出國報告(出國類別:出席國際學術會議)

出席2012年製造科學與工程技術國際學術會議(ICMSE 2012)案

服務單位:國立暨南國際大學

姓名職稱:應光系 林錦正主任

派赴國家:大陸

出國期間:2012.03.27~2012.03.30

報告日期:2013.04.26

摘要

第三屆製造科學與工程技術國際學術會議（The 3rd International Conference on Manufacturing Science and Engineering, ICMSE-2012）於 2012 年 03 月 27 日～29 日在中國廈門國際展示中心召開。會議旨在提供一個國際論壇的演講和討論製造科學與工程技術最新的進展。ICMSE 也涵蓋其他主題的探討。依大會規劃，所有被接受的論文將轉載至 EI 國際期刊 Advanced Materials Research，筆者投稿之論文已在出席會議前被接受刊登在 Advanced Materials Research。筆者投稿論文題目為: Gamma-Ray Radiation-Induced Surface Hydrophobic Effects in Invar Alloy. (珈瑪輻射對 Invar 合金所引起的表面疏水效應)

目 錄

一、目的

(一)計畫目標:國立暨南國際大學科技學院應用材料及光電工程學系為了推動產學合作，促進學系與產業關係，並增進學生實務經驗，由應用材料及光電工程學系系主任林錦正教授承接行政院國家科學委員會中部科學工業園區管理局高科技設備前瞻技術發展計畫:「OLED顯示器製程關鍵零組件製作技術開發計劃」/學研子計畫,計畫名稱: 有機發光平面顯示器蒸鍍網罩INVAR基材黃光微影濕蝕刻製程參數與鋁金屬蒸鍍模擬之基礎研究，合作廠商為景智電子股份有限公司。本次出席2012年製造科學與工程技術國際學術會議(ICMSE 2012)即是為執行本計畫績效指標中的論文發表。

(二)主題: 珈瑪輻射對Invar合金所引起的表面疏水效應之探討

(三)緣起: 網罩（Shadow Mask ，又稱蔭罩、蔭蔽、孔罩、孔屏、網板等）是彩色陰極射線管(Color Cathode Ray Tube, CCRT)中使CRT由黑白變成彩色的關鍵元件。1970 年代後期，由於CCRT 之功能能契合監視器的顯示要求，伴隨著個人電腦的發展,已成為電腦監視器用的主要顯示

器。隨著電腦應用的功能及範圍擴大， CCRT 的產品及製造技術也在近十年中有長足的進步。隨著消費者對電子產品高顯示畫質的期待，OLED 顯示器的影像解析度必須朝向高畫素(SVGA, XGA, SXGA...)發展，OLED 重複蒸鍍製程中的金屬罩或 PLED 塗佈製程中的金屬網板，不可避免地亦須滿足高精密度的尺寸公差要求，這種高精密度的尺寸公差要求隨著顯示器大型化而更趨嚴峻。OLED 蒸鍍用金屬罩，其三個要求即為開口 Pattern 之尺寸公差、累積定位公差及與 Pattern 低熱膨脹特性。金屬罩(Metal Mask)在 OLED 顯示器中的主要功能，在於提供不同顏色發光薄膜之高解析度蒸鍍量產化製程所需之遮罩。如果蒸鍍用金屬罩尺寸及定位精度不佳，將造成 OLED 顯示器不同顏色（發光薄膜）錯亂現象，嚴重影響各有機發光層發光效率不均，而使得顯示面無法呈現高解析度的文字、畫面或圖案。雖然發光薄膜材料良窳決定了顯示板亮度及壽命等，但金屬罩卻決定了顯示板其畫質的均勻性。OLED 上游元件材料包括 ITO 陽極玻璃基板、各層有機發光材料（電洞注入層、電洞傳輸層、有機發光層、電子傳輸層、電子注

