Development and Characterization of a Low-cost Magnetron Sputtering System for Deposition of Metal Films on Borosilicate Glass Substrates

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March 8, 2023

Abstract

In this study, we present the development and characterization of a homegrown magnetron sputtering system (MSS) constructed from repurposed materials. Borosilicate glass slides were used as the substrate material, while Al, Fe, and Cu were employed as target materials. The system was validated, optimized, and successfully utilized for the deposition of Cu, Al, and Fe films onto the glass substrate. The resulting samples were analyzed for their structure, morphology, photoluminescence, and electrical conductivity using XRD, EDS, SEM, and the 4-point probe method. Our findings indicate that the MSS produces results that compare favorably with standard sputtering equipment, but with significant potential for further refinement. Overall, our work highlights the feasibility and versatility of utilizing homegrown sputtering systems for a range of research applications.
Development and Characterization of a Low-cost Magnetron Sputtering System for Deposition of Metal Films on Borosilicate Glass Substrates

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Funding information
This work did not receive any funding.

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KEYWORDS
magnetron, sputtering, thin-film, deposition, conductive film

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1 | INTRODUCTION

The demand for thin conductive films has increased significantly in recent years, leading to the use of various complex methods to coat substrates with conductive films. These methods include physical vapor deposition (PVD), chemical vapor deposition (CVD), electrolysis, and others [1]. However, in this article, our focus is on metal film sputtering deposition. The technique was first observed accidentally in the 1850s in the electrical vacuum devices of the time. Research into the process culminated in the first sputtered diode in the 1940s. It remains one the most used commercial techniques for making semiconductor devices today despite its deposition rates and economic yields being lower than the other techniques [2]. The basis of MS is an intense magnetic field that generates an electrical ionic plasma in a vacuum. The method does not require melting and evaporation of the source material, making it suitable for coating many substrates with thin conducting/nonconducting films with metals, alloys, and compounds. Films of up to 5 pm thickness are possible using MS [3].

This article is divided into two parts. The first part covers the construction, and evaluation based on standard spectroscopic measurements for the morphology, crystalline sizes, and film conductivity of the sputtered films. The second part describes the optimization stage of the MSS which is confirmed using the standard spectroscopic techniques of scanning electron microscopy (SEM), energy dispersive spectroscopy (EDS), x-ray diffraction (XRD), and photoluminescence spectroscopy (PL). The results compare favorably with those from standard sputtering equipment.

2 | EXPERIMENTAL

2.1 | Construction of the MSS

The MSS unit consists of a high-voltage power supply (HVPS), a vacuum sputtering chamber, a magnet arrangement, and an argon (Ar) gas circulation system. Fig. 1 shows the HVPS unit. It is a microwave oven high-voltage transformer operated in the step-up mode, with the primary driven by a variac. The secondary output voltage is rectified and doubled using the Cockcroft and Walton [4] method, to give an output voltage range of 0V to 6 kV. The transformation ratio (n) of the microwave oven transformer is approximately 9. This was established by injecting a known low voltage at its input and measuring the output. High-voltage wiring and construction techniques were used for the HVPS.

![Figure 1](image-url)

**Figure 1** A circuit diagram of a high-voltage DC power supply unit.
The sputtering chamber consists of a glass jar with a thick wall. The necessary holes were drilled in the glass using a tiling bit. The holes were the inlet for the argon (Ar) gas, and the outlet to the vacuum pump. Ar was chosen as the sputtering gas. It has two roles. First, it flushes oxygen out of the system prior to the ignition of the plasma. Second, it ionizes easily and can be accelerated and used to bombard the target, which leads to sputtering. The 20 mTorr vacuum atmosphere was created inside the chamber using two vacuum pumps connected in series. The magnet consisted of a single-ring magnet, also extracted from the magnetron itself. The measured strengths averaged 400 Gauss at its center. This field is concentrated in a small region that coincides with the placement of the target. With the MSS in operation, the plasma is observed within this region. The MSS was run in a precise sequence. With the HVPS off, the target was placed onto its mount inside the chamber. The chamber was then pumped down to the required pressure with the Ar cylinder closed. The pump was then stopped, and the Ar tap opened for approximately 20 seconds to flush Ar through the system. This causes the chamber to pressure rise. The Ar tap was then closed and the vacuum pump restarted until the chamber reached the required vacuum pressure. The HVPS output was then gradually raised from 0V until a plasma discharge was observed. The color of the plasma depends on the target material. The plasma was observed when the variac output voltage was at least 165 Vrms. This corresponds to a peak acceleration voltage of 4.2 kV. Fig. 2 is an image of the MSS in operation.

