# การออกแบบและสร้างระบบแมกนิตรอนสปัตเตอริงแบบพัลส์ดีซีสองขั้ว สำหรับการสังเคราะห์ฟิล์มบาง

## Design and Construction of Bipolar Pulsed-DC Magnetron Sputtering System for Thin Films Synthesis

วีระศักดิ์ ซอมขุนทด (Weerasak Somkhunthot)<sup>1\*</sup>
ธนูสิทธิ์ บุรินทร์ประโคน (Thanusit Burinprakhon)<sup>2</sup>
นุวัติ พิมพะบุตร (Nuwat Pimpabute)<sup>3</sup>
เอียน โทมัส (Ian Thomas)<sup>4</sup>
วิทยา อมรกิจบำรุง (Vittaya Amornkitbamrung)<sup>5</sup>

## บทคัดย่อ

ได้พัฒนาระบบแมกนิตรอนสปัตเตอริงแบบพัลส์ดีซีสองขั้วไม่สมมาตรสำหรับการสังเคราะห์ฟิล์มบาง ระบบ ประกอบด้วยเครื่องจ่ายไฟฟ้าแบบพัลส์ดีซี ปืนแมกนิตรอนสปัตเตอริงแบบระนาบวงกลมไม่สมดุล เป้าทองแดง เส้นผ่านศูนย์กลางสองนิ้ว และระบบสุญญากาศ จากการทดสอบโดยใช้แก๊สอาร์กอนความดัน 40 มิลลิทอร์ พบว่า ที่ความต่างศักย์ไฟฟ้าลบของแคโทดระหว่าง -500 ถึง -600 โวลต์ และความต่างศักย์ไฟฟ้าบวกของ แอโนด 0-120 โวลต์ สามารถทำให้เกิดแมกนิตรอนอาร์กอนพลาสมาได้สำเร็จ โดยทดสอบที่ความถี่พัลส์ 17 กิโลเฮิรตซ์ และทำให้เกิดความหนาแน่นกระแสที่ผิวของเป้าประมาณ 15-25 มิลลิแอมแปร์ต่อตารางเชนติเมตร เงื่อนไขปฏิบัติการดังกล่าวนี้สามารถนำไปประยุกต์ใช้สำหรับการเตรียมฟิล์มบางทางเทอร์อิเล็กทริกของ  $\mathrm{Na\,Co_2O_4}$  และสารประกอบออกไซด์อื่น ๆ ได้

#### **Abstract**

An asymmetric bipolar pulsed-dc magnetron sputtering system has been developed for thin films synthesis. The system consists of a pulsed-dc power supply, an unbalanced circular planar magnetron sputtering gun supporting a two inch diameter target, and a vacuum system. A test with a copper target under argon atmosphere of 40 mtorr showed that the pulsed-dc magnetron argon plasma can be successfully generated with a cathode negative voltage between -500 to -600 volts and the reverse positive pulse of 0-120 volts. The pulse frequency was 17 kHz and the corresponding current density at the target surface was approximately in the range of 15-25 mA/cm<sup>2</sup>. These operating conditions could be applied for the preparation of Na  $_x^{\text{CO}}_{2}^{\text{O}}_{4}$  thermoelectric thin films and other oxide compounds.

คำสำคัญ: ระบบแมกนิตรอนสปัตเตอริงแบบพัลส์ดีซี การสังเคราะห์ฟิล์มบาง **Keywords:** Pulsed-dc magnetron sputtering system, thin films synthesis

นักศึกษาปริญญาเอก ภาควิชาฟิสิกส์ คณะวิทยาศาสตร์ มหาวิทยาลัยขอนแก่น, ผู้ช่วยศาสตราจารย์ มหาวิทยาลัยราชภัฏเลย

<sup>&</sup>lt;sup>2</sup>ผู้ช่วยศาสตราจารย์ ภาควิชาฟิสิกส์ คณะวิทยาศาสตร์ มหาวิทยาลัยขอนแก่น

<sup>&</sup>lt;sup>ู้</sup>นักศึกษาปริญญาเอก ภาควิชาฟิสิกส์ คณะวิทยาศาสตร์ มหาวิทยาลัยขอนแก่น, อาจารย์ มหาวิทยาลัยราชภัฏเลย