入層、陰極）與鍍膜所需之金屬罩。金屬罩數目需求隨著面板的全彩化而增加,如使用小分子有機電激發光材料之 OLED 顯示器，必須利用熱蒸鍍(ThermalEvaporation) 的方式來蒸鍍多層有機膜材。在量產化全彩面板的製造過程中,為了避免不同材料間的相互污染,必須使用多腔體的真空設備及應用不同圖形之金屬罩，來進行不同顏色發光薄膜之蒸鍍製程。隨著顯示器「全彩化」、「高解析度化」及「大面積化」的產品發展趨勢下,金屬罩圖形尺寸精度及定位精度的要求將日趨嚴格。至於蒸鍍用高定位精度金屬罩的材質選用，依各廠家設計需求而有不同，常見採用的金屬板材有不銹鋼、鎳基或鐵鎳合金。其中不銹鋼具有最佳的剛性與強度；若需考慮高溫蒸鍍熱膨脹係數變化時，低熱膨脹係數之鐵鎳合金(INVAR-Fe-36Ni)則是最佳選擇。國內提供之量產鋼板／金屬罩，其精度（即尺寸公差）穩定性並不足以適用於高解析度蒸鍍。隨著國內對蒸鍍線路、孔徑及 Pitch 愈來愈精密的需求，需使用精密鋼板的國內廠商皆向日本購買。基於價格與往返溝通之耗時，國內開發高精密金屬罩蝕刻與電鑄技術，甚

至開發可結合電鑄、蝕刻、雷射加工之精密金屬複合加工技術與產品，已是刻不容緩。

(四)欲達成事項:將近半年來對 INVAR 的研究成果以論文方式發表，同時觀摩各領域學者發表之成果做為後續研究之參考。

二、過程

第三屆製造科學與工程技術國際學術會議（The 3rd International Conference on Manufacturing Science and Engineering, ICMSE-2012ICMSE-2012）於 2012 年 03 月 27 日～29 日在中國廈門國際展示中心召開。會議旨在提供一個國際論壇的演講和討論製造科學與工程技術最新的進展。ICMSE 也涵蓋其他主題的探討。依大會規劃所有被接受的論文將轉載至 EI 國際期刊 Advanced Materials Research，筆者投稿論文已在出席會議前被接受刊登在 Advanced Materials Research。

會議邀請世界各國製造科學與工程技術專家作大會報告並提供各個領域論文發表者口頭報告，這次會議提供世界國相關領域的研究人員之間交流和了解國際最新進展的一次很好的機會。

本次會議參加人數約 300 人，筆者主要是參加 New Materials and Advanced Materials 主題的討論，會議中以論文方式發表。大會這一次並沒有對於壁報論文有專區發表與討論，主要還是以論文刊登方式為主。筆者以通訊作者發表題目為” **Gamma-Ray Radiation-Induced Surface Hydrophobic Effects in Invar Alloy** ”之論文。

大會議程安排 3 月 27 日為註冊報到日，筆者於 3 月 27 日 18:45 搭乘復興航空 GE3666 班機由高雄小港機場起飛直抵廈門機場，當日抵達廈門機場。經過海關及提領行李等手續，再搭計程車抵達下榻旅館華林大酒店，此處即為大會報到處，距離大會地址約 5 分鐘腳程，選擇此處居住非常方便。

03 月 28 日是大會開幕式，兩日大會共安排 9 場 keynote speech, 四大會議主題，陣容相當龐大。9 位 keynote speaker 分別為: *Prof. Yu Zhou, Prof. Qunpeng Zhong, Prof. Quing Liu, Prof. Shandong Tu, Prof. Jun Sun, Prof. Yuefeng Gu, Prof. Luchang Qin, Prof. Nabil Gindy, Prof. Han huang,*

四大會議主題分別為:

Automation Equipment and Systems
Advanced Manufacturing Technology
Advanced Materials and Materials Processing
Manufacturing Processing and System

其餘時間大會皆安排口頭報告。筆者兩日於會場選了不少有興趣的主題聽講，獲益良多。

筆者所發表論文主要是發現有關於 INVAR 表面經珈瑪輻射表面會發

生疏水效應。本論文是筆者與國立高雄大學楊證富教授及工研院化材所主任陳興華博士共同研究成果，另外三位是筆者暨大的研究生。全文以英文發表，其英文摘要如下:

Abstract. In this paper, we report a new phenomenon observed in the gamma-ray radiation-induced hydrophobic effects on an Invar surface: When the Invar alloy is subjected to different doses of gamma-ray irradiation, the contact angle increases with the radiation dose. Invar samples with exposed to a higher dose appear more hydrophobic, but this tendency disappears following post-irradiation etching. The contact angles of the irradiated and etched Invar samples can be restored back to a stable value with small deviation after 30 min of annealing at 150℃. X-ray diffraction (XRD) analysis found no crystalline structural changes. High resolution field emission scanning microscope (FE-SEM) analyses showed that irradiation might induce crack-like surfaces which could be removed at higher radiation dose in the following acid etchings. It is believed that the chemical bonds of Invar oxide on the surface were broken by the gamma-ray irradiation, thus raising the likelihood of binding with free ions in the air and resulting in the exclusion of the hydrophilic OH bonds, leaving a hydrophobic post-irradiation Invar surface.

