTiO_3$ along the [110] direction. Sr-O, Ti, and O columns are clearly resolved. Inset, lower right corner: Simulated HRTEM image (underfocus, 80 nm; thickness, 2.76 nm). A structure model of $SrTiO_3$ viewed along the [110] direction is shown in the left corner. The

圖一

2. 第二場由美國加州大學Santa Barbara 分校的David R. Clark 教授以”Monitoring temperature and damage using luminescence”為題所發表的演講, Clark 教授在grain boundary方面的研究非常成名, 他指出thermal barrier coating已廣泛的使用在高溫零件上, 但高溫零件在運作時的真實溫度卻很難量測, 他們研究室深入探討高溫螢光的發光行為, 並開發出數種高溫螢光材料, 這些高溫螢光材料位於ZrO2-Y2O3-M2O3(M為稀土金屬元素)三元系統中, 他並指出研究室所開發出的高溫螢光材料目前已可偵測1000C的溫度.

3. 第三場由日本大阪大學的 Y. Miyamoto 教授發表, 這個演講是筆者以為本次會議中講的最精彩的演講, 他的題目是 New ceramics development by computer-aided design and processing, Miyamoto教授先介紹一個他們所開發出的Micro Stereo-Lithography System, 此系統並已由一家日本公司予以商業化並在市場上行銷, Miyamoto教授指出可以他們所開發出的這個系統, 進行self assembly的能力, 因系統搭配CAD軟體, 使他們可製作出非常小, 如奈米尺度的複雜結構, 其中一例, 筆者以數位相機照下, 請見圖二, 他再以此奈米尺度的複雜結構, 設計及開發出許多光晶體(Photonic crystal). 他們的研究將self assembly技術應用在photonic crystal, 充分發揮了self assembly的優勢, 並據此開發出可量產 photonic crystal 的方法, 他也證明以此法所得的 photonic crystal, 除具有優異性質, 並且具有再現性.

Schematic diagram of Menger sponge structures

圖二

4. 第四場由希臘 Ioannina大學的 Gournis教授發表, 他的題目是 Composites with carbon nano-structures: filling interlayers and nanotube, 他review目前在奈米複合材料上的一些發展, 演講內容乏善可陳.

III. 感想及建議

北京清華大學的材料學系以一各學系的力量, 每兩年辦一次國際會議顯示他們的企圖心及豐富的資源. 國內材料科系普遍不願意舉辦國際會議, 除資源有限, 並須自付盈虧, 壓力很大. 建議教育部或國科會須更積極以大量資金鼓勵學校舉辦相關活動. 大陸學生的認真及積極性一直令筆者憂心, 例如他們對從國外來的演講者的演講所投入的熱情及積極, 令人擔心國內研究生的好逸惡勞, 以後將如何面對來自對岸的競爭. 國內研究生也不喜歡聽演講, 這也將造成我們在舉辦國際會議時, 所必須承擔太少聽眾及冷場的風險.

此次會議除看到所有陶瓷領域的題目, 大陸方面將有人在研究, 但也可看到極具創意的研究, 顯示大陸科技政策及經費的提升, 對研究水準的提升收到不錯的效果.

IV. 攜回資料

會議論文摘要集

國際合作研究計畫心得報告書
暨
赴法國Joseph Fourier大學參加雙聯博士學位口試
回國報告

段維新
材料科學及工程學系
96年8月1日

一. 背景說明

在 NSC-CNRS 雙邊協議下, 報告人接受兩年國科會的支持, 執行與法國 Joseph Fourier大學Michel Depeux教授一起合作的研究計畫, 同時本校與位於法國 Grenoble的 Joseph Fourier大學亦訂有雙聯博士學位合約, 本系李炤佑同學繼取得本校及Joseph Fourier-Grenoble大學的雙碩士學位後, 參與本校與Joseph Fourier大學的雙聯博士學制, 並訂下合約, 依此合約, 李炤佑同學前兩年(July, 2003-August, 2005)在本校材料科學及工程學研究所進行研究, 後兩年(July, 2005-August, 2007)則在Joseph Fourier-Grenoble大學的機械系進行研究. 研究題目為: 金屬/陶瓷界面工程介電陶瓷與金屬電極之接著強度研究(Metal/ceramic interface engineering - adhesion strength measurement between dielectric ceramic and electrode metals).

