行政院國家科學委員會專題研究計畫 成果報告

以電子自旋共振研究稀磁半導體氧化物奈米顆粒暨薄膜
研究成果報告(精簡版)

計畫類別：個別型
計畫編號：NSC 99-2112-M-034-001-
執行期間：99年08月01日至101年01月31日
執行單位：中國文化大學物理學系

計畫主持人：江文中

計畫參與人員：大專生-兼任助理人員：陳經瑞
大專生-兼任助理人員：陳品寰

公開資訊：本計畫可公開查詢

中華民國 101年04月30日

中文摘要：本計畫係研究稀磁半導體氧化物二氧化鈦（TiO2）之奈米顆粒與薄膜。我們將深入探討該類材料在不同摻雜條件下呈現鐵磁性的機制，同時嘗試對缺陷密度、微結構、形態以及薄膜介面進行調制，以釐清自旋如何透過偏振子交換作用達成有序排列。

近年來研究人員對於稀磁半導體材料之興趣持續加溫，尤其是過渡金屬氧化物系統，原因是它們在適當的摻雜下，除了呈現出鐵磁性外，其居里溫度可超過室溫，因此在自旋電子學元件的發展上與應用上深具潛力。本計畫針對稀磁半導體氧化物 TiO2 系統，探索其在奈米顆粒及薄膜型態下的鐵磁性行為。在先前的研究工作中，我們發現 TiO2 奈米顆粒在電子自旋共振量測中呈現一些有趣的現象，尤其經過鐵、鉻與鎳摻雜後，其自旋訊號與鐵磁訊號具有某種程度的關聯，可用來探討自旋或鐵磁序化的機制。本專題計畫具有三重目標：（一）嘗試以不同的製程與鍍膜技術將上述材料製作成奈米顆粒與薄膜，並且有系統地研究其結構特性；（二）以電子自旋共振做為主要實驗工具，探討 TiO2 鐵磁序化的機制。實驗的變因將包括溫度、摻雜物、摻雜密度、缺陷密度以及微結構的調制等，並配合結構與其他磁性分析，了解該類材料系統結構與磁性表現之關聯；（三）藉由電子自旋共振在不同外場方向下所呈現的異向表現，研究 TiO2 薄膜的異向性，並深入剖析由介面誘發的相關磁性行為。因執行期間計畫主持人遭逢家中變故，原計畫中關於薄膜的部分未及完成，期於後續計畫中繼續深入探討。

中文關鍵詞：稀磁半導體、二氧化鈦、氧化鋅、奈米顆粒、薄膜、鐵磁序化、摻雜物、摻雜密度、缺陷密度、電子自旋共振、異向性、偏振子

英文摘要：This project is about the study of dilute magnetic semiconductor (DMS) oxide TiO2 in the forms of nanoparticle and thin film. We would like to investigate the mechanism of ferromagnetic ordering displayed by these materials under different doping conditions. By tuning some experimental parameters such as defect concentration, micro-structure, morphology and interface, we hope to get deeper insight into how localized spins align themselves into ferromagnetism through the exchange interaction of bound polarons.

There has been an increasing interest in the topic of dilute magnetic semiconductors in recent years, and especially in transition-metal oxide systems because when properly doped, they not only exhibit a ferromagnetic ordering, but the Curies temperature is also above room temperature. Therefore DMS materials have great potential for the development of future spintronic devices. This project intends to focus on TiO2 DMS oxide system and to study the magnetic behaviors of its nanoparticle and thin-film forms. In our preliminary study of TiO2 nanoparticles, we have found some interesting phenomena. When doped with Fe, Cr, and Ni, the ESR and FMR signals are correlated in some sense, and can therefore be used to probe the spin or ferromagnetic ordering mechanism. The aim of this study has three folds: (1) to make nanoparticles and thin films of TiO2 with various techniques, and to characterize their structural properties systematically； (2) to study the magnetic ordering mechanism of these materials, using Electron Spin Resonance (ESR) as a primary tool. The experimental variables would include temperature, dopant, doping concentration, defect concentration, micro-structure variation, etc. By carefully tuning and characterizing the structural and magnetic properties, we hope to reveal more information of how ferromagnetism correlates with the structural imperfections； (3) to explore other related magnetic issues in the thin-film type samples such as magnetic anisotropy. We would also like to explore the interface-induced effect and see how it would correspond in the anisotropic behavior of ESR signals.