中文扼要說明如下:

在這篇論文中我們發表了一個新現象: INVAR 表面經珈瑪輻射表面會誘發疏水效應。當 INVAR 薄片經過不同劑量的高能珈瑪輻射照射，其表面接觸角隨劑量增加而增加。也就是說 INVAR 接受較高劑量的珈瑪輻射表面越疏水。但是當我們將這些照過珈瑪輻射的金屬片利用氯化鐵蝕刻，所有的疏水特性趨勢皆消失。代表疏水特性來自 INVAR 表面的變化。另外這些疏水特性的變化可以經由 150℃, 30 分鐘的處理到達一個穩定狀態。利用 X 光繞射量測發現不同劑量的高能珈瑪輻射

照射之 INVAR 晶相皆未改變，但利用高解析度電子顯微鏡觀察則發現 INVAR 表面有許多龜裂產生，而蝕刻後所有龜裂狀皆消失，所以我們推測我們所觀察到的疏水效應來自 INVAR 表面氧化層的輻射效應。當表面氧化層經珈瑪輻射照射，斷裂的鍵結會與空氣中的自由離子結合排斥氫氧基的鍵結。

3 月 29 日晚間大會結束，隔日筆者於 3 月 30 日搭乘廈門航空 GE3665 班機由廈門機場起飛直抵高雄小港機場。

三、心得與建議事項

筆者這幾年每年平均出國參加國際研討會約 1~2 次，漸次發現許多大型研討會皆由大陸各大學所主辦，而且規模都相當大。以下是幾張會議地點與會場內及大會開會時的實景:

大會舉辦地點: 中國廈門國際展示中心　　　參與學者到會議現場

會議現場實況_1　　　會議現場實況_2

會議現場實況_3

筆者將這幾張實景相片放置於心得處是要在本報告中展現大陸不斷崛起的趨勢，包含各大型的國際研討會，相較於國內過去曾轟轟烈烈每兩年舉辦一次的國際電機電子研討會(IEDMS)也逐年式微，最終都得併國科會年度成果報告壯聲勢，但在大陸相同性質的國際電機電子研討會(ICSICT)卻每年吸引上千人與會，消長之間值得學術界警惕。

具體建議相關單位應對國內舉辦大型國際研討會建立鼓勵制度，並應大幅提高補助經費，因為研討會能否辦得有聲有色關鍵在於能否請到世界第一流學者與會，而這些 keynote speaker 常常出席費與相關交通食宿相當可觀，遠超過政府補助。國內各大學更應體認大陸的崛起，強化自身的研發能力迎接挑戰。

四、附錄

附錄 1 論文接受函

2012 International Conference on
Manufacturing Science and Engineering

March 24-25 Xiamen, China

Notification of Paper Acceptance

Dear Authors,

The Scientific Committee has completed its review of your paper submitted for the 3rd International Conference on Manufacturing Science and Engineering (ICMSE 2012). The final decision is made base on the peer-review reports, the scientific merits and the relevance.

We are pleased to inform you that your paper as follow has now been accepted by the Scientific Committee of ICMSE 2012 and will be published in international journal "Advanced Materials Research", and will be indexed by EI COMPENDEX, Thomson ISTP and Elsevier SCOPUS.

Manuscript Number	**V9612**
Authors	**Cheng-Fu Yang, Wei-Wen Wang, Hsin-Hwa Chen, Wei-Tan Sun, Chi-Lin Shiau, Jing-Jenn Lin**
Title	**Gamma-Ray Radiation-Induced Surface Hydrophobic Effects in Invar Alloy**

Notes:

1. Please revise your manuscript according to the detailed comments and suggestions from the referees. And make sure that your paper is in strict accordance with the format of the journal.

2. Please read the attached registration form carefully and make sure that you pay the registration fees in time.

Any questions, please do not hesitate to contact us.

