

出國報告(出國類別:參訪與出席國際研討會)

複合材料合成與國際材料磨損研討會心得

服務機關：國立虎尾科技大學

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一、摘要

材料科技的發展對於機械元件之壽命延伸與表面特性改善微精密機械元件性能提升之主要方法，而本研討會為國際知名期刊 Wear 所舉辦之大型國際研討會，與會人士多為國際各界材料與磨潤研究學有專精之學者，藉由與各界學者切磋材料改質技術、磨潤設計與磨耗機制對於滾珠螺桿磨潤研究與磨耗抑制研究，具有很大的幫助。本次行程中亦包含前往本校李利講座教授實驗室參訪，了解工業用高分子複合材料之製作方式，以及其應用特性，並與李利教授進行座談，了解其實驗室目前對於此複合材料之成果、應用狀況、產業應用前景以及可以合作研究方向。

二、目的

本次出國參訪之主要目的在於與國際學者交流材料磨潤科技之研究心得與得知最新材料研究之發展趨勢，增長材料磨潤研究之技巧，以及拓展國內材料科技之發展與應用。

三、過程

行程上於2011年3月31日從桃園機場搭機出發，並於4月1日抵達俄亥俄州哥倫布市，之後前往俄亥俄州立大學 李利教授實驗室參訪。此參訪行程可學習一種特殊噴塗技術應用於材料表面改質，對於提升螺桿材料壽命具有其應用性。4月4日至6日共三天前往費城參與「第18屆國際材料磨損研討會」並發表論文。之後於8日返抵國門。

本次參訪俄亥俄州立大學李利教授實驗室行程，共三天。李利教授為美國國家科學院院士，並為本校之講座教授，其研究領域為高分子材料合成與相關應用，本次有幸獲邀參訪其實驗室，對於其研究課題之一高機械性能高分子複合材料合成技術，進行了解與學習，並與李利院士進行學術交流座談。參訪照片請見圖 1-3，期間除了實驗室參訪外，並實際參與學生複合材料實驗，攜回樣本試片兩片，進行其奈米機械性質與材料成分分析，其成果將作為後續學術研究之基礎。此外與李教授之座談會更談到未來期開發之複合材料可以藉由國內之精密機械加工技術進行量產型機台試作，提昇產品競爭性。此外「第 18 屆國際材料磨損研討會」為全球知名之磨潤學會議，與會學者包括歐洲、亞洲與美洲各國之知名學者與研究生共約 500 人，並收錄了超過四百篇以上的論文發表，為國際上具有相當規模之研討會。圖 4 為建國科大高文顯教授，與我一同與會發表論文，圖 5 為本人參與論文發表之照片。圖 6-圖 8 為本次參加會議所攜回之資料，內容包括材料表面改質與磨潤壽命提昇驗證，以及人工關節磨潤研究等相當豐富之參考資料。本次發表之論文，首頁為圖 9 所示，為兩種普遍應用於產業界之工具鋼 SKS3 與 SKH51，經由氮電漿注入後之表面改質層磨潤特性研究，此研究之特點在於利用電漿氮化改質可以短時間內達到傳統氣體氮化改質層，並更加強其表面磨潤特性，達到極佳之表面硬度與韌性。而此兩種鋼材由於內部 Cr 元素含量之高低不同，得到深淺不同特性之氮化改質層，SKS3 之 Cr 含量低，因此改質層較 SKH51 淺，因此表面硬度也較低，因此磨耗量較大，氮是摩擦係數較低。文中並探討氮化白層組織之組成與對於磨潤之影響。