⁴อาจารย์ ภาควิชาฟิสิกส์ คณะวิทยาศาสตร์ มหาวิทยาลัยขอนแก่น

⁵รองศาสตราจารย์ ภาควิชาฟิสิกส์ คณะวิทยาศาสตร์ มหาวิทยาลัยขอนแก่น

<sup>\*</sup>corresponding author, e-mail: weerasak1963@yahoo.co.th

#### Introduction

Pulsed-dc magnetron sputtering is the latest development of sputtering technology for thin films deposition. It has many advantages over others, namely, it is versatile and provides the ability to deposit thin films of oxide compounds at high deposition rate and to eliminate arcing problems of poisoned targets (Este and Westwood, 1998). Within the last decade, this technique has been widely applied for the deposition of thin films of many functional materials of improved performance. For example, Lee et al. used bipolar pulsed-dc magnetron sputtering to prepare ITO films for electrochromic device applications. Better electrochromic performance was found in comparison to that measured with commercially available ITO films (Lee et al., 2002). Wang et al. prepared MoS<sub>v</sub> coatings by unbalanced magnetron sputtering system. It was found that MoS coatings deposited by simple dc power tended to form rougher surfaces and had a higher edge orientation of crystallites with worse tribological performance than those prepared by bipolar pulseddc power (Wang et al., 2002). Andújar et al. reported preparation of hydrogenated amorphous carbon (a-C:H) films from asymmetric bipolar pulsed-dc methane discharges. The results indicate that bipolar pulsed-dc is a promising technology for deposition of high quality DLC thin films, when compared with rf or dc discharge (Andújar et al., 2003). Subsequently, this technique has also been successfully used for the deposition of TiN on surgical tools, Al<sub>2</sub>O<sub>3</sub> hard coatings on cemented carbide cutting tools, and TiN/Si<sub>3</sub>N<sub>4</sub> superhard nanocomposite coatings (Park et al., 2003; ú strand et al., 2002; Fink et al., 2004; Barshilia et al., 2006). Furthermore, a variety of the hard nitride coatings (TiN, CrN, TiAlN and  $Si_{_{3}N_{_{4}}}$ ) with superior properties and low defect density can be prepared using asymmetric bipolar pulsed-dc generator (Barshilia and Rajam, 2006). Thus, it can be seen that a pulsed-dc magnetron sputtering system is a very useful platform for thin films synthesis. It is of interest here to develop an in-house-made unit which would be beneficial for long-term and self-sustained research.

This paper reports the current state in the development of a pulse-dc magnetron sputtering system at Physics Department, Faculty of Science, Khon Kaen University. The construction details and a preliminary test on the current-voltage characteristics of the system are presented.

## **Pulsed-DC Magnetron Sputtering System**

The developed pulsed-dc magnetron sputtering consists of a pulsed-dc power supply, a magnetron sputtering gun, and a vacuum system, are as follows.

The pulsed-dc power supply can be divided into of three major parts. Firstly, the dc power sources and power controls are a pair of high voltage dc power supplies utilizing a phase control circuit for power delivery. Secondly, the pulse generator and the power switches are a pair of pulsing and power switching circuits. Thirdly, the control systems are feedback circuits for current and voltage control, display and safety measures. These are shown in a block diagram in Figure 1. The details have been reported elsewhere (Somkhunthot et al., 2006; Somkhunthot et al., 2007).

A magnetron sputtering gun designed and constructed in this work is shown in Figure 2. Its key components are a magnet assembly, a high voltage cathode, and a target. The magnetron is an unbalanced circular planar type using neodymium

ring magnets (Nd<sub>2</sub>Fe<sub>14</sub>B: 57.15 mm OD, 48.26 mm ID, 6.35 mm thick, SuperMagnetMan). The target holder was designed to accommodate a disc or powder targets, and allow easy target replacement. The gun assembly is water cooled.

The vacuum system is composed of a vacuum stainless steel chamber (main cylinder: 250 mm OD, 200.4 mm ID, 250 mm high) and a two stage rotary pump (BOC EDWARDS, model E2M40). The chamber was initially designed at the Physics Department, Khon Kaen University and was constructed by the Mechanical Shop, National Synchrotron Research Center, Thailand and is shown in Figure 3. The base pressure of the system is 10 mtorr.

## **Experiment**

For preliminary assessment of plasma generation, the developed bipolar pulsed–dc magnetron sputtering system was tested using a two inch diameter copper target (industrial grade). The chamber was first pumped down to 10 mtorr. With argon (Ar 99.99%) gas flow rate of 15 sccm the total working pressure was 40 mtorr. The reverse positive and cathode negative pulse widths of the power supply were fixed at 10 ms  $(+t_{on})$  and 20 ms  $(-t_{on})$ , respectively. The pulse off times were kept constant at 14 ms  $(+t_{off}$  and  $-t_{off})$  to ensure complete turning OFF of the power switch FETs. These values of timings give the corresponding pulse frequency of 17 kHz.

The experimental setup for test on the bipolar pulsed-dc magnetron sputtering system is shown in Figure 4. The voltage waveform between cathode and anode was measured by a x100 high voltage probe (Model HP-9258). The test of plasma generation was performed using asymmetric bipolar

pulsed-dc power. In this work, the pulsed-dc magnetron argon plasma was tested by varying the reverse positive voltage (+ $V_{\rm dc1}$ ) of 0-120 V while the cathode negative voltage (- $V_{\rm dc2}$ ) was kept constant at -500, -550, and -600 V. The discharge currents  $I_{\rm S1}$  and  $I_{\rm S2}$  were measured by internally connected r.m.s. ammeters (A1 and A2 in Figure 1).

