Fabrication and Properties of Erbium-Doped Gallo-Germanate Glass Thin Films via RF-Sputtering

Tomasz Ragiń,*[1] Jakub Markiewicz,[1] Krzysztof Markowski,[1] Karolina Sadowska,[1] Marcin Kochanowicz,[1] Piotr Miluski,[1] Jacek Żmojda,[1] Jan Dorosz,[1] Agata Baranowska,[2] Dominik Dorosz,[3] and Artur Rydosz[4]

[1]Faculty of Electrical Engineering, Bialystok University of Technology, Wiejska 45D, 15-351 Białystok, Poland
[2]Faculty of Mechanical Engineering, Bialystok University of Technology, Wiejska 45C, 15-351 Białystok, Poland
[3]Faculty of Materials Science and Ceramics, AGH University of Science and Technology,
30 Mickiewicza Av.,30-059 Kraków, Poland
[4]Faculty of Computer Science, Electronics and Telecommunications, AGH University of Science and Technology,
30 Mickiewicza Av., 30-059 Kraków, Poland

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This paper presents novel research on amorphous thin films doped with Er^{3+} ions deposited using the RF-sputtering method. The obtained layers' surface morphology, chemical composition, and optical properties were examined. Scanning electron microscopy analysis revealed a homogeneous layer structure with thicknesses ranging from 160–185 nm. Energy-dispersive spectroscopy confirmed the presence of elements such as Ge, Ba, and Ga. Optical properties were measured using photoluminescence spectroscopy, showing emission in the range of 1520–1550 nm, indicating potential for the integrated optics emission sources.

Thin-film deposition technologies play a pivotal role in developing photonic and optoelectronic applications by enabling precise control over material composition, structural properties, and optical characteristics. Among various deposition techniques, radio frequency sputtering has become an effective method for producing uniform, high-quality thin films with strong adhesion to substrates [1–3]. This technique is widely applied in the fabrication of optical coatings, semiconductor devices, and integrated photonic waveguides, as it enables the deposition of materials with well-defined physical and chemical properties. Compared to other deposition methods, RF-sputtering provides better stoichiometric control, scalability, and process repeatability, making it a preferred choice for fabricating thin films doped with rare-earth ions for photonic applications [4–5]. One of the most studied materials in photonics is erbium-doped glasses and fibers, which are particularly valuable for fiber-optic telecommunications, optical amplifiers, and integrated photonic circuits [6–7]. Incorporating Er^{3+} ions into materials enables efficient near-infrared emission of around 1.5 μm, a wavelength corresponding to the third optical communication window. However, the performance of rare-earth doped thin films strongly depends on process parameters, including sputtering power, working gas composition, and substrate temperature, which affect film morphology and optical properties [2, 8]. Recent advancements in RF-sputtering have improved control over thin-film characteristics, including precise thickness regulation, dopant distribution, and surface morphology. Studies have shown that plasma density, deposition rate, and ion bombardment energy significantly impact the crystallinity, refractive index, and optical transparency of the films. In particular, post-deposition thermal treatments have been demonstrated to modify the local environment of Er^{3+} ions, enhancing luminescence properties and reducing quenching effects [9]. Advanced characterization techniques such as atomic force microscopy, scanning electron microscopy, and energy-dispersive X-ray spectroscopy are used to thoroughly investigate the microstructural and optical properties of RF-sputtered films. These techniques provide essential information about surface roughness, grain structure, elemental composition, and phonon interactions, which directly affect the optical performance of the deposited layers. Furthermore, photoluminescence spectroscopy has been widely used to evaluate emission efficiency, energy transfer dynamics, and radiative lifetime, confirming the suitability of erbium-doped films for optical amplification and integrated photonic devices. Another significant advantage of thin films deposited by RF-sputtering is their compatibility with silicon-based photonic platforms, which allows them to be seamlessly integrated into chip-scale optical communication networks, sensors, and laser systems. The ability to produce high-quality erbium-doped germanium-gallium-barium oxide thin films on both silicon and glass substrates creates new opportunities for miniaturized optical components with enhanced signal processing capabilities.