圖 1 俄亥俄州立大學行政大樓



圖 2 李利院士實驗室與博士生討論



圖 3 與李利院士(中間)合影



圖 4 參加國際材料磨損研討會

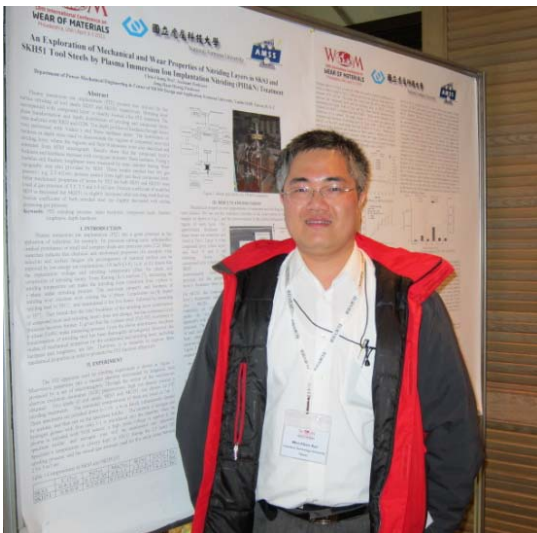


圖 5 論文張貼與發表

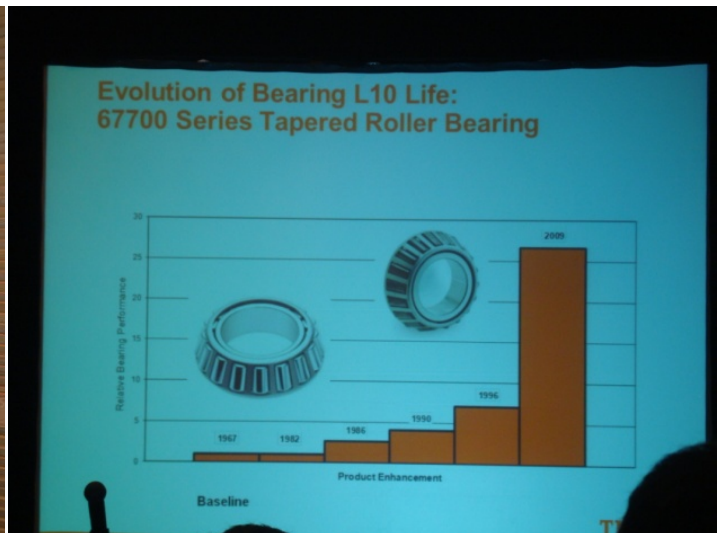


圖 6 參考資料-軸承鍍膜延壽趨勢

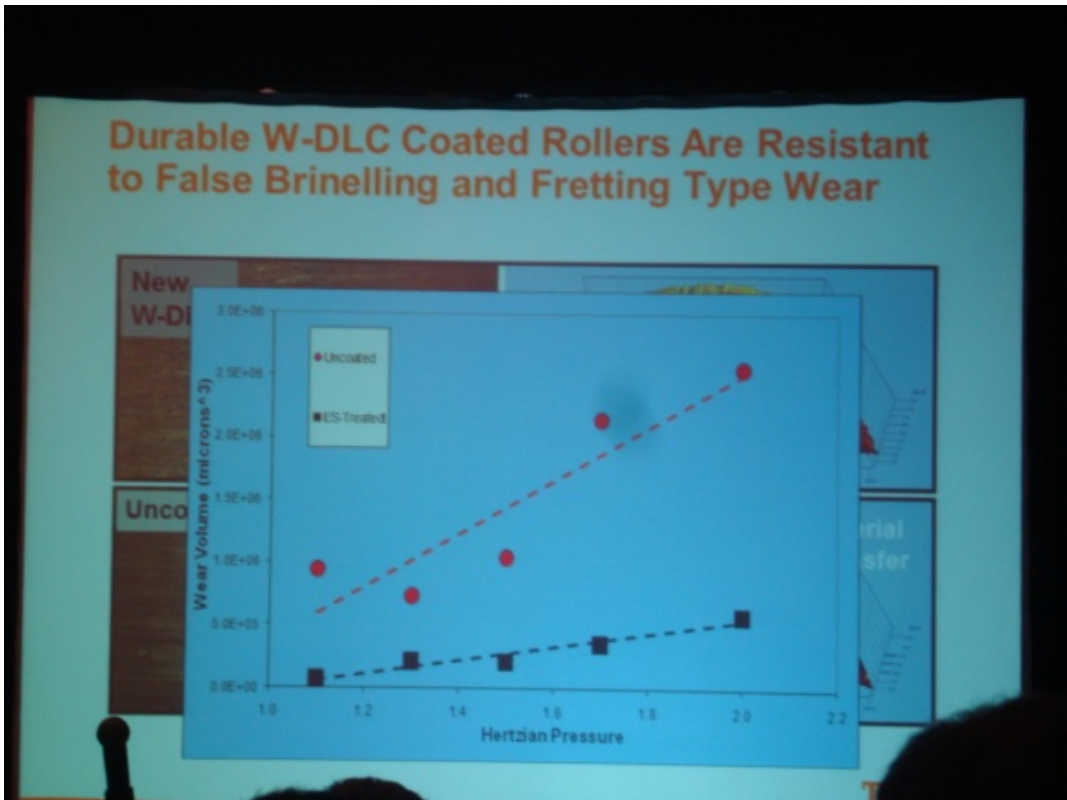