![Image of MSS in operation](image-url)

**FIGURE 2** The MSS in operation using a copper target, which gives a bluish-purple plasma. The plasma appears more intense to the camera’s image sensor than to the naked eye.

### 2.2 MSS optimization

The initial results of using the MSS showed intense sputtering on the substrate in the vicinity of the magnets. This caused intense heating of the substrate. A magnetic shield was fashioned using modified holders for both the substrate and the target. The sputtering was seen to depend also on the shape of the magnetic field. A stronger field appeared to deteriorate the quality of sputtering. The magnet assembly was replaced with a smaller ring magnet to concentrate the magnetic field more centrally on the target. Up to this point, Cu targets produced a visible sputtered film on the glass substrates placed about 7 mm away from the copper target. The sputtering time was 5 minutes for each run.
3 | RESULTS AND DISCUSSION

The morphology, elemental composition, structure, and electrical conductivity of the sputtered thin film were investigated using scanning electron microscopy (SEM), energy dispersive X-ray spectroscopy (EDS), photoluminescent spectroscopy (PL), x-ray diffraction (XRD) and current-voltage measurements, respectively. The modifications are based on the foregoing constructed system and the results obtained from them. The constructed and optimized system was then used to sputter additional metallic films, and secondary results were obtained.

3.1 | SEM and EDS measurements

Fig. 4 (a)-(f) show the images from the SEM measurements for Cu, Al, and Fe-coated samples. With respect to the SEM images of Al (top and bottom center images), at high magnification, it is apparent that sputtered material has the appearance of nanorods on the glass substrate. The average particle size is estimated at around 30 nm, which is comparable to the reported literature range of 20–50 nm for Al [5]. The magnetic field strength inside the sputtering chamber and sputtering rate were key factors affecting the resultant particle size and orientation. More well-oriented nanorods seemed to be the result of having longer sputtering rates. In Fig. 4 (b), plant-like structures that resemble polymers could be explained by some burning of the glass substrate. Nevertheless, on the EDS spectrum shown in Fig. 5 (b), the formation of polymers was discounted due to the absence of hydrogen, which is expected from polymers due to H-C bonds [6]. A randomly chosen initial sample thought to be Al was used. Commercial Al casings often include additives such as Si, Cu, and even Mg for additional mechanical strength. Fig. 3 shows SEM images of two samples of Al. Fig. 3 (a) shows the images made from sputtering a random Al sample, while Fig. 3 (b) shows a literature-sourced SEM of a sputtered Al sample with known Si influence. The appearance of the EDS spectra for Al could also be accounted for by added impurities present in the random Al sample. The EDS results presented in Fig. 5 (a)-(f) indicate the presence of oxygen, which suggests incomplete pumping of the sputtering chamber, possibly due to a low-efficiency vacuum pump or out-gassing from the chamber sealants. However, the absence of hydrocarbons and
contaminating polymer byproducts suggests that they did not significantly contribute to the final film composition. The elemental carbon signal in the spectra arises from the carbon pre-coating of the substrate for SEM characterization. The dominant Si peak is due to the high-temperature decomposition of the borosilicate glass substrate during MSS operation, which also contributes to the oxygen signal. The presence of Ca in the spectra is attributed to the sample preparation, which involved the use of Ca as an alkalinity regulator for EDS characterization. The observed Mg signal is likely due to the presence of Mg in the starting Al material, which is a common constituent in Al-Mg alloys used in the laboratory. The results suggest that the quality of the sputtered film is influenced by the purity of the initial sputtering material. Surprisingly, the EDS spectra for samples (c) and (f) did not show any Fe signal despite using an Fe target. However, SEM analysis revealed the presence of sputtered Fe with a crystallite size of 18.5 nm. The presence of Si and Mg suggests that the starting material was likely a metallic alloy.