The Committee of ICMSE 2012
Hong Kong Industrial Technology Centre
ICMSE.NET

2012-01-02

Referee's Report Form

Paper Title:

Gamma-Ray Radiation-Induced Surface Hydrophobic Effects in Invar Alloy

Author(s):

Cheng-Fu Yang, Wei-Wen Wang, Hsin-Hwa Chen, Wei-Tan Sun, Chi-Lin Shiau, Jing-Jenn Lin

A. Style and Organization:

1. Is the paper clearly presented and well organized? YES
2. Is the English satisfactory? YES
3. Is the title appropriate? YES
4. Are the figures, tables, and their captions clear? YES
5. Are the references to related work adequate? YES

B. Scientific Quality (Please check appropriate box):

- ❑ √ Contains significant contributions to the advancement of the subject.
- ❑ √ Sound, original, and of interest.
- ❑ Does not add to knowledge of the subject.
- ❑ Contains fundamental errors.

C. Recommendation (Please check appropriate box):

- ❑ √ Publish as it is.
- ❑ Publish with minor revision noted in evaluation statement.
- ❑ Publish with major revision.
- ❑ Reject.

D. Comments: Please summarize the reasons for your recommendation in a statement below or on the reverse side of this sheet. If the authors are Chinese, you can write comments in Chinese.

Referee's Report Form

Paper Title:

Gamma-Ray Radiation-Induced Surface Hydrophobic Effects in Invar Alloy

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- ❑ Publish with major revision.
- ❑ Reject.

D. Comments: Please summarize the reasons for your recommendation in a statement below or on the reverse side of this sheet. If the authors are Chinese, you can write comments in Chinese.

附錄 2 大會議程封面


2012 年制造科学与工程
国际学术会议
The 3rd International Conference on
Manufacturing Science and Engineering
ICMSE 2012
Xiamen, China
March 27-29, 2012
Co-organized by:
Fujian University of Technology, China
Xiamen University, China
Fuzhou University, China
Huaqiao University, China
University of Wollongong, Australia
Fujian Mechanical Engineering Society, China
Hong Kong Industrial Technology Research Centre

附錄 3 刊登論文

Advanced Materials Research Vols. 482-484 (2012) pp 1585-1591
Online available since 2012/Feb/27 at www.scientific.net

doi:10.4028/www.scientific.net/AMR.482-484.1585

Gamma-Ray Radiation-Induced Surface Hydrophobic Effects in Invar Alloy

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Keywords: gamma-ray, radiation, Invar, hydrophobic.

Abstract. In this paper, we report a new phenomenon observed in the gamma-ray radiation-induced hydrophobic effects on an Invar surface: When the Invar alloy is subjected to different doses of gamma-ray irradiation, the contact angle increases with the radiation dose. Invar samples with exposed to a higher dose appear more hydrophobic, but this tendency disappears following post-irradiation etching. The contact angles of the irradiated and etched Invar samples can be restored back to a stable value with small deviation after 30 min of annealing at 150℃. X-ray diffraction (XRD) analysis found no crystalline structural changes. High resolution field emission scanning microscope (FE-SEM) analyses showed that irradiation might induce crack-like surfaces which could be removed at higher radiation dose in the following acid etchings. It is believed that the chemical bonds of Invar oxide on the surface were broken by the gamma-ray irradiation, thus raising the likelihood of binding with free ions in the air and resulting in the exclusion of the hydrophilic OH bonds, leaving a hydrophobic post-irradiation Invar surface.

Introduction

Since the discovery of Invar (Fe-Ni alloy) in 1897, researchers have developed various types of Invar alloys and applications [1-3]. Due to their near-zero thermal expansion coefficient over a wide temperature range, Invars are widely used in applications requiring high dimensional stability despite variations in temperature, such as clocks and watches, internal combustion engine pistons, bimetal strips, glass-to-metal seals, thermostatic strips, microwave guides, laser housings, precision optical instrumentation, liquid natural gas containers in tankers, printed circuit board cores and microelectronic packaging [2,4]. Invar is also widely used as shadow mask for large-screen televisions or in high-resolution color CRT monitors. Bombarding the CRT with electrons produces many hot arches and the low-coefficient Invar shadow mask can prevent the deflection of the electron beam, thus providing high picture clarity. Recent developments in organic light emitting diode (OLED) displays have increased the demand for Invar alloy shadow masks. In OLED displays using small molecule organic light-emitting materials, the thermal evaporation approach is commonly used to deposit the multi-layer organic membrane. For large, full-color HD displays, Invar shadow masks provide the best solution to avoid thermal-induced position deviations in the red, green and blue sub-pixel evaporation processes[5-7].