執行此雙聯博士學位合約, 本校材料科學及工程學系的研究指導教授為本人, Joseph Fourier-Grenoble大學的研究指導教授則為機械系的Michel Depeux教授. 李炤佑同學先在本系準備試樣, 然後在Joseph Fourier-Grenoble大學的機械系進行界面分析及測試, 故本研究為一實質合作研究. 且一起發表研究成果:

1. Chao-Yu Lee, Michel Dupeux, Wei-Hsing Tuan, “Adhesion strength of Ag/BaTiO3 interface”, Scripta Mater., 54(3), 453-457 (2006) (SCI impact factor: 2.112).
2. Chao-Yu Lee, Michel Dupeux, Wei-Hsing Tuan, “Influence of firing temperature on interface adhesion between screen-printed Ag film and $BaTiO_3$ substrate”, Materials Science & Engineering A 467, 125-131 (2007) (SCI impact factor: 1.445).

經費方面, 本計畫的出國經費主要提供給李炤佑同學回國準備試樣及參加本校之預口試(proposal examination), 以達到本系對博士學位的要求.

李炤佑同學在克服法國的生活及語言的困難後, 陸續克服研究上的困難, 於今年一月申請本系的Proposal考試, 並且通過, 再於今年四月向Joseph Fourier-Grenoble大學提出博士學位考試申請, 在先經過兩位校外教授(一位教授為比利時魯文大學的Pordoen教授, 一位教授為成功大學的黃肇瑞教授)審查通過後, 訂下於今年7月16日於Grenoble的 Joseph Fourier大學進行口試,

二. 口試過程

口試委員計有七位:

Joseph Fourier-Grenoble大學材料系的Jacques FOULETIER 教授
Ecole Centrale de Lyon及CNRS的Jean-luc LOUBET博士
Joseph Fourier-Grenoble大學機械系的Muriel BRACCINI博士
比利時魯文大學的Podon教授(同時為論文審查委員),
成功大學材料系的黃肇瑞教授(同時為論文審查委員),
Joseph Fourier-Grenoble大學機械系的Michel Depeux教授(同時為研究指導教授),
台灣大學材料科學及工程學系的段維新教授(同時為研究指導教授).

口試在 Joseph Fourier-Grenoble大學機械系的一間大教室(Lecture theater)進行, 依 Joseph Fourier-Grenoble大學的規定, 口試的題目及其時間表已在兩週前在 Joseph Fourier-Grenoble大學及Grenoble地區所有研究單位公佈, 故有約十幾位旁聽的人, 在口試前已在口試地點等候, 所有口試委員先在口試地點旁邊的一個小會議室開會, 決定由誰擔任口試的主席, 因論文審查委員及指導教授不可擔任主席, 故最後決定由剩下的三位口試委員中最資深的Jacques FOULETIER教授擔任主席, 然後口試委員正式進入口試教室, 由主席宣佈李炤佑同學的名字及口試題目後, 正式開始口試, 李炤佑同學先進行30分鐘的簡報, 然後由口試委員進行詢答, 最先由論文審查委員提出問題, 而指導教授則在最後提出問題, 李炤佑同學回答問題的狀況不錯, 口試詢答約進行一個小時, 然後主席宣佈停止口試, 所有委員進入會議室, 討論評分, 在經過約15分鐘的討論後, 達成決議, 決議為通過博士學位考試, 並且不須對論文進行修正, 在填完本校及 Joseph Fourier-Grenoble大學的表格後, 所有口試委員回到口試的教室, 向等候多時的李炤佑同學及其朋友宣佈口試結果, 完成口試.

依法國習俗, 李炤佑博士在口試旁的教室準備香檳及許多食物, 邀請他的朋友及所有口試委員參加, 一起慶祝.

三. 致謝及感想

1. 本人感謝國科會, 台大及 Joseph Fourier-Grenoble大學的支持及資助, 使我們能一起培養出一位優秀且具國際觀的人才.
2. 法國對口試過程的嚴謹, 頗有參考價值.
3. 希望能透過這個過程, 了解與國外大學互動的優點, 及指導教授所需付出的時間及精力.

四. 附件

一 , 口試時的實況照片
二, 口試後所有口試委員與李博士的合照.

附件一，口試時的實況照片

附件二，口試後所有口試委員與李博士的合照