### 3.2 XRD measurements

The XRD pattern of Cu particles deposited on glass substrate at higher sputtering voltage is shown in Fig. 6. XRD analysis is useful for crystal structure and orientation. The positions of peaks at 43.50°, 50.58°, 55.08° and 74.35° are illustrated in the diffraction spectrum. The peaks match the JCPDS (04-0836) standard data for Cu, with
the major peaks indicating the (111), (200), and (220) reflections. This implies that the deposited material is face-centered cubic (FCC) Cu. The observed (111) peak intensity is higher than for the peaks (200), (210), and, (220). This indicates high crystallinity due to the using a higher sputtering voltage and a stronger magnetic field, which appear to improve the deposition rate. Applying the Scherrer formula \[7\] gave an average deposited grain size of 48.21 nm, which is within the expected range 1 - 100 nm using sputtering \[8\]. The (210) reflection is due to impurities in the target sheet. Using the Bragg diffraction equation i.e.

\[ n\lambda = 2d \sin \theta, \]

which gives \(a = 3.726\)Å for the lattice parameter with \(n = 1\). This value closely matches 3.615 Å, the known value for Cu \[9, 10\]. Table 1 shows the data over the 2\(\theta\) measurement range. Fig. 6 shows the experimental dataset. Column 3 in Table 1 takes on approximately integer values when the constant divisor is 46 (=183-137). FCC materials exhibit high-intensity due to the (111) reflection. The three peaks at 2\(\theta\) values of 43.504\(^{\circ}\), 50.583\(^{\circ}\), and 74.356\(^{\circ}\) corresponding to (111), (200), and (220) planes of Cu are observed and compared with the standard powder diffraction card of JCPDS, copper file No. 04–0836. A comparison between the Cu experimentally obtained X-ray diffraction angle and the standard diffraction angle is illustrated in Table 2. The XRD analysis indicates that the resulting particles are FCC Cu nanoparticles sputter coated on the glass substrate. The average particle size was estimated at the diffraction peaks \[11, 12, 13, 14\] using the Debye-Scherrer formula:

\[ D = \frac{0.9\lambda}{\beta \cos \theta}, \]

FIGURE 5  EDS images of Cu (a and d), Al (b and e), and Fe (c and f), respectively. The magnification is x 10K (x 20K) in the top (bottom) row.
**FIGURE 6** XRD spectrum showing crystal structure of sputtered Cu.

**TABLE 1** The measured XRD peaks and their indexes.

<table>
<thead>
<tr>
<th>$2\theta$ (°)</th>
<th>$1000\times\sin^2\theta$</th>
<th>$1000\times\sin^2\theta/46$</th>
<th>Reflection</th>
<th>Calculation</th>
</tr>
</thead>
<tbody>
<tr>
<td>43.504</td>
<td>137</td>
<td>3</td>
<td>(111)</td>
<td>$1^2+1^2+1^2 = 3$</td>
</tr>
<tr>
<td>50.583</td>
<td>183</td>
<td>4</td>
<td>(200)</td>
<td>$2^2+0^2+0^2 = 4$</td>
</tr>
<tr>
<td>74.356</td>
<td>365</td>
<td>8</td>
<td>(220)</td>
<td>$2^2+2^2+0^2 = 8$</td>
</tr>
</tbody>
</table>

**TABLE 2** Experimental and standard diffraction $2\theta$ angles for the Cu specimen

<table>
<thead>
<tr>
<th>Experimental $2\theta_E$</th>
<th>Cu standard $2\theta_S$ (JCPDS 04-0836)</th>
</tr>
</thead>
<tbody>
<tr>
<td>43.504°</td>
<td>43.297°</td>
</tr>
<tr>
<td>50.583°</td>
<td>50.433°</td>
</tr>
<tr>
<td>74.356°</td>
<td>74.130°</td>
</tr>
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</table>
TABLE 3  The grain size of Cu nanoparticles.