Invar alloy is also used in satellite instrumentation because its dimensional stability can easily overcome significant temperature variations satellite experience when moving from shadow to sunlight [8]. Materials used in spacecraft fabrication have to deal with high-speed energetic charged particles such as protons, alpha particles, carbon and ion nuclei which may cause lattice displacement damage, single event effects, noise in sensors and spacecraft electrostatic charging. They also must deal with ionizing radiation including gamma-rays, X-rays and electron flux, which can also damage metal alloys or dielectric materials in spacecraft, especially electronic devices with oxide layers [9]. Unlike the pure metal, alloys may occur structural and phase transformation by ionizing radiation [10]. As Invar alloys are important materials used in spacecraft instrumentations, investigating ionizing radiation effects on Invar alloys is one of important issues. This study describes a newly-discovered hydrophobic phenomenon on the surface of Invar alloy following gamma-ray irradiation.

Experimental

A cold-rolled NAS 36 Invar sheet with a thickness of 40 μm is commercially available from TOYO SEIHAKU Corp. Table 1 lists the chemical composition of Invar containing wt. 35.0~37.0% of nickel, with small quantities of carbon, silicon, manganese, phosphorous, sulfur, chromium, and a balance of iron. All the 1cm x 1cm Invar samples for gamma-ray tests were cut from the same roll and divided into several batches. Five sample batches were separately subjected to Co^{60} gamma-ray irradiation with doses of 1, 3, 5, 7 and 10 Mrads, while one non-irradiated batch served as a control. Following irradiation, all samples were left in ambient conditions to stabilize for seven days. The contact angle goniometer used is Sindatek Model 100SB in determining surface hydrophilic characteristics. XRD was used to identify crystalline structural changes, and the surface morphologies were examined by high resolution FE-SEM.

Table 1. The constituents of NAS 36 Invar alloy.

(%)

	C	Si	Mn	P	S	Ni	Cr	Fe
NAS 36	≦0.05	≦0.30	≦0.80	≦0.010	≦0.010	35.0~37.0	≦0.25	Bal

Results and discussions

Figure 1 shows the contact angle as a function of the doses of gamma-ray radiation. The control sample without irradiation (0 Mrad) is also depicted for comparison. The contact angles were measured with the same volume of water droplets. As shown in Fig.1, the contact angle increases with irradiation dose up to 7 Mrad, after which it levels out. As the gamma-ray irradiation dose increases, the Invar surface becomes more hydrophobic. Figure 2 presents the XRD patterns with increasing radiation doses, which show no changes to the composition or structure of the Invar sheet. Figures 3(a)-3(d) shows the surface morphologies of high resolution FESEM with different radiation doses (magnification factor 100,000 X). As shown in Fig. 3(a), despite the rolled imprints, the image show uniformly smooth surface. However, crack-like surfaces are observed in Figs. 3(b)-3(d). Unlike very high energy cosmic-charged particles in the GeV range (which most likely produce structural changes in the metal alloy), the moderate energy in the MeV range of gamma-ray ionizing radiation most seriously affects dielectric materials. The crack-like compositions are believed to be the native Invar oxide on the surface. The gamma-ray irradiation-induced hydrophobic Invar surface is possibly attributed to damage to the surface Invar oxide.

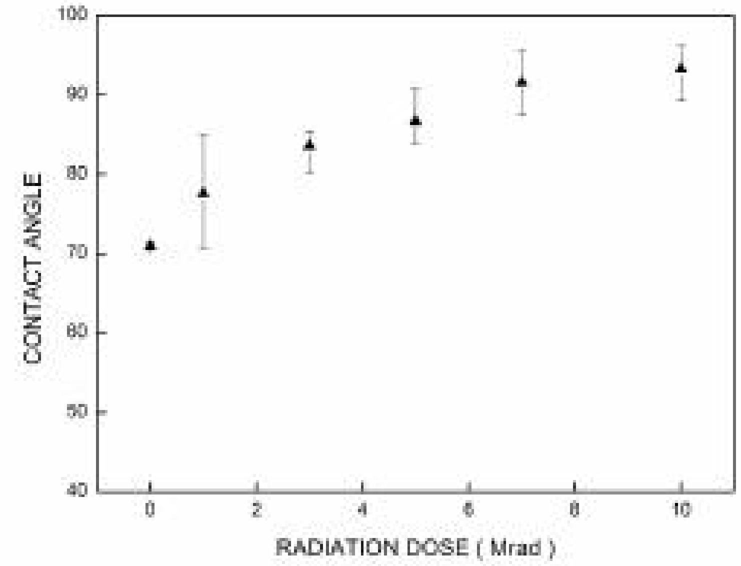

Fig. 1. Contact angle as a function of the doses of gamma-ray radiation.

