STUDY OF THE EMISSIVITY OF COPPER OXIDE THIN FILMS BY CYLINDRICAL MAGNETRON SPUTTERING

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Abstract

High-energy electron beams operating in accelerator vacuum chambers can easily cause heat accumulation on the surfaces of in-vacuum components. When convective cooling and effective conductive heat dissipation are limited, the thermal radiation capability of material surfaces becomes an important heat dissipation mechanism. In this study, copper oxide (CuO) thin films were deposited on Oxygen-Free Copper (OFC) substrates to investigate their enhancement of surface thermal radiation characteristics. Experimental results show that the prepared CuO films exhibit dense columnar grain structures with a monoclinic CuO (002) preferred orientation. Emissivity measurements indicate that the average high-temperature emissivity of bare OFC substrates is approximately 0.1, while CuO-coated samples can achieve values up to 0.52, demonstrating improved infrared thermal radiation performance. The coating system setup, experimental procedures, and emissivity measurement methods will be further described in this paper.

INTRODUCTION

High-energy electron beams operating in accelerator vacuum chambers can easily cause heat accumulation on the surfaces of in-vacuum components due to beam-induced heating effects [1]. If the generated heat cannot be effectively dissipated, it may lead to degradation of mechanical properties and increased vacuum outgassing, thereby affecting beam stability. When in-vacuum components cannot effectively dissipate heat through water-cooling convection and the materials themselves possess limited thermal conductivity, thermal radiation from the material surface becomes an important heat dissipation mechanism. In recent years, high-emissivity coatings have gradually been applied in the thermal management of optoelectronic and high-heat-load components [2].

Among these materials, copper oxide (CuO), owing to its excellent high-temperature stability and infrared optical properties, has been widely utilized in thermal control coatings and infrared-related applications [3]. Therefore, in this study, copper oxide (CuO) thin films were deposited on Oxygen-Free Copper (OFC) substrates to investigate their film structures and high-temperature emissivity characteristics, with the aim of evaluating their feasibility as thermal dissipation coatings for in-vacuum components in accelerator vacuum chambers.

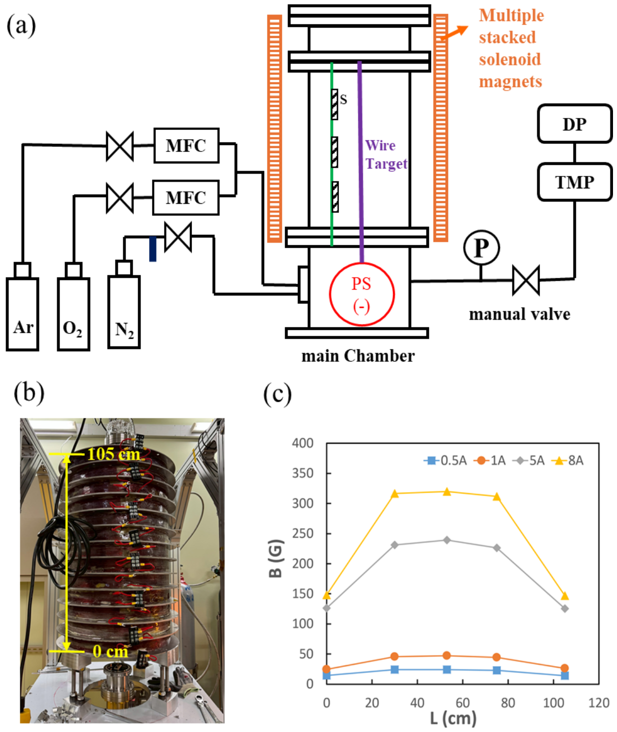

Figure 1: Cylindrical magnetron sputtering system. (a) Schematic diagram, (b) photograph of the sputtering system, and (c) magnetic field strength measurement.

COATING SYSTEM AND EXPERIMENT

The thin films in this study were prepared using cylindrical magnetron sputtering [4], and the sputtering system is shown in Fig. 1(a) and Fig. 1(b). A 99.99% purity bare copper twisted wire was used as the target material. The sputtering power was supplied by a DC power supply (PS), and 99.999% (5N) argon or oxygen gas was introduced as the reaction gas. The base pressure was 2×10^{-6} torr. Stacked solenoid magnets connected in series were used to generate the magnetic field. Under an operating current of 8 A, a relatively uniform magnetic field could be obtained in the center region (30–75 cm), with a magnetic field strength of approximately 320 Gauss, as shown in Fig. 1(c). Corning glass and Oxygen-Free Copper Substrates (OFC) were used depending on the requirements for electrical, thickness, structural, and emissivity measurements. The target-to-substrate distance was fixed at 30 mm.

Before deposition, the glass substrates were ultrasonically cleaned using acetone, isopropyl alcohol (IPA), and alcohol, followed by drying. The OFC substrates were ultrasonically cleaned using soap water, 5% Citranox solution, and deionized water, followed by dry air blowing and

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storage. The experimental parameters are summarized in Table 1. In Experiment (1), Corning glass substrates were used for the pure Ar process, and metallic Cu thin films were deposited at a deposition pressure of 1.5×10^{-2} torr. In Experiment (2) and Experiment (3), Corning glass and OFC substrates were used for the pure O_2 process, where CuO thin films were deposited at a deposition pressure of 3.2×10^{-2} torr.