圖 7 參考資料-W-DLC 鍍層磨耗實驗分析

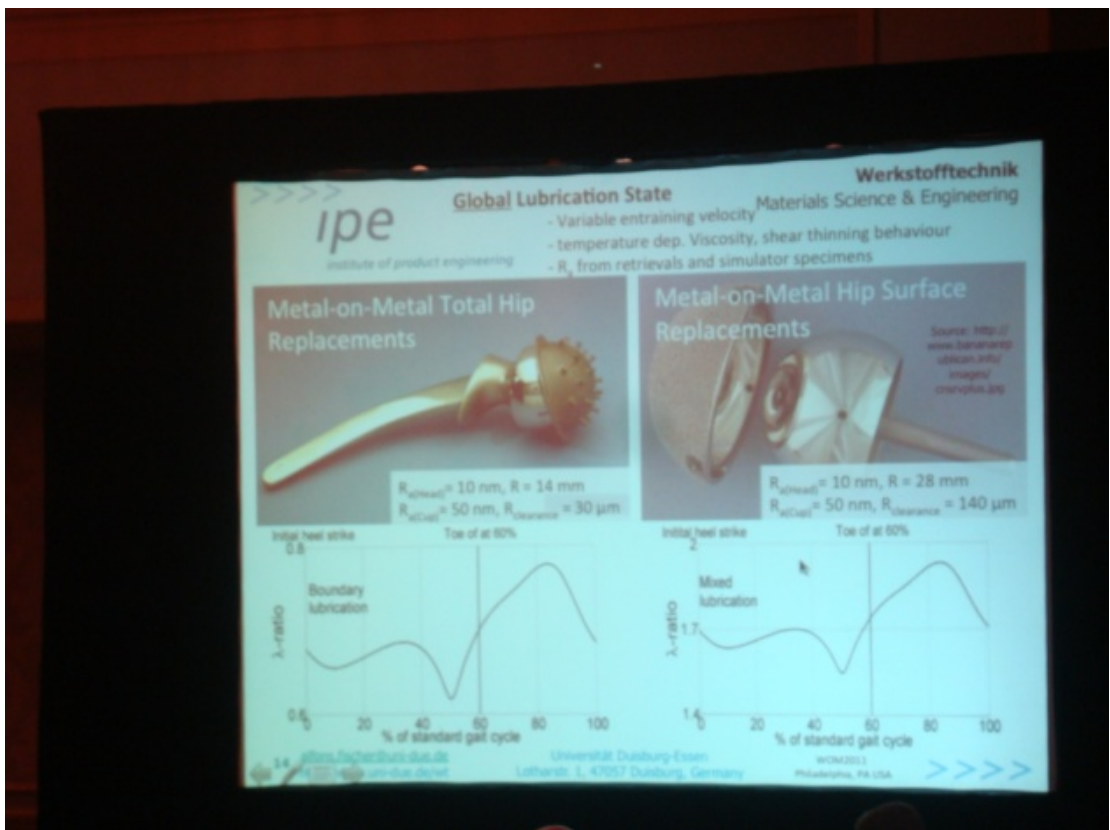


圖 8 參考資料-人工關節磨耗研究

An Exploration of Mechanical and Wear Properties of Nitriding Layers in SKS3 and SKH51 Tool Steels by Plasma Immersion Ion Implantation Nitriding (PIII&N) Treatment

