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### ABSTRACT

In this contribution, the effect of gas pressure and gas flow rate on the voltage (power) required to strike & maintain an argon plasma and the current – voltage characteristics as well as the deposition rates were investigated in detail using both ferromagnetic Ni as well as  $Ni_{81}Fe_{19}$  targets. A reduction in the voltage required to strike the plasma as the gas pressure and gas flow rate increased in the sputtering chamber and was attributed to the high probability of the ionisation process with increased gas pressure and gas flow rates. The relation between the voltage required to strike the glow discharge and the plasma current, showed a strong linear dependence under all gas pressures and gas flow rates investigated here. The current voltage relations showed a transition regions which were more clearly noticeable at low gas pressures and gas flow rates. They were attributed to the small amount of gas that existed in the chamber which in turn reduced the ionisation process. For constant gas pressure, lower voltage is required to strike the plasma when the gas flow rate is high and vice versa. The effect of the plasma power on the deposition rate under different gas pressure and flow rate was also considered here. The deposition rate was directly proportional to the plasma power where it increased linearly with increased plasma power. This means that the number of ejected atoms from the target increased linearly by increasing the target power. However, there was a slight reduction in the deposition rate by increasing the gas pressure and flow rates especially at high plasma power. This was attributed to the collisions enhancement among the ejected particlesor/and the gas atoms or due to the reduction in the thermal energy of the sputtered atoms and changing their direction far from the substrate.

Keywords: Current-Voltage characteristics, Glow discharge, Gas flow rate, Gas pressure, Plasma power.

### **INTRODUCTION**

A range of fabrication techniques have been widely used to deposit ferromagnetic thin films and nanostructures. Each technique has its own compensations and drawbacks. Among these techniques, sputtering has been proved to be simple, fast and low cost technique. Sputtering, however, can be defined as the removal of the surface material when it is bombarded with highly energetic ions. It was first discovered in 1852 by W. R. Grove when he was working on the electrical conductivity of gases [1].

In a sputtering system, an electric field is applied between two metallic electrodes known as the cathode and the anode that are sited in an evacuated chamber. The electrodes can be connected to a D.C. or R.F. electrical power supply [1]. A schematic description of a conventional sputtering system is shown in Figure 1(a). The target material is usually placed on the surface of the cathode. The chamber is filled with a low pressure inert gas, such as; argon, xenon and krypton. The gas becomes positively ionised when a sufficient electric field is applied to strike the glow discharge (plasma) between the two electrodes [1-3].

Due to the negative voltage of the target, the positive ions are accelerated towards the target. As a result different phenomena can occur depending on the type of the material making up the target, the ion type, the ion energy, and other factors, including gas flow rate, gas pressure, plasma power, electrode dimensions, electrode spacing, the ratio of the electrode dimensions to the electrode spacing as well as the target history and deposition geometry[1-8]. These phenomena are: the ejection of atoms from the target, scattering and neutralisation of ions, the production of secondary electrons and ions, and ion implantation in the target with or without simultaneous target atom ejection [1,4].

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**Figure1.** Schematic diagram of (a) conventional, and (b) magnetron sputtering systems. Atoms are ejected from the target surface as a result of bombardment by ions such as argon or krypton ions. A series of permanent magnets is placed behind the target to capture the escaping electrons and increase the sputtered atoms.

The most important phenomenon in sputtering system is the ejection of atoms from the target. There are two different theories explaining this ejection [1,4]. The first theory is thermal vaporisation in which the ejection of atoms is due to the local vaporisation of the target surface as a result of heating due to the bombardment of high energy ions [1,4]. The second concept is the direct momentum transfer in which the sputtering occurs as a result of the transfer of kinetic energy from the incident ions to the target surface atoms and sputtering occurs as a result of the collision cascade in the surface of the target [1,4].