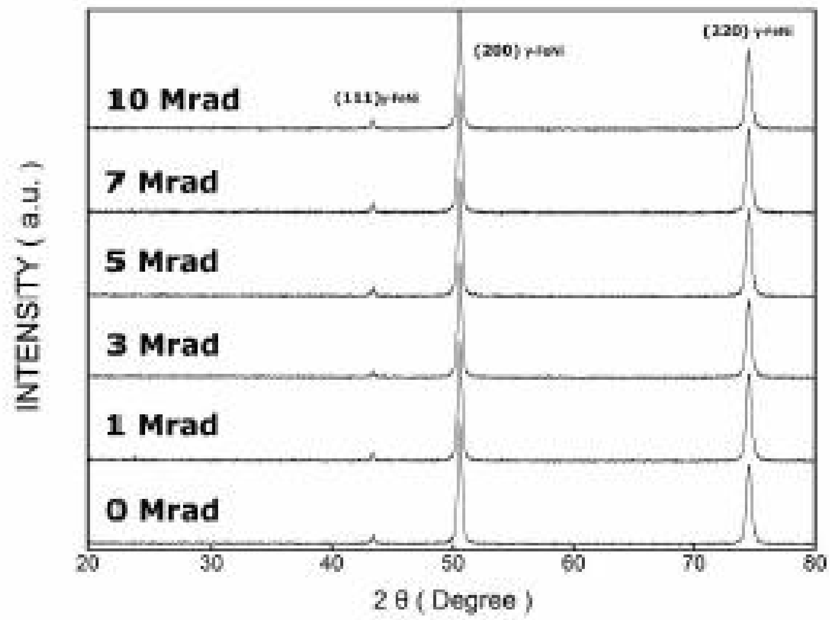

Fig. 2. XRD patterns of Invar with increasing radiation doses.

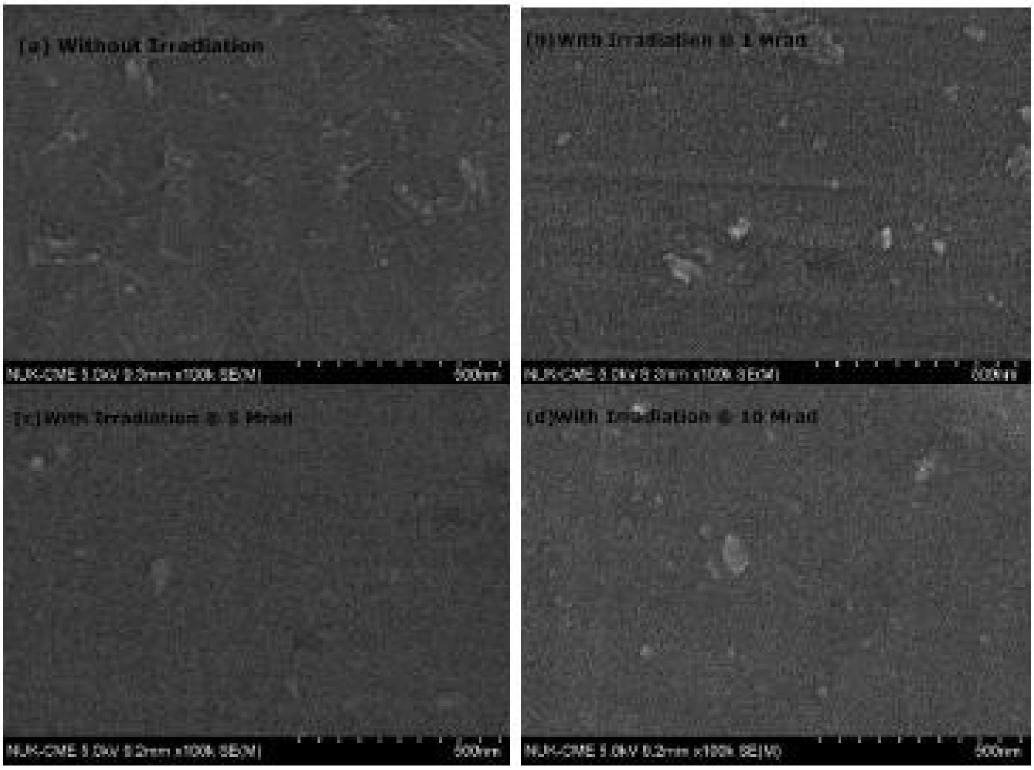

Fig. 3 Surface morphologies of high resolution FE-SEM with radiation doses of (a) 0 Mrad, (b) 1 Mrad, (c) 5 Mrad, (d) 10 Mrad.