The glass composition was developed as a target material for RF-sputtering and is based on germanium, barium, and gallium oxides, with erbium ions serving as the active dopant. Its molar composition is $59.5GeO_2$ – $40(BaO–Ga_2O_3)$ – $0.5Er_2O_3$, and the material is characterized by a density of 4.65 g/cm^3, a refractive

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[*] E-mail: tomasz.ragin@pb.edu.pl

index of 1.74 at 633 nm, and a glass transition temperature (Tg) of 622 °C, which makes it well-suited for thin-film deposition and optical applications in the near-infrared range. The glass was synthesized using a high-temperature melting process in a resistive furnace at 1500 °C, followed by controlled pouring into pre-prepared molds to shape the material. After cooling, the obtained glass was mechanically processed, including grinding and polishing, to achieve a smooth, high-quality surface suitable for thin-film deposition. The prepared glass was used to fabricate custom RF-sputtering targets attached to copper substrates using a two-component electrically and thermally conductive adhesive to ensure stable utilization during thin-film deposition. The RF-sputtering deposition was performed using an EVD22O-TT tabletop deposition system (Plasmionique Inc.). Before deposition, soda-lime glass substrates were plasma-cleaned at 50 W for 10 minutes to improve surface adhesion. The RF-sputtering process was optimized using a "soft-start" approach, where the magnetron power was gradually increased from the minimum to a maximum of 50 W. The deposition was conducted in high vacuum conditions at a pressure of 10 mTorr, with argon as the working gas at a flow rate of 20 sccm. The substrates were heated to 60 °C, positioned 60 mm from the magnetron, and rotated at 5 rpm to ensure uniform deposition. After one hour of deposition, followed by a soft shutdown of the magnetron, homogeneous amorphous layers of approx. 170 nm thickness were successfully formed on the glass substrates. The morphology of the films was examined using scanning electron microscopy with backscattered and secondary electron detectors, confirming the high uniformity of the surface. The chemical composition was investigated using energy-dispersive X-ray spectroscopy, verifying the consistency between the deposited layers and the target material. The optical properties were evaluated using an FLS1000 spectrofluorometer from Edinburgh Instruments.

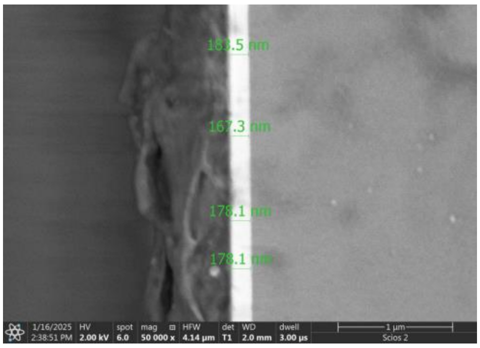

Fig. 1. X-SEM of RF-sputtered erbium-doped thin film.

The microstructural characterization of the erbium-doped thin films was performed using a scanning electron microscope equipped with a backscattered electron detector. This technique provides compositional contrast, enabling the identification of material uniformity, layer interfaces, and structural integrity. The obtained images reveal a well-defined film-substrate interface, confirming the successful deposition of the amorphous thin films onto the glass substrates (Fig. 1). The contrast observed in the images reflects differences in atomic number between the deposited layer and the substrate, with brighter areas corresponding to elements with higher atomic numbers, such as barium and germanium. The uniformity of the deposited layer suggests that the RF-sputtering process ensured homogeneous film coverage without significant variations in thickness. Furthermore, the measured thickness of the deposited films ranges from 167.3 nm to 183.5 nm, as observed in the high-magnification cross-sectional images. The minimal variation in thickness suggests that the substrate rotation at 5 rpm contributed to the uniformity of the deposited layers, helping to minimize shadowing effects during the sputtering process [10]. Additionally, no visible delamination or significant defects were observed at the film-substrate interface, confirming strong adhesion of the thin films.

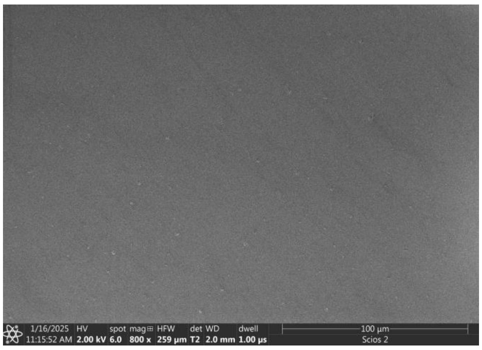

Fig. 2. Surface morphology of developed thin film.

The surface morphology of the produced thin films was analyzed using scanning electron microscopy equipped with a secondary electron detector, which provides high-resolution imaging of the topography and microstructural features of the deposited layer. The obtained image shows a smooth and homogeneous surface with no visible cracks, pinholes, or large-scale defects (Fig. 2). The uniform contrast suggests that the RF-sputtering process facilitated an even film distribution, ensuring a consistent morphology across the entire sample. At this magnification, small granular features can be observed, which may correspond to fine surface texture typical for amorphous thin films deposited under similar conditions. These surface characteristics can be further analyzed using atomic force microscopy to quantify the roughness parameters. Furthermore, no significant agglomeration or clustering of dopant is evident.