英文關鍵詞： dilute magnetic semiconductor, TiO2, ZnO, nanoparticle, thin film, ferromagnetic ordering, dopant, doping concentration, defect concentration, electron spin resonance, anisotropy, polaron

行政院國家科學委員會補助專題研究計畫 成果報告

以電子自旋共振研究稀磁半導體氧化物奈米顆粒暨薄膜

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執行單位： 中國文化大學物理系

計畫主持人： 江 文 中
共同主持人： 無
計畫參與人員： 陳經瑞、陳品寰

應繳報告類型(依經費核定清單規定繳交)： 精簡報告

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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

中　華　民　國　101　年　4　月　30　日

（一）中、英文摘要及關鍵詞 (keywords)

關鍵詞: 稀磁半導體、二氧化鈦、氧化鋅、奈米顆粒、薄膜、鐵磁序化、摻雜物、摻雜密度、缺陷密度、電子自旋共振、異向性、偏振子

本計畫係研究稀磁半導體氧化物二氧化鈦 (TiO_2) 之奈米顆粒與薄膜。我們將深入探討該類材料在不同摻雜條件下呈現鐵磁性的機制，同時嘗試對缺陷密度、微結構、形態以及薄膜介面進行調制，以釐清自旋如何透過偏振子交換作用達成有序排列。

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Keywords: dilute magnetic semiconductor, TiO2, ZnO, nanoparticle, thin film, ferromagnetic ordering, dopant, doping concentration, defect concentration, electron spin resonance, anisotropy, polaron

This project is about the study of dilute magnetic semiconductor (DMS) oxide TiO_2 in the forms of nanoparticle and thin film. We would like to investigate the mechanism of ferromagnetic ordering displayed by these materials under different doping conditions. By tuning some experimental parameters such as defect concentration, micro-structure, morphology and interface, we hope to get deeper insight into how localized spins align themselves into ferromagnetism through the

exchange interaction of bound polarons.

There has been an increasing interest in the topic of dilute magnetic semiconductors in recent years, and especially in transition-metal oxide systems because when properly doped, they not only exhibit a ferromagnetic ordering, but the Curies temperature is also above room temperature. Therefore DMS materials have great potential for the development of future spintronic devices. This project intends to focus on TiO_2 DMS oxide system and to study the magnetic behaviors of its nanoparticle and thin-film forms. In our preliminary study of TiO_2 nanoparticles, we have found some interesting phenomena. When doped with Fe, Cr, and Ni, the ESR and FMR signals are correlated in some sense, and can therefore be used to probe the spin or ferromagnetic ordering mechanism. The aim of this study has three folds: (1) to make nanoparticles and thin films of TiO_2 with various techniques, and to characterize their structural properties systematically; (2) to study the magnetic ordering mechanism of these materials, using Electron Spin Resonance (ESR) as a primary tool. The experimental variables would include temperature, dopant, doping concentration, defect concentration, micro-structure variation, etc. By carefully tuning and characterizing the structural and magnetic properties, we hope to reveal more information of how ferromagnetism correlates with the structural imperfections; (3) to explore other related magnetic issues in the thin-film type samples such as magnetic anisotropy. We would also like to explore the interface-induced effect and see how it would correspond in the anisotropic behavior of ESR signals.