In the conventional sputtering system, however, high gas pressure is required to create the plasma between the two electrodes because there is a problem that not all the free electrons contribute to produce the plasma, thus reduces the rate of material deposited on to the substrate and increases the contamination [1,4]. Placing permanent magnets behind the target as schematically shown in Figure 1(b), to form a magnetron source was found to be effective in reducing the gas pressure needed for producing the plasma, increasing the rate of material deposited on to the substrate up to one order of magnitude with a very low contamination in the deposited films [1,9]. This is because the permanent magnet produces a magnetic field that extends parallel to the target surface and perpendicular to the plasma, thus captures the escaping electrons and densifying them in the immediate area of the target where they move in a spiral motion and as a result increase the ion bombardment and hence the number of sputtered atoms [9].

As an example, Figure 2 shows photographic images of a Ni target before and after sputtering, which clearly demonstrates the effect of using a permanent magnet behind the target. The ratio of the number of atoms removed from the target material to the number of incident ions is referred to as the sputtering yield [1,4].



**Figure2.** Photographs of a Ni target (a) before and (b) after sputtering showing the race track effect of the permanent magnet (magnetron) to densify and confine the plasma on the target.

The sputtering yield is strongly dependent on the energy of the incident ions, target materials and angle of incidence. The sputter yield decreases with the increase in the incident angles [1,4]. The crystalline structure of the target surface also significantly depends on whether it is poly-crystalline, single crystal or amorphous [4].

Since each sputtering system has its own characteristics depending on different conditions and parameters mentioned above, thus, this

article critically discusses the effect of gas pressure and gas flow rate on the voltage needed to strike the glow discharge and maintain it. The current - voltage characteristics of the plasma and the effect of the plasma power on the deposition rate under these conditions is also analyzed. All these measurements were performed using two different compositions of ferromagnetic Ni and NiFe targets.

### **EXPERIMENTAL PROCEDURE**

The magnetron sputtering system used here to investigate the most significant characteristics is shown in Figure 3(a). The lowest base pressure achieved within this system was around  $2 \times 10^{-6}$ T using a combination of rotary and turbo molecular pumps. This pressure may have been limited by the very large O-ring seal on the top flange. The sputtering gas used here was argon gas with a purity of 99.9 %, which entered the vacuum chamber through a variable leak gate valve. The gas flow rate in the chamber was controlled via this gate valve. When this valve was closed, a low flow rate was achieved and when it was half opened, medium flow rate was

defined, and a high flow rate was obtained with a fully open gate valve.

Different targets of ferromagnetic materials can be used with this system, but in this investigation, ferromagnetic Ni and Ni<sub>81</sub>Fe<sub>19</sub> were used. They were discs with a 50 mm diameter and 99.99 % purity. The target was mounted by mechanical supports on a water cooled backing plate and surrounded by a metallic shield, which acts as the anode. The construction of the magnetron and the target is shown in Figure 3(b). Throughout the work presented here, the distance between the anode and the target surface (cathode) was kept constant at ~3 mm. The distance between the target surface and the substrate holder was also kept constant at ~90 mm.

To provide the power needed to strike and maintain an argon plasma, the anode and the cathode were connected to a DC power supply with a maximum voltage of  $\sim 600$  volts and a power  $\sim 3.6$  kW. The target voltage and plasma current were measured with digital multimeters.



**Figure3.** Photographs of (a) DC magnetron sputtering system used in this article to investigate the most significant characteristics of it, and (b) the target set of this system in which the cathode is a planar disc of the material to be deposited surrounded by a metallic anode shield.

The deposition rate and film thickness were monitored during the film growth using a quartz crystal oscillator. This oscillator was calibrated using X-ray reflectivity scan [10].

Before starting the work presented here, the chamber was flushed with argon gas for approximately 5 minutes, in order to remove the residual air molecules or water vapour inside the chamber if any were left after evacuation. To clean the target surface from oxides or any other contaminates, a pre-sputtering process was performed for a time of ~5 minutes.

#### **RESULTS AND DISCUSSION**

The following subsections thoroughly discuss the most important results obtained from the characterisation of the magnetron sputtering system which is shown in Figure 3. A detailed analysis was performed to study the effect of gas pressure (3 mT - 8 mT) and gas flow rates (low, medium and high) in the sputtering

chamber on the voltage (power) required to strike the glow discharge & maintain it. The relation between the plasma power and the deposition rate under these conditions was also investigated here. The gas flow rates were controlled via the gate valve available in the way of the gas outlet passage, as discussed in the experimental section. All these measurements were performed using two ferromagnetic compositions of Ni and Ni<sub>81</sub>Fe<sub>19</sub> targets.