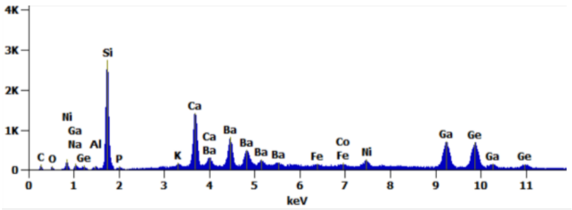

Fig. 3. Elemental composition analyzed by EDS.

Energy-dispersive X-ray spectroscopy was performed to assess the elemental composition of the deposited thin films. The obtained spectrum confirms the presence of key elements from both the sputtered layer and the underlying substrate (Fig. 3). The detected peaks correspond to germanium, gallium, and barium, which are consistent with the composition of the deposited film. However, strong signals from silicon (Si) and calcium (Ca), originating from the soda-lime glass substrate, are also observed. The presence of substrate-related elements in the spectrum is primarily due to the shallow interaction depth of the electron beam and the relatively low thickness (~170 nm) of the deposited layer. Since EDS has a penetration depth typically in the range of hundreds of nanometers to a few microns, contributions from the underlying substrate are inevitable when analyzing ultra-thin films. Despite these factors, the characteristic peaks of the deposited film elements (Ge, Ga, Ba) confirm successful thin-film formation. The detection of Ni and Co at low intensities is likely due to instrumental contamination or trace elements present in the chamber environment. It should be noted that erbium peaks are not visible in the spectrum, which is likely due to the low dopant concentration (0.5 mol% Er_2O_3) being below the detection limit of the EDS technique. This is a standard limitation when analyzing rare-earth elements present in small quantities, especially in amorphous thin films of submicron thickness.

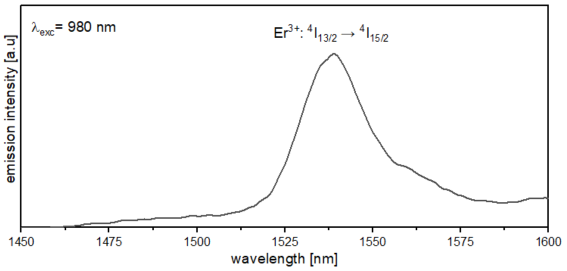

Fig. 4. IR emission spectrum of erbium-doped thin film.

The photoluminescence spectrum of the RF-sputtered erbium-doped thin film was recorded using an FLS1000 spectrofluorometer (Edinburgh Instruments) under 980 nm excitation. The obtained emission spectrum exhibits a well-defined peak at the range of 1530–1550 nm, corresponding to the $^4I_{13/2} \rightarrow {}^4I_{15/2}$ transition of Er^{3+} ions, which is characteristic of erbium-doped optical materials. The spectral profile presents a broadband emission, attributed to the amorphous nature of the host matrix. The absence of sharp peaks further confirms the lack of long-range crystallinity, consistent with the expected properties of amorphous thin films. The observed emission intensity indicates an efficient excitation of Er^{3+} ions, with no visible signs of strong concentration quenching within the tested doping level.

This study successfully demonstrated the fabrication and characterization of erbium-doped germanium-gallium-barium oxide thin film deposited using radio frequency sputtering. The results confirm that the developed thin films exhibit strong structural integrity, stable chemical composition, and promising optical properties. The morphological analysis performed using scanning electron microscopy revealed a homogeneous, uniform film surface with well-defined film-substrate interfaces. The absence of major structural defects, cracks, or delamination confirmed the strong adhesion of the thin films to the glass substrate, while the measured thickness (~170 nm) was consistent with the target deposition parameters. The energy-dispersive X-ray spectroscopy analysis verified the presence of key elements from the deposited film (Ge, Ga, Ba) while also detecting contributions from the underlying substrate (Si, Ca). The photoluminescence measurements, performed under 980 nm excitation, exhibited a strong 1.55 μm emission, corresponding to the $^4I_{13/2} \rightarrow {}^4I_{15/2}$ transition of Er^{3+} ions, which is essential for optical amplification applications. The emission profile showed broadband characteristics attributed to the amorphous nature of the host matrix, indicating the absence of long-range crystallinity. Obtained results confirm that RF-sputtered erbium-doped thin films are promising candidates for optical waveguides, amplifiers, and integrated photonic devices.

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