To further study the cause of the hydrophobic effects, Invar alloys were subjected to different radiation doses prior to etching with a substance comprised of 60% HNO_3 and 40% CH_3COOH for 30 s, after which the contact angles and the surface morphologies were examined. Figure 4 shows curve of contact angle and irradiation dose for the irradiated etched and the control etched samples, and Fig. 5 shows the correspondence FE-SEM images of the etched surfaces. As shown in Fig. 4, the increase in contact angle with radiation dose (observed in Fig. 1) has disappeared. The contact angle is determined by the surface conditions of the Invar alloy. Given high-resolution FE-SEM in Figs. 5(a)-5(d), the etched surface remained reduced crack-like distributions at 1 Mrad (Fig. 5(a)), and became very smooth at higher radiation doses (Figs. 5(b)-5(d)). Gamma-ray radiation is believed to break the surface bonds of the Invar oxide, resulting in crack-like surface, and tends to more easily to be removed at higher dose of irradiation by the following acid etching. It is believed that the contact angle is influenced by the chemical bonds of the Invar surface, the resulting dangling bonds in the Invar oxide induced by irradiation increase the possibility of binding with free ions in the air, thus excluding the hydrophilic OH bonds, making the post-irradiated Invar surface more hydrophobic.

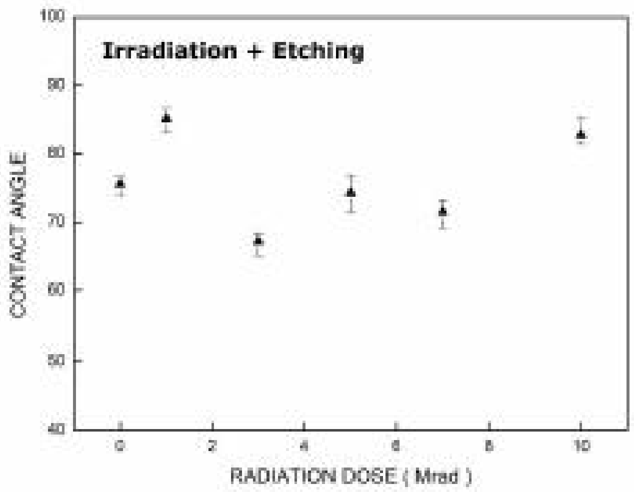

Fig. 4. Contact angle as a function of the doses of gamma-ray radiation for the irradiated etched and the control etched samples.