Table 1: Thin Film Coating Parameter

Experiment	**Flow rate [sccm]**	**Power [W]**	**Time [hr]**
(1)	Ar: 20	10	2 hr
(2)	O_2: 20	20	25 hr
(3)	O_2: 20	30	27 hr

MATERIAL ANALYSIS

The thin film samples deposited on Corning glass in Experiment (1–3) were measured using a surface profilometer for thickness analysis. In Experiment (1), the metallic Cu thin film deposited at DC 10 W for 2 hr had a thickness of 102 nm. The sheet resistance (Rs) was measured using a Keithley 2450 source meter, and the resistivity (ρ) of the metallic Cu film was calculated by multiplying the sheet resistance by the film thickness, resulting in approximately 2.53×10^{-7} ohm-m. This value was one order higher than the theoretical value. Increasing the sputtering power could enlarge the columnar structure inside the film and reduce the number of grain boundaries, thereby improving the electrical conductivity [5]. To improve the electrical conductivity and reduce the deposition time, the sputtering power was increased to 20 W and 30 W for long-time deposition. The CuO thin films prepared in Experiment (2) and Experiment (3) had thicknesses of 1.2 µm and 2 µm, respectively. Phase structure analysis was performed using an ANalytical Empyrean X-ray diffraction system (XRD), and the results are shown in Fig. 2. According to the diffraction peak comparison, the Experiment (1) sample showed not only the Cu phase structure but also a weak Cu_2O (111) diffraction peak, which may be the reason for the slightly higher resistivity.

The analysis results of Experiment (2) and Experiment (3) samples showed CuO monoclinic structures, with the major diffraction peak corresponding to the (002) crystal plane. Cross-sectional observation and elemental analysis of the Experiment (3) sample were carried out using Plasma Focused Ion Beam (PFIB) SEM/EDS, and the results are shown in Fig. 3. The prepared CuO thin film exhibited a dense columnar grain structure. In the elemental analysis, the atomic percentages of Cu and O were 64.09 and 35.91, respectively. In addition, elemental mapping showed that Cu and O were uniformly distributed throughout the film, while the OFC substrate mainly consisted of Cu with a small amount of O.

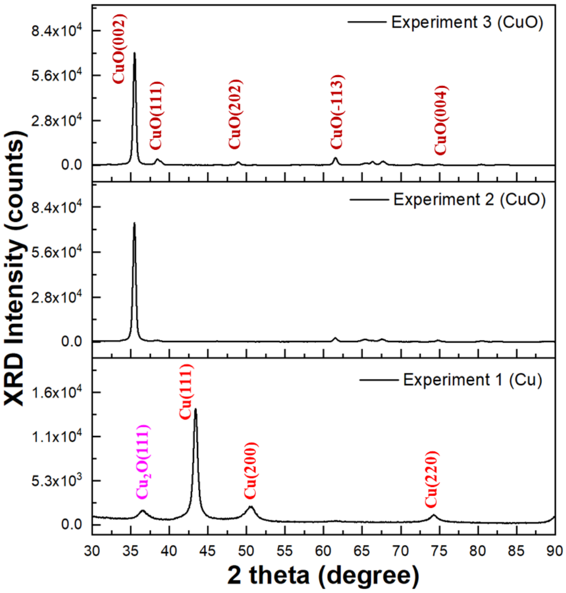

Figure 2: XRD phase structure analysis of thin films.

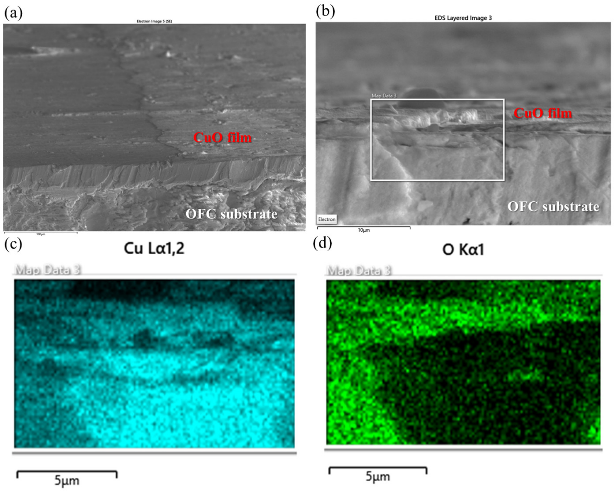

Figure 3: SEM and EDS analysis of the thin film. (a) Cross-sectional observation of the CuO thin film and OFC substrate. (b), (c), and (d) Elemental analysis and mapping observation.

Figure 4: Photograph of the 2 µm CuO thin film and OFC substrate samples.