# Effect of Gas Pressure and Gas Flow Rates on the Plasma Voltage

The effect of gas pressure on the voltage which was needed to strike the glow discharge & maintain it under three different gas flow rates was investigated. Examples of the results obtained from this work are shown in Figure 4 using (a) Ni target and (b) Ni<sub>81</sub>Fe<sub>19</sub> target and (c) both results of Ni and Ni<sub>81</sub>Fe<sub>19</sub> targets.



**Figure4.** The relation between argon gas pressure and the voltage required to strike the glow discharge & maintain it using (a) Ni, (b)  $Ni_{81}Fe_{19}$  and (c) both results of Ni and  $Ni_{81}Fe_{19}$  targets, under three different gas flow rates as indicated in the figure captions. The dashed lines are used as a guide to the eye.

The results of this work show, in general, a reduction in the voltage required to strike the glow discharge & maintain it when the gas pressure is increased in the sputtering chamber. The voltage required to strike the glow discharge & maintaining it for Ni target is higher than for Ni<sub>81</sub>Fe<sub>19</sub> target under all pressures investigated here (see, Figure 4(c)).

As an example, the average voltage required to strike the plasma and is maintained at ambient gas pressures of ~3 mT (see, Figure 4(a)) is ~400 V and ~350 V for Ni and Ni<sub>81</sub>Fe<sub>19</sub> targets, respectively. It should also be noted that there is a very slight variation in the voltage required to strike the plasma & maintain it by changing the gas flow rate in the chamber. For instance, the voltage needed to strike the plasma & maintain it at a pressure of ~3 mT (see, Figure 4(a)) using Ni target was around 410 V and 395 V for low and high gas flow rates, respectively. These variations, however, can be attributed to the high probability of the ionization process as the gas pressure or/and gas flow rates is increased in the chamber.

### Effect of Gas Pressure and Gas Flow Rates on the Current–Voltage of the Plasma

The relation between the voltage required to strike the glow discharge & maintain it and the current of the plasma was investigated here in detail. These investigations were conducted using different gas pressures and gas flow rates, as shown in Figure 5(a-d) using both Ni & Ni<sub>81</sub>Fe<sub>19</sub> targets at a constant gas flow rate and at a constant gas pressure, as shown in Figure 6(a-d) using Ni<sub>81</sub>Fe<sub>19</sub> as an example target.

In general, the results of this study demonstrate a very strong linear dependence of plasma current with the applied voltage under all gas pressures and gas flow rates used here. This means that a slight increase in the voltage increases the ionization process which in turn increases the current of the plasma.



**Figure5.** Current - voltage characteristics of the plasma using (a-b) Ni and (c-d)  $Ni_{81}Fe_{19}$  targets under different gas pressures and gas flow rates as indicated in the figure captions. The dashed lines are used as a guide to the eye. Note the difference in the current scale bars for the different target materials.



**Figure6.** Current - voltage characteristics of the plasma using  $Ni_{81}Fe_{19}$  as an example target under different gas pressures and gas flow rates as indicated in the figure captions. The dashed lines are used as a guide to the eye.

This relation also demonstrated a transition regions which were more clearly noticeable at low gas pressures and gas flow rates (see, for example, Figure 6(a)). These transition regions might appear as a result of the small amount of gas which exist at low pressures and flow rates, which in turn reduces the ionization process and hence the plasma current.

Again from Figures 5 & 6, there is a remarkable effect of the gas flow rate and gas pressure on the voltage required to strike the plasma & maintain it. For constant gas pressure (Figure 6(a-d)), lower voltage is required to strike the plasma when the gas flow rate is high, whilst higher voltage is required to strike the plasma when the low gas flow rate is used. There is clearly no effect of gas flow rate on this behaviour, as the gas pressure increased in the sputtering chamber to more than 6 mT (see, Figure 6(a-d)). The discrepancy in the voltage needed to strike the plasma & maintain it can be attributed to the differences in the gas ionisation with the change in the gas pressure and gas flow rates.