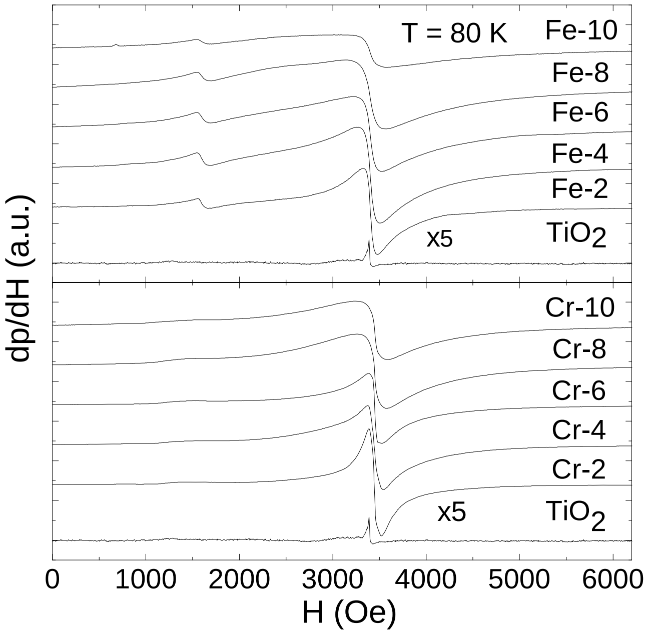
(二) 報告內容

To date, one of the challenging issues that remain controversial is the origin and mechanism of ferromagnetic ordering in doped-oxide type dilute magnetic semiconductors (DMS) such as TiO_2 and ZnO. Among the various theoretical predictions, two mechanisms suggested for the ordering: i.e. carrier mediated exchange and bound magnetic polaron (BMP) models, seem most widely accepted [1, 2]. Recent works of Z. L. Lu *et al*. have suggested that there are two distinct ferromagnetic mechanisms in different conductivity regimes for oxide-based DMS [3]. But exactly how polarons are developed and evolved in the insulating regime, and how the BMP mechanism crosses-over to the other are still questionable. Besides, either BMP or carrier mediated exchange is strongly governed by how dopants are incorporated and distributed in the host lattice. Oxygen vacancies and defects also play crucial roles in the magnetic ordering mechanism. A complete understanding of these issues requires an immense collection of studies from every possible perspective.

Here we prepare our Fe-, Cr-, and Ni-doped TiO_2 nanoparticles by the sol-gel method with a wide range of doping concentration. The low processing temperature results in a single anatase phase consistently in all samples, as verified by x-ray diffraction. The *M*-doped titanium dioxide nanoparticles (where M = Fe, Cr, and Ni) were synthesized by the sol-gel method for its advantage of obtaining good chemical homogeneity and unique metastable structures at relatively low reaction temperatures. The method incorporates metal inorganic salts as precursors. During the process of gel formation, metal ions were dispersed in the porous TiO_2 matrix and later powders of pure TiO_2 (with no metal ion added) and *M*-doped TiO_2 nanoparticles were prepared by drying, grinding and thermal treatment at different temperatures. Previous studies have demonstrated that thermal treatments affect both the morphology and the atomic-scale structure of TiO_2 nanoparticles in different ways [4, 5]. In our study, we chose to filter and dry the *M*-doped TiO_2 powders at 120˚C, whereas the sintering temperature was kept at 300˚C for five hours. The nominal partial concentration x for $Ti_{1-x}M_xO_2$ is set at 2%, 4%, 6%, 8%, and 10%. However, the maximum Ni concentration, as measured by Energy Dispersive X-ray analysis (EDX), is found to be only 2.3% for the sample which has a nominal value of 10%. The concentrations of Fe and Cr, on the other hand, are much closer to the nominal values.

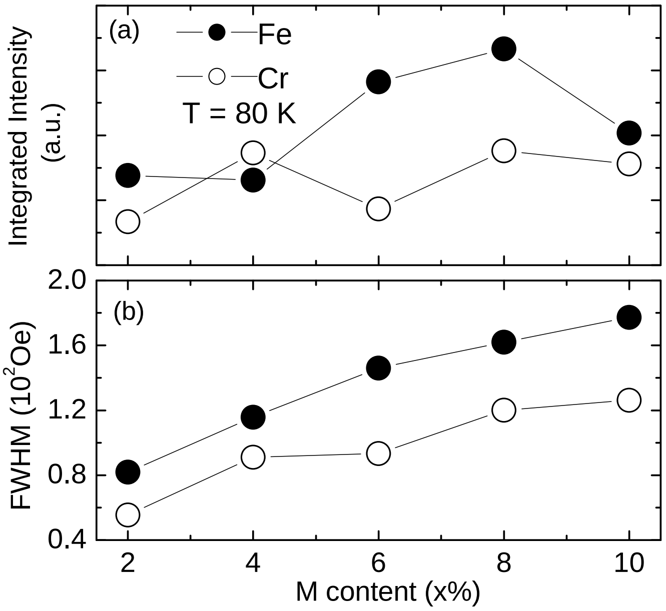
The magnetization vs. field (*M-H)* curves for Fe- Cr-, and Ni-doped samples were measured at 5K. Without doping, the TiO_2 nanoparticles are non-magnetic. At 2% concentration, both Fe- and Cr-doped particles exhibit weak hysteresis while the former display larger magnetization with field sweeping. In both cases, the size of magnetization increases monotonically with doping content while the saturation magnetization is not fully reached at field strength as large as 7.0 T. This could be caused by the occurrence of a spin-glass-like state that demands large field energy to overcome the anisotropy field and the dipole field of the nanoparticles [6]. The coercive field also increases with doping concentration. Measurement of Ni-doped particles shows weak paramagnetic behavior with magnetization at least an order of magnitude smaller than those of Fe- and Cr- doped samples.