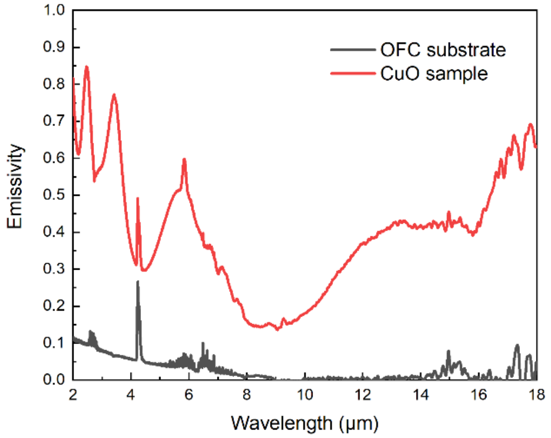

Figure 5: Emissivity measurement results of the OFC substrate and CuO sample at room temperature.

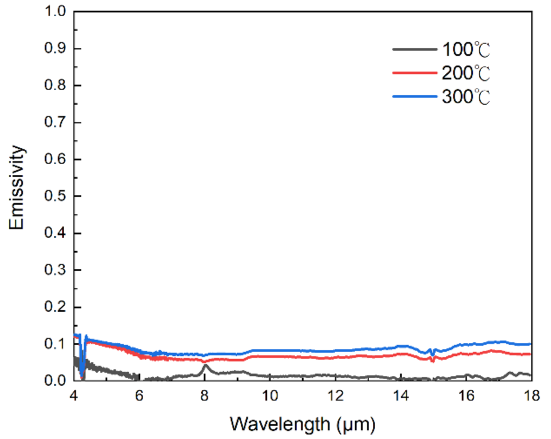

Figure 6: High-temperature emissivity measurement results of the OFC substrate.

MATERIAL EMISSIVITY ANALYSIS

The experimental samples are shown in Fig. 4. The Experiment (3) 2 μm CuO thin film sample and the OFC substrate were measured for emissivity at room temperature (~25°C), 100°C, 200°C, and 300°C. The room-temperature emissivity measurement was performed using a Fourier Transform Infrared Spectrometer (FTIR, Nicolet 6700, Thermo Fisher Scientific) combined with an integrating sphere system (IntegratIR™, PIKE Technologies, θ = 12°). The measurement results are shown in Fig. 5. The results show that the emissivity of the CuO-coated sample was higher than that of the OFC substrate. In the short-wave infrared (SWIR) region, the emissivity reached up to 0.85, and the average emissivity in the wavelength range of 4–18 μm was approximately 0.42, which was much higher than that of the OFC substrate (0.04). Oscillation behavior with peaks and valleys was observed at some wavelengths, which may be attributed to optical interference of the thin film. In addition, a peak at 4.3 μm was caused by environmental CO_2 interference.

For high-temperature emissivity measurement, the samples were placed inside a heating vacuum chamber, and the thermal radiation was transmitted through a ZnS window to the FTIR spectrometer for detection. The detected intensity was divided by the blackbody oven (reference) intensity to obtain the high-temperature emissivity value [6]. The experimental results are shown in Fig. 6 and Fig. 7. The results show that the emissivity increased with increasing heating temperature. For the OFC substrate, the emissivity increased by approximately 0.1 from 100°C to 200°C, while there was no significant difference between 200°C and 300°C. The average emissivity in the wavelength range of 4–18 μm was approximately 0.1. For the CuO sample, the high-temperature emissivity also increased with increasing temperature, and the overall emissivity was higher than that measured at room temperature. The average emissivity in the wavelength range of 4–18 μm was approximately 0.52.

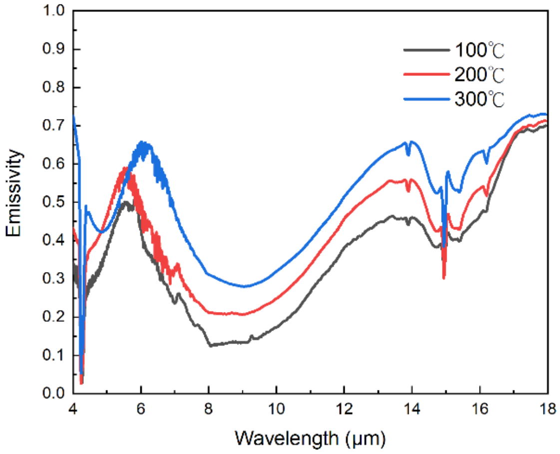

Figure 7: High-temperature emissivity measurement results of the CuO sample.

CONCLUSION

In this study, CuO thin films were successfully prepared using cylindrical magnetron sputtering. The prepared films exhibited a dense columnar grain structure and a strong CuO monoclinic (002) phase structure. In the emissivity measurements, the CuO thin films prepared in this work significantly modified the infrared emissivity behavior of the Oxygen-Free Copper substrate, and the emissivity at high temperature was enhanced by approximately five times. The results indicate that the CuO coating has potential for application as a thermal dissipation coating for in-vacuum components in accelerator vacuum chambers.

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