# Effect of Plasma Power on the Deposition Rate

The ratio of the generated ions during the ionisation process to the sputtered atoms from the target and deposition on the substrate is known as the deposition rate,  $A^{\circ}/s$ . The relation between the plasma power and the deposition rates was investigated here in detail using different gas pressures and gas flow rates. The results of such measurements are shown in Figure 7(a-f) using Ni or Ni<sub>81</sub>Fe<sub>19</sub> as an example targets. Clearly, the deposition rate is directly

proportional to the plasma power, where, it increases linearly with the increased plasma power. As an example, when Ni is used as a target, the deposition rate increases from 1.8 A<sup>o</sup>/s to 5.2 A<sup>o</sup>/s with the increased plasma power from 150 W to 600 W (see, Figure 7(a)). This is a very high deposition rate in comparison with the deposition rates of R.F. sputtering systems, where the deposition rate in these systems was found to be less than 0.05 A<sup>o</sup>/s using higher plasma powers [4-5]. There is also obviously no effect of gas flow rate or gas pressure on this linear dependency. It should also be noted that the deposition rate of Ni target is lower than the deposition rate of  $Ni_{81}Fe_{19}$ target under the same conditions (see, Figure 7(a-b)). For example, the deposition rate of Ni and  $Ni_{81}Fe_{19}$  when they were used as targets at a power of 150 W is 1.6A°/s and 3.2 Ao/s, respectively.

This variation might be due to the difference in the atomic weight and size of the ejected atoms of different materials. There is a slight reduction in the deposition rate by increasing the gas pressure and gas flow rates, especially at high plasma power, as can be seen in Figure 7(c-d) and Figure 7(e-f), respectively. This might be due to the enhancement of the collisions of the ejected atoms with each other or/and the gas atoms/ions or due to the reduction of the thermal energy of the sputtered atoms and changing their direction and accordingly the deposition kinetics are changed. Thus, the deposition rate can be modified by changing the gas pressures, gas flow rates and the plasma voltage applied between the two electrodes.





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**Figure7.** The relation between the plasma power and the deposition rate using Ni or  $Ni_{81}Fe_{19}$  as a target under (a-d) constant gas flow rate and (c-f) constant gas pressure, as indicated in the figure captions. The dashed lines are used as a guide to the eye.

#### CONCLUSION

Here, the detailed analysis of the magnetron sputtering system characterisation was analysed. These investigations were performed to study the effect of gas pressure and gas flow rate on the voltage required to strike the glow discharge & maintain it, the current – voltage characteristics and the relation between the plasma power and the deposition rate under these conditions. All these measurements were carried out using both Ni and Ni<sub>81</sub>Fe<sub>19</sub> targets.

There was a remarkable effect of the gas flow rate and gas pressure on the power needed to strike the plasma & maintain it where a reduction in the voltage required to strike the plasma was noticed as the gas pressure and gas flow rate increased in the chamber. This behaviour was attributed to the high probability of the ionization process with the increased gas pressure and gas flow rate. The relation between the voltage required to strike the glow discharge & maintain it and the plasma current showed a strong linear dependence under all gas pressures and gas flow rates investigated here. The current

- voltage relations showed a transition regions which were more noticeable at low gas pressure and gas flow rate. They were attributed to the amount of gas that existed in the chamber which in turn affect the ionisation process. For constant gas pressure, lower voltage is required to strike the plasma & maintain it when the gas flow rate is high and vice versa. The effect of the plasma power on the deposition rate under different gas pressures and gas flow rates was also analysed here. The deposition rate was directly proportional to the plasma power, where it increased linearly with the increased plasma power. A slight reduction in the deposition rate was noticed with the increased gas pressure and This was attributed to the flow rate. enhancement of the collisions of the ejected atoms with each other and/or the gas atoms/ions and hence a reduction in their thermal energy and changing their direction.

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