The following figure shows the ESR spectra at 80 K for 0% (un-doped), 2%, 4%, 6%, 8%, and 10% Fe- and Cr-doped TiO_2 nanoparticles, in which the field derivative of specific microwave power absorption, dp/dH, is registered as a function of DC magnetic field H. The ESR measurements were carried out using the commercially available *X*-band (ν = 9.53 GHz) Bruker EMX spectrometer equipped with temperature variation accessory. The un-doped sample exhibits a small resonance line at g ~ 2.0, which can be attributed to the free electrons trapped by oxygen vacancies or structural defects created during the nanoparticle fabrication [7]. Previous studies have shown that TiO_2 nanoparticles are prone to defects, oxygen vacancies, and the occurrence of this free-electron spin resonance signal is rather sensitive to the TiO_2 particle size [8]. The smaller the particle size, the larger the content of oxygen vacancies and defects, and hence the signal is more likely to be observed. When a small amount of Fe or Cr is added into the matrix, the free-electron spin signal is replaced by a broad peak of enhanced intensity that grows with doping concentration. The exact g-value of this peak is less well-defined but is close to the value of 2. The peak's broad linewidth suggests that this is the ferromagnetic resonance (FMR) due to the dipolar interactions among the free spins induced by the doping. The Ni-doped samples exhibit no such peak enhancement and the spectra resemble that of pure TiO_2, evidencing again the limitation of Ti^{4+}/Ni^{2+} substitution. In additional to the broad FMR peak, a minor resonance line is detected for Fe- and Cr-doped samples at g ~ 4.30 and g ~ 4.12, respectively.



Field derivative ESR spectra for pure TiO_2, Fe-doped, and Cr-doped nanoparticles of various concentration, measured at 80 K. The intensity of pure TiO_2 spectrum is magnified by a factor of 5 for visual clarity.

A more quantitative analysis is made by correlating the microwave absorption intensity and the linewidth of the main resonance peak with doping concentration. The absorption intensity integrated over the main peak for Fe- and Cr-doped particles is plotted as a function of doping concentration, as shown below. For Fe-doped samples, the intensity increases monotonically with doping concentration except for the 10% sample, whose intensity drops by a notable amount from the 8% one. One also notices that the magnetization and the coercivity increments with the Fe content are also monotonic except from 8% to 10%. The cause of the nonlinearity could be that either a certain fraction of the Fe^{3+} ions are in the paramagnetic state, or there exist antiferromagnetic interactions when the content of Fe^{3+} ions is raised beyond a certain point [9]. The absorption intensity of Cr-doped samples fluctuates with Cr-doping, but exhibits an overall growing tendency. The full-width half-maximum (FWHM) of the resonance peak is plotted also as a function of doping concentration for Fe- and Cr-doped samples,. The linewidth in both cases increases with doping concentration. Comparing the spectra of Cr- and Fe-doped samples, the latter contain larger and broader ferromagnetic resonance signal, indicating a stronger coupling effect in the iron-based system.



Plots of (a) integrated intensity, and (b) full-width half-maximum of the main ferromagnetic resonance peak as functions of *M* content, where *M* denotes Fe or Cr. Ni-doped particles exhibit no such peak.

Temperature dependence of the integrated FMR peak intensity for Fe- and Cr-doped samples is observed to be like this: within the temperature variation range (80 ~ 450 K), the spectra preserve the original shape with no sign of phase transformation. The intensity of the main peak exhibits the characteristic decrease of FMR signal with temperature. From 80 K to 450 K, the decrease is more than 60% for most samples. The results indicate that our Fe- and Cr-doped TiO_2 particle systems are ferromagnetic and have Curie temperatures well above room-temperature.