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Abstract

Plasma immersion ion implantation (PIII) process was utilized for the surface nitriding of tool steels SKS3 and SKH51 respectively. Nitriding layer accompanied with compound layer is usually formed after PIII treatment. The phase transformation and depth distribution of nitriding and compound layers were analyzed with XRD and GDS. The depth profiles of hardness through layers were performed with Vicker's and Nano hardness tester. The distribution of hardness in depth were used to discriminate the regions of compound layer and nitriding layer, where the regions and their thicknesses were also identified and estimated from SEM micrograph. Results show that the compound layer's thickness and hardness increase with rising gas pressure. Nano hardness, Young's modulus and fracture toughness were measured by nano indenter testing. Wear topography was also provided by SEM. These results implied that low gas pressure (e.g. 2.5 mTorr) process cannot form tight and thick compound layer. Better mechanical properties of layers by PIII for both SKS3 and SKH51 were found at gas pressure of 3.5, 5.5 and 6.5 mTorr. Friction coefficient of modified SKS3 is decreased but SKH51 is slightly increased after nitriding modification. Friction coefficient of both nitrided steel are slightly decreased with raising processing gas pressure.

Keywords: PIII nitriding process, nano hardness, compound layer, fracture toughness, depth hardness

I. INTRODUCTION

Plasma immersion ion implantation (PIII) has a good potential in the application of industries, for example, for precision cutting tools, orthopaedics (medical prostheses) or small and complex shape aero precision parts [1,2]. Many researches indicate that chemical and mechanical properties (for example, wear reduction and surface fatigue life prolongation) of material surface can be improved by low energy ion implantation (100 keV)[3-5]. Li et al.[6] denote that the implantation voltage and nitriding temperature affect the phase and composition of nitriding layers. From Karaog (lu's analysis [7], increasing the nitriding temperature can make the nitriding layer transform from ϵ -phase to γ' -phase under nitriding process. The anti-wear property and hardness of nitriding layer increase with raising the γ' -phase. Leineweber et al.[8] heated nitriding steel to 550°C and maintained it for five hours, followed by annealing to 357°C. They found that the total thickness of the nitriding layer (combination of compound layer and nitriding layer) does not change, but the compound layer thickness becomes thinner. It gives that the ϵ -phase steel (Fe2-3N) transforms to γ' -phase (Fe4N) under annealing process. From the above information, the phase transformation of nitriding steel has been thoroughly investigated. However, the studies of mechanical properties for the compound and nitriding layer, including hardness and toughness, are few. Therefore, it is valuable to explore these mechanical properties in order to promote the PIII function effectively.

II. EXPERIMENT

The PIII apparatus used for nitriding experiment is shown in Figure 1. Microwave penetrates into a vacuum chamber surrounded by magnetic field produced by a set of electromagnets. Through the action of the well-known electron cyclotron resonance (ECR) phenomenon, high ion density plasma is obtained. Two kinds of tool steels, SKS3 and SKH51, are chosen for PIII nitriding treatments. The elemental compositions of them are listed in Tab. 1. These specimens are polished down to 0.05 μm in finish, subsequently cleaned by acetone, and then put on the specimen holder. The mixture of nitrogen and hydrogen gasses with flow ratio 1:1 is introduced into the chamber. Once the plasma is initiated with ECR source, a high pulse voltage is applied at the specimen holder, and nitrogen ions will be accelerated into specimen. Specimen's temperature is always kept at 530°C during the 2.5 hours PIII nitriding process, and the mixed gas pressure used for this study varies between 2.5-6.5 mTorr.

Table 1 Compositions of SKS3 and SKH51[9]

	C (%)	Si(%)	Mn(%)	W(%)	Cr(%)	Fe
SKS3	0.9-1.05	0.15-0.35	0.8-1.1	1.2-1.6	0.9-1.2	Bal
SKH51	0.8-0.9	0.2-0.45	0.15-0.4	5.5-6.75	3.8-4.4	Bal

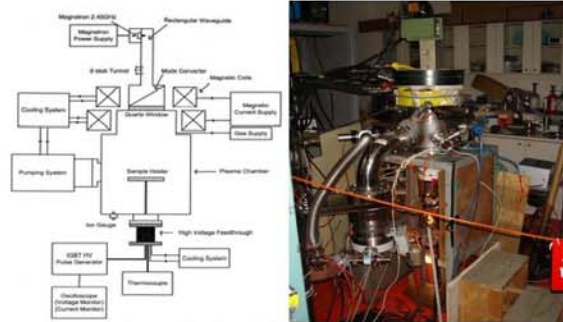


Figure 1 Sketch and photo of a PIII&N equipment.