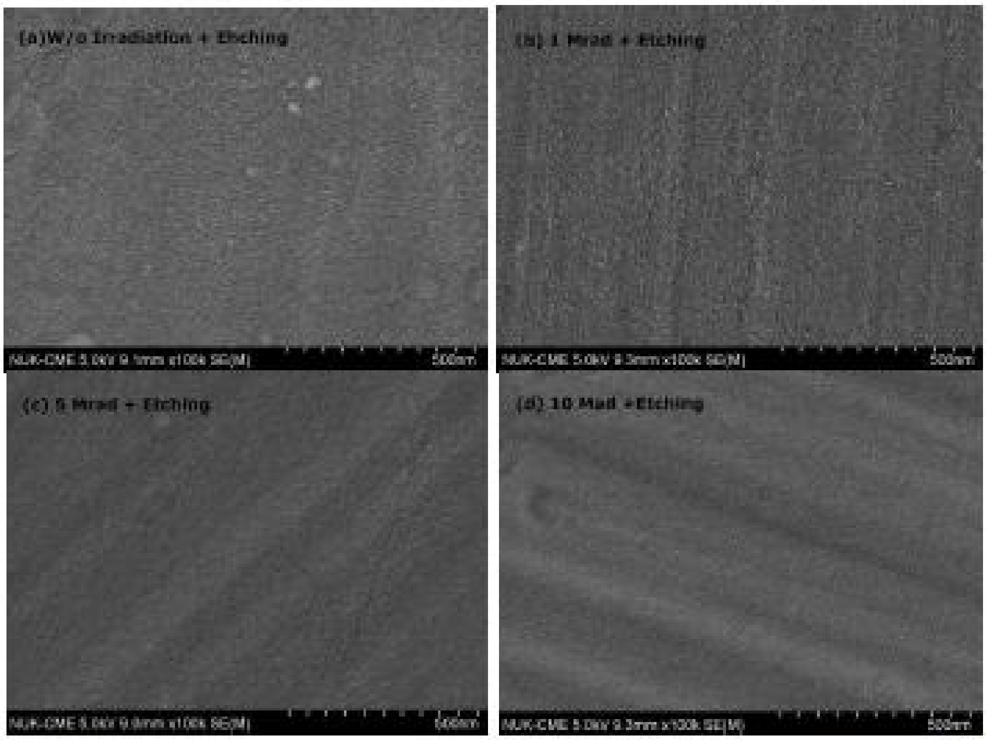

Fig. 5. FE-SEM images of the irradiated etched surface with radiation doses of (a) 0 Mrad, (b) 1 Mrad, (c) 5 Mrad, (d) 10 Mrad.

Finally, the post-irradiated samples shown in Figs. 4 and 5 were divided into three batches. Each batch was subjected to post-etching annealing in an oven with an atmospheric environment at 50, 100 and 150℃ for 30 min. After all samples had cooled to room temperature, the contact angle of each sample was determined. Figure 6 shows the correspondence curve of contact angle and radiation dose for different annealing temperatures. Regardless of radiation dose, the contact angle tends to a stable value with little deviation following a 150℃, 30 min anneal. It is believed that a higher-temperature anneal of the post-etching Invar will result in a more fully-oxidized and stable surface, which further supports the possibility that damage to the Invar oxide is the main cause of the gamma-ray irradiation-induced hydrophobic surface.

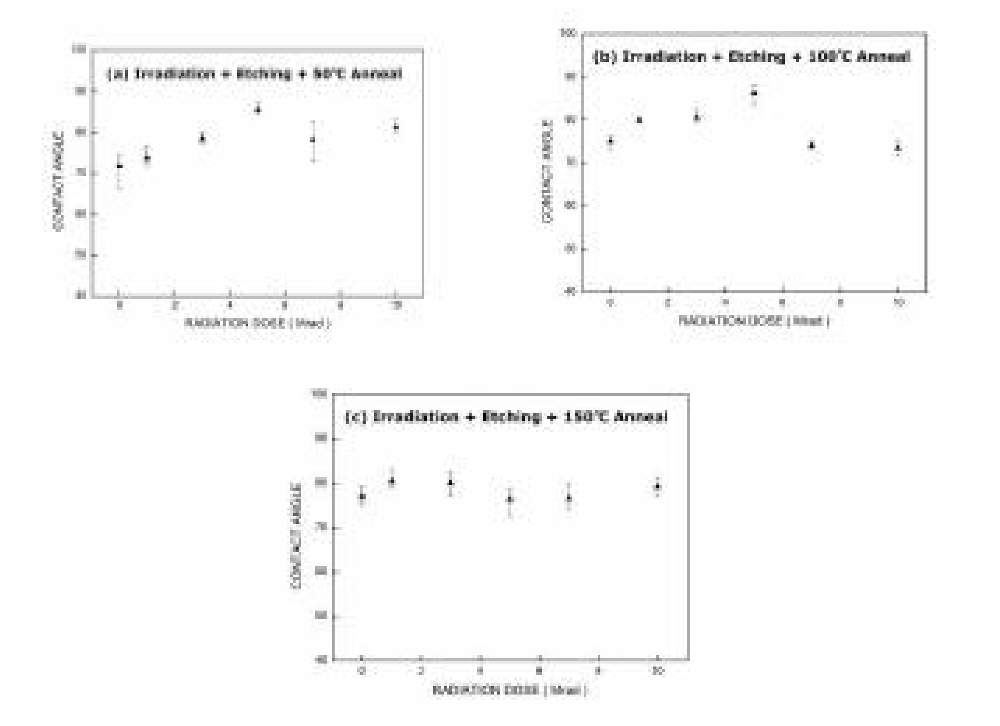

Fig. 6. Contact angle as a function of the doses of gamma-ray radiation for the irradiated etched and the control etched samples with 30 min anneals of (a) 50℃, (b) 100℃, (c) 150℃.

Conclusions

The contact angle of the Invar sheet increased with gamma-ray radiation dose. Post-irradiation etching and annealing experiments indicate that the increased contact angle was due to changes in surface conditions. It is believed that the reactive chemical sites in the Invar oxide induced by gamma-ray irradiation were responsible for the hydrophobic phenomenon. The irradiation-induced chemical reaction on the Invar surface must be confirmed by further investigation.

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