The origin of the lower-field (g ~ 4.3 for Fe and g ~ 4.12 for Cr) resonance lines can be understood as follows. The g ~ 4.3 signal seen in the ESR spectra of Fe-doped samples is characteristic of isolated Fe^{3+} ions in a high spin configuration [10]. Previous studies have suggested that these Fe^{3+} ions are of rhombic symmetry and are mainly located in the anatase phase [11]. The general increase of the signal intensity with Fe-doping (except for the 10% sample) indicates the increase and the growing symmetry of the isolated Fe^{3+} ions. A coupling mechanism among these isolated ions is likely due to the broad and increasing linewidth of the peak. The resonance peak of g ~ 4.12 for Cr is less profound, and is likely due to similar mechanism.

In summary, the spectra of electron spin resonance on three series of metal-doped TiO_2 nanoarticles are investigated. The Ni-doped samples show low level of solubility, which makes Ni a non-ideal dopant for creating DMS materials. For Fe- and Cr-doped samples, a similar trend of temperature dependent signal intensity is observed, indicating the same magnetic coupling mechanism. The major resonance peak occurs at g ~2 with linewidth around 100 Oe, suggesting a dipolar interaction mechanism of doping-induced free spins in the nanoparticle system. On the other hand, the minor resonance peak is observed at g ~ 4.43(4.12), implying a magnetic coupling between Fe^{3+}-Fe^{3+} (Cr^{3+}-Cr^{3+}).

(三) 參考文獻

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(四) 計畫成果自評

1. 本計畫執行過程中，我們成功地利用溶膠凝膠法製作出一系列二氧化鈦奈米顆粒樣品，並在其中摻雜不同成份比例的鐵、鉻及鎳。X 光繞射實驗結果顯示該系列樣品為單一銳鈦礦結構，其中鐵、鉻與鈦的置換效果良好，而鎳則因原子大小與鈦差距較大，所以置換率受到限制。電子自旋共振與磁化率測量結果顯示，二氧化鈦於摻雜鐵、鉻之後，在室溫呈現微弱鐵磁性，其強度隨摻雜比例遞增；另一方面，鎳摻雜則因置換比例的限制，無鐵磁性呈現。這些實驗成果提供了該系列材料做為磁性半導體應用的有用資訊。此外，透過自旋共振，我們了解磁序化的主要機制來自於 trapped electrons 之間的磁偶極交互作用。這些結果將有助於釐清磁性半導體之所以具有磁性表現的基本物理問題。目前所有成果已大致撰寫完成，期於近期內發表於相關領域期刊。

2. 原計畫所提出關於磁性半導體氧化物薄膜製程方面，因合作夥伴時程協調問題，加上計畫主持人在計畫執行期間遭逢家庭變故，故無法於計畫結束前著手進行相關實驗，殊屬遺憾。由於在二氧化鈦奈米顆粒系統中，我們的確觀察到一些有趣的磁序化現象，因此令人好奇在薄膜系統中，其磁性是否呈現類似的行為，或完全是另一回事。不過將此類具有摻雜的材料製作成薄膜，在技術上將是另一項考驗。

3. 在未來展望方面，短期之內可透過合作關係尋求現成的磁性半導體氧化物薄膜樣品。舉例而言，成大黃榮俊教授團隊於二00九年發表了一系列關於氧化鋅單晶薄膜樣品，並利用摻雜 Co、Ga 等誘發其鐵磁性，以研究該磁性半導體磁序化的機制。研究顯示在不同的摻雜比例中，磁序化的成因並非單一。若能以電子自旋共振測量該系列薄膜樣品，相信可以獲得更多磁交互作用、甚至磁異向性方面的資訊。