<table>
<thead>
<tr>
<th>2θ</th>
<th>hkl</th>
<th>θ</th>
<th>β</th>
<th>D</th>
<th>d</th>
<th>a</th>
</tr>
</thead>
<tbody>
<tr>
<td>(°)</td>
<td></td>
<td>(°)</td>
<td>(rad)</td>
<td>(nm)</td>
<td>(Å)</td>
<td>(Å)</td>
</tr>
<tr>
<td>43.504</td>
<td>(111)</td>
<td>21.752</td>
<td>0.0036</td>
<td>41.48</td>
<td>2.079</td>
<td>3.60</td>
</tr>
<tr>
<td>50.583</td>
<td>(200)</td>
<td>25.292</td>
<td>0.0026</td>
<td>59.00</td>
<td>1.803</td>
<td>3.61</td>
</tr>
<tr>
<td>74.356</td>
<td>(220)</td>
<td>37.178</td>
<td>0.0039</td>
<td>44.15</td>
<td>1.275</td>
<td>3.61</td>
</tr>
</tbody>
</table>

where \( D \) is the particle size, \( \lambda \) is the wavelength of X-ray (0.1541 nm), \( \beta \) is the full-width at half-maximum (FWHM) in radians, \( \theta \) is the diffraction angle, and the constant (0.9) is the Scherrer constant. The change in 2\( \theta \) (radians) is then (experimental - standard) = (2\( \theta \)E - 2\( \theta \)S). The plane spacing, \( d \), and the lattice parameter, \( a \), can then be calculated using Eq. 1. Table 3 shows the \( \beta \) (rad), \( D \) (nm), \( d \) (Å) and \( a \) (Å) from the 2\( \theta \) positions of the planes indicated in the XRD in Figure 6. The crystal structure of Cu is FCC, with a unit cell edge \( a=3.60 \) Å. The equations above were used in the calculation of the expected 2\( \theta \) positions of the first three peaks in the diffraction pattern, \( a \), and the \( d \) for each peak as illustrated in Table 3. The experimental results are in good agreement with the library standard and the literature [15].

3.3 | PL measurements

Fig. 7 (a) shows the PL measurement done on an MSS sputtered Al thin film. The excitation (1), and the reflected (2) peaks, are the equivalent de Broglie wavelengths which are equivalent to the electron energies [16]. Peak 2 (655 nm) is lower than peak 1 (320 nm) due to diffuse electron scattering and trapping in the substrate and thin film. The measured peak 2 wavelength agrees with the literature value of 633 nm [17]. Fig. 4 (a) and (d) shows the Cu thin film at 10K and 20K magnifications, respectively. The figures show a smooth and continuous Cu film, but with visible grains and uncoated spots. The film appears to be well-compacted with no visible cracks. This is in contrast to other methods.

![Figure 7](image_url)