III. RESULTS AND DISCUSSION

Mechanical properties and compositions of compound and nitriding layers were studied. We can see the multilayer structure in the cross section of steel sample, as shown in Fig.2, and the arrows marked in the picture distinguish the region of each layer. The approximate thickness of these layers are extracted and listed in Tab.2. Layer A is the compound layer (white layer) and layer B and C are nitriding layers. The compound layer's thickness of SKS3 increases monotonically with gas pressure, but the nitriding layer's thickness does not.

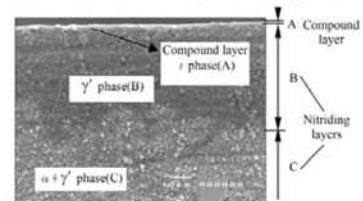


Figure 2 SEM micrograph of SKH51 with the processing gas pressure of 2.5 mTorr.

Table 2 Layer thicknesses of SKS3 and SKH51 at different gas pressures. (approximately measure)

Gas Pressure (mTorr)	Estimated Thickness (μm)					
	Compound Layer A		Nitriding Layer B		Nitriding Layer C	
	SKS3	SKH51	SKS3	SKH51	SKS3	SKH51
2.5	0.5	0.74	23.2	40.1	55.3	88.8
3.5	0.6	0.73	17.6	16.4	44.7	143
4.5	0.75	1.85	17.7	157	65.2	0
5.5	0.9	1.82	17.9	186	61.8	0
6.5	1.02	1.63	19	185	61	0

For SKH51, the compound layer's thicknesses increase also SKH51. Their intensities of diffraction peaks do not change apparently with varying gas pressures. The composition of each nitriding layer has been obtained from the analyses of XRD and GDOS. Figure 3 (a) and (b) show the results of XRD for SKS3 and SKH51 steels, respectively. Two components, $\text{Fe}_{2.5}\text{N}$ and Fe_4N , appear in SKS3, and SKH51.

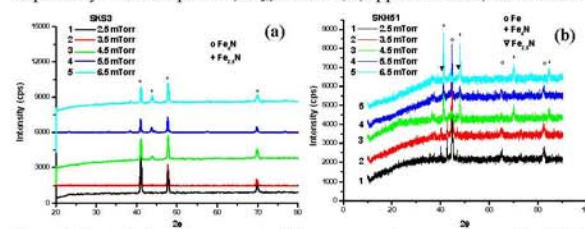


Figure 3 Crystal phase structures at different processing gas pressures by XRD analysis for (a) SKS3;(b) SKH51.

Figure 4 shows results of atomic ratio of nitrogen (analyzed by GDOS) and hardness in depth of these two types of steels. The atomic ratio of nitrogen and hardness decrease with increasing depth. Comparing among SEM micrograph in Fig. 2, the phase structures in Fig. 3, distributions of atomic ratio of nitrogen in Fig. 4, we can divide the depth into four regions. Region A is a compound layer, and its main composition is $\epsilon\text{-Fe}_{2.5}\text{N}$. Region B and C are nitriding layers, and their compositions are $\gamma'\text{-Fe}_4\text{N}$ and $\gamma'\text{-Fe}_4\text{N}+\alpha\text{-Fe}$, respectively. Also the hardness in depth decreases along with the atomic ratio of

四、心得與建議

國內精密機械加工技術優良，且產業聯結集中，英美等先進國家在開發高精度加工平台上成本昂貴且不易，因此國內學界可以善用此優勢拓展與海外合作開發之契機。此次參與此國際材料磨耗研討會，得知材料科技之發展趨勢，由其是複合多層陶瓷與金屬薄膜可有效增加軸承磨耗壽命，未來可嘗試應用於滾珠螺桿等精密元件研究，提昇國內精密機械之技術。