4. 本計畫承多位物理界友人協助，其中最主要的合作者為國立台灣大學凝態研究中心林昭吟博士，實驗方面則有藍文伶、湯詠秀等，在此謹伸謝忱。

5. 本計畫所培育之二位大學部兼任研究助理，皆獲良好基礎研究訓練。目前兩位仍在本校大學部肄業，均有投考物理相關研究所，繼續進修的計畫。

無研發成果推廣資料

99 年度專題研究計畫研究成果彙整表

計畫主持人：江文中　**計畫編號：**99-2112-M-034-001-

計畫名稱：以電子自旋共振研究稀磁半導體氧化物奈米顆粒暨薄膜

成果項目			量化			單位	備註（質化說明：如數個計畫共同成果、成果列為該期刊之封面故事...等）
			實際已達成數（被接受或已發表）	預期總達成數(含實際已達成數)	本計畫實際貢獻百分比		
國內	論文著作	期刊論文	0	0	100%	篇	
		研究報告/技術報告	1	1	100%		
		研討會論文	0	0	100%		
		專書	0	0	100%		
	專利	申請中件數	0	0	100%	件	
		已獲得件數	0	0	100%		
	技術移轉	件數	0	0	100%	件	
		權利金	0	0	100%	千元	
	參與計畫人力（本國籍）	碩士生	0	0	100%	人次	
		博士生	0	0	100%		
		博士後研究員	0	0	100%		
		專任助理	0	0	100%		
國外	論文著作	期刊論文	0	1	100%	篇	
		研究報告/技術報告	0	0	100%		
		研討會論文	0	0	100%		
		專書	0	0	100%	章/本	
	專利	申請中件數	0	0	100%	件	
		已獲得件數	0	0	100%		
	技術移轉	件數	0	0	100%	件	
		權利金	0	0	100%	千元	
	參與計畫人力（外國籍）	碩士生	0	0	100%	人次	
		博士生	0	0	100%		
		博士後研究員	0	0	100%		
		專任助理	0	0	100%		

其他成果 (無法以量化表達之成果如辦理學術活動、獲得獎項、重要國際合作、研究成果國際影響力及其他協助產業技術發展之具體效益事項等，請以文字敘述填列。)	無

🟥	**成果項目**	**量化**	**名稱或內容性質簡述**
科教處計畫加填項目	測驗工具(含質性與量性)	0	
	課程/模組	0	
	電腦及網路系統或工具	0	
	教材	0	
	舉辦之活動/競賽	0	
	研討會/工作坊	0	
	電子報、網站	0	
	計畫成果推廣之參與（閱聽）人數	0	

國科會補助專題研究計畫成果報告自評表

請就研究內容與原計畫相符程度、達成預期目標情況、研究成果之學術或應用價值（簡要敘述成果所代表之意義、價值、影響或進一步發展之可能性）、是否適合在學術期刊發表或申請專利、主要發現或其他有關價值等，作一綜合評估。

1. 請就研究內容與原計畫相符程度、達成預期目標情況作一綜合評估

■達成目標
□未達成目標（請說明，以 100 字為限）
　　□實驗失敗
　　□因故實驗中斷
　　□其他原因
說明：

2. 研究成果在學術期刊發表或申請專利等情形：

論文：□已發表 ■未發表之文稿 □撰寫中 □無
專利：□已獲得 □申請中 ■無
技轉：□已技轉 □洽談中 ■無
其他：（以 100 字為限）

3. 請依學術成就、技術創新、社會影響等方面，評估研究成果之學術或應用價值（簡要敘述成果所代表之意義、價值、影響或進一步發展之可能性）（以 500 字為限）

本計畫執行過程中，我們成功地利用溶膠凝膠法製作出一系列二氧化鈦奈米顆粒樣品，並在其中摻雜不同成份比例的鐵、鉻及鎳。X 光繞射實驗結果顯示該系列樣品為單一銳鈦礦結構，其中鐵、鉻與鈦的置換效果良好，而鎳則因原子大小與鈦差距較大，所以置換率受到限制。電子自旋共振與磁化率測量結果顯示，二氧化鈦於摻雜鐵、鉻之後，在室溫呈現微弱鐵磁性，其強度隨摻雜比例遞增；另一方面，鎳摻雜則因置換比例的限制，無鐵磁性呈現。這些實驗成果提供了該系列材料做為磁性半導體應用的有用資訊。此外，透過自旋共振，我們了解磁序化的主要機制來自於 trapped electrons 之間的磁偶極交互作用。這些結果將有助於釐清磁性半導體之所以具有磁性表現的基本物理問題。目前所有成果已大致撰寫完成，期於近期內發表於相關領域期刊。