**FIGURE 7**  Measured PL spectra. In (a) for Al, showing strong emission at 320nm. Peak 1 is the excitation, and peak 2 is the response. In (b) for Cu. The deconvolution shows strong emission at 325nm.
such as electrode-less plating, which leave visible cracks in the film [18]. During deposition, however, changes in the parameters of the sputtering system are expected to influence the morphology of the Cu thin film, as suggested by the SEM images. Fig. 4 (d) shows spherical, granular Cu nanoparticles. This indicates that even very low Ar gas pressures during sputtering deposition can increase the substrate sputtering rate with the overall result that the shape of the Cu particles is altered to some extent. Fig. 5 (a) and (d) are the EDS spectra of the Cu sample. They indicate the presence of predominantly low-quality Cu, suggesting that the sample is also an alloy. However, the foregoing XRD results indicate that the film is of good quality and is predominantly monoclinic Cu [19]. Deconvolution indicates the recombination of bound excitons to charged vacancies. These vacancies are associated with single-charged copper Cu$^{+}$ and with double-charged copper Cu$^{2+}$. With increasing wavelength, the deconvoluted peak intensities decrease exponentially, but not at the same rate. An increase in their relative intensity indicates fewer structural defects, strongly suggesting a direct dipole transition. PL can distinguish the Cu$^{2+}$ cations in octahedral (Oh) and tetrahedral (Th) sites. The peak intensity increases with the concentration of deposited Cu [20]. These measurements show that the fraction of tetrahedral Cu increases, and as Cu content decreases, the resolution of the four peaks becomes weaker. In other words, this indicates that as Cu content decreases, the local structure around Cu$^{2+}$ cations becomes less uniform. The Cu species deposited on a substrate can be considered as Cu$^{2+}$ ions. Fig. 7 (b) shows the Cu PL spectrum with its deconvolution. Three derived sets of Gaussian curves estimate the fraction of Cu$^{2+}$ cations in tetrahedral and octahedral sites. The three Gaussian peaks appear at 410 nm, 439 nm, and 507 nm wavelengths, which correspond to the peak positions of Cu$_{\text{Th}}$ and Cu$_{\text{Oh}}$ at 413 nm and 515 nm, respectively. Thus, Cu emits luminescence at $\lambda_{\text{max}}$=325 nm when excited at 413 nm and 515 nm in the Cu sample. The results correctly show the expected neutral Cu lines at 324.75, 327.40, 510.55, 515.32, and 521.82 nm, and ionic Cu lines in the range of 406.71, and 657.71 nm [21].

3.4 | Electrical measurements

The electrical resistivity of the sputtered Cu thin films were measured using the 4-point probe method. Table 4 shows the dependence of the film conductivity on the deposition rate. The control of the HVPS power and sputtering temperature were not strictly done in this work. The minimum deposition rate used is approximately five times higher than in a commercial sputtering unit. The magnetic field at the target surface weakened progressively. This impacted the growth rate of the Cu target and substrate on repeated runs of the MSS.

**TABLE 4** The electrical measurements of Cu at 10 A

<table>
<thead>
<tr>
<th>Sputtering rate ($\mu$/min)</th>
<th>Voltage (V)</th>
<th>Power density (mW/m$^2$)</th>
<th>Resistivity ($\mu\Omega\text{m} \times 10^{-2}$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.8</td>
<td>160</td>
<td>2.5</td>
<td>1.5</td>
</tr>
<tr>
<td>4.7</td>
<td>250</td>
<td>6.67</td>
<td>1.7</td>
</tr>
</tbody>
</table>

4 | CONCLUSIONS

This study presents a low-cost DC magnetron sputtering system capable of producing thin conductive metal films on glass substrates. Our ultimate goal is to develop a stable system that consistently yields thin films with reproducible
optoelectronic properties, which are critical for applications in photovoltaics and photoconductive sensors. We have demonstrated the viability of our homegrown magnetron sputtering system (MSS) by successfully depositing metal films of Cu, Al, and Fe onto borosilicate glass substrates. These materials were chosen due to their availability. The resulting samples were analyzed using standard spectrometry techniques (x-ray, energy-dispersive, FTIR, SEM) and electrical measurements (current-voltage). Our results indicate that the low-cost MSS is a promising tool and that further optimization can lead to more uniform and conductive metallic thin films. Our findings underscore the importance of homegrown sputtering systems in advancing research in optoelectronics and demonstrate the potential for low-cost systems to enable the widespread use of these technologies.

**conflict of interest**

The authors declare that there are no conflicts of interest in this work.

**Supporting Information**

A supporting presentation for this work can be viewed at https://www.youtube.com/watch?v=QcUojAFZxU4

**references**