### **Results and Discussion**

The resulting current-voltage characteristic is shown in Figure 5. From this figure, as the reverse positive voltage  $+V_{\text{dc1}}$  is varied from 0-120V, the magnitude of the output current  $I_{s_1}$  increased from 0 to 40 mA ( $I \propto V^{1/2}$ ). With the cathode negative voltage – $V_{\rm dc2}$  fixed at –500, –550, and 600 V, the output current  $I_{s_2}$  was constant at 80, 110, and 140 mA, respectively. These results indicate that the output of each power supply is independent. The corresponding current density at the target surface was approximately in the range of 15-25 mA/cm<sup>2</sup>. The photographs and waveforms of pulsed-dc magnetron argon plasma for  $-V_{dc2}$  = -500 V and  $+V_{\text{dc1}} = 0-120 \text{ V}$  are shown in Figure 6, indicating good operation in asymmetric mode and close to theoretical pulse. The results suggest that the constructed bipolar pulsed-dc unbalanced magnetron sputtering system can be applied for the deposition of oxide compounds such as ITO, ZnO and Na<sub>x</sub>Co<sub>2</sub>O<sub>4</sub>.

### **Conclusions**

The under development asymmetric bipolar pulsed-dc magnetron sputtering system can be successfully used to generate argon plasma at the cathode negative voltage between -500 to -600 volts, the reverse positive pulse of 0-120 volts, and

the total argon pressure of 40 mtorr. When the pulse frequency was 17 kHz the corresponding current density at the target surface was approximately in the range of  $15-25 \text{ mA/cm}^2$ . It is expected that the system can be applied for the preparation of Na  $_x$ Co $_2$ O $_4$ thermoelectric thin films and other oxide compounds. This will further investigated.

## **Acknowledgment**

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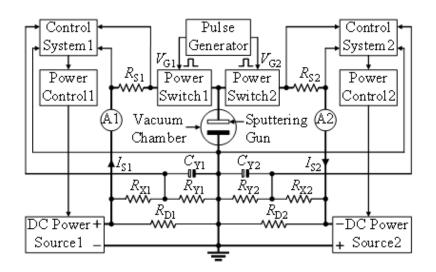


Figure 1 Block diagram of the pulsed-dc power supply for the magnetron sputtering system

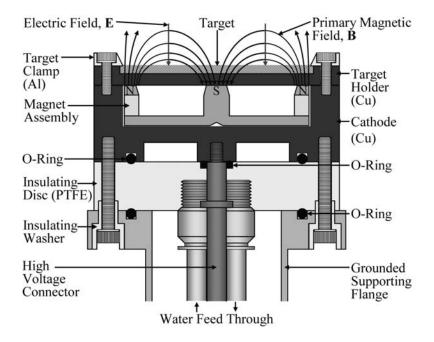


Figure 2 The designed magnetron sputtering gun

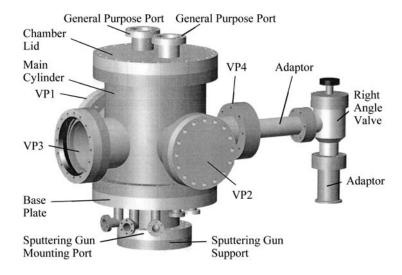


Figure 3 The designed vacuum chamber

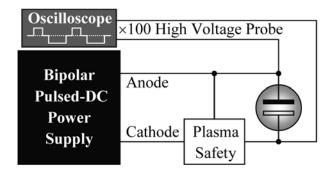


Figure 4 Experimental setup of the bipolar pulsed-dc magnetron sputtering system

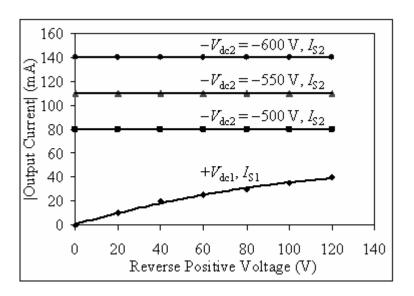
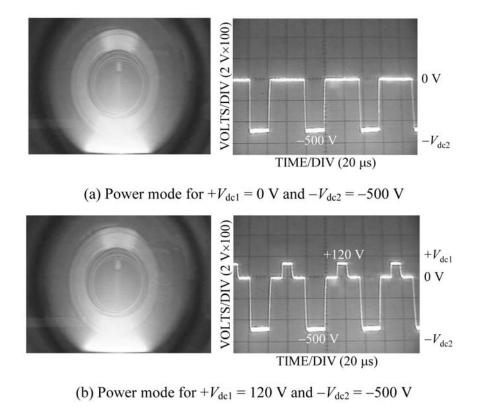


Figure 5 Plot of the current-voltage characteristic for the bipolar pulsed-dc magnetron argon plasma



**Figure 6** Photograph and waveforms of asymmetric bipolar pulsed-dc magnetron argon plasma pulsed at a frequency of 17 kHz