Spectral data of refractive index and extinction coefficient for thin films of titanium group metals used for fabrication of optical microstructures

Dmitrij A. Belousov a,*, Vadim S. Terent'ev a, Evgeny V. Spesivtsev b, Victor P. Korolkov a

a Institute of Automation and Electrometry of the SB RAS, Koptyuga Avenue, 1, Novosibirsk, 630090, Russian Federation

b Rzhanov Institute of Semiconductor Physics of SB RAS, Lavrentieva Avenue, 13, Novosibirsk, 630090, Russian Federation

ABSTRACT

In this data paper we share the information on refractive index and extinction coefficient of metallic films of the titanium group (Ti, Zr, Hf), measured by ellipsometry in the wavelength range of 300 – 1100 nm. The presented data can be used to indirectly measure the thickness of metal films when they are sputtered onto a substrate, using the measured data on transmission and reflection coefficients depending on the wavelength of the probe beam, as well as to calculate the energy characteristics of diffraction gratings, formed on the surface of these films, under rigorous electromagnetic theory. The data were used in the research article “Increasing the spatial resolution of direct laser writing of diffractive structures on thin films of titanium group metals” [1].

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1. Data

Here we show measured data of spectral dependence of refractive index (Fig. 1) and extinction coefficient (Fig. 2) for thin films of Hf, Ti, and Zr. The zirconium film has minimal values of extinction coefficient and refractive index in spectral range of 350–1100 nm.

2. Experimental design, materials, and methods

The data were obtained by the method of spectral ellipsometry using the device “ELLIPS-1991”, developed in Rzhanov Institute of Semiconductor Physics of SB RAS. As a light source, this device uses a 75 W xenon arc lamp, and the spectral measurement range is 300–1100 nm with a resolution of 2 nm. The probe beam diameter onto sample surface has a size of 1 mm. The measurements were made on Ti and Zr films with 1% transmission and Hf film with 0.13% transmission at 650 nm wavelength. The refractive indices n and the extinction coefficients k of the metal films were calculated from the measured ellipsometric spectrums using the software of this ellipsometer. The measurements were made on Ti and Zr films with 1% transmission and Hf film with 0.13% transmission at 650 nm wavelength. The probe beam diameter onto sample surface has a size of 1 mm. The measurements were made on Ti and Zr films with 1% transmission and Hf film with 0.13% transmission at 650 nm wavelength. The probe beam diameter onto sample surface has a size of 1 mm. The measurements were made on Ti and Zr films with 1% transmission and Hf film with 0.13% transmission at 650 nm wavelength. The probe beam diameter onto sample surface has a size of 1 mm. The measurements were made on Ti and Zr films with 1% transmission and Hf film with 0.13% transmission at 650 nm wavelength. The probe beam diameter onto sample surface has a size of 1 mm. The measurements were made on Ti and Zr films with 1% transmission and Hf film with 0.13% transmission at 650 nm wavelength. The probe beam diameter onto sample surface has a size of 1 mm. The measurements were made on Ti and Zr films with 1% transmission and Hf film with 0.13% transmission at 650 nm wavelength. The probe beam diameter onto sample surface has a size of 1 mm.

The cleaned substrate was placed in a vacuum chamber and heated for 30 minutes in a vacuum of 10⁻¹ mmHg at 200° C. After warming up, the chamber was pumped out to a pressure of 5 × 10⁻⁵ mmHg. The substrate was located at a distance of 25 mm from the surface of the magnetron target (Kurt j. Lesker TORUS TM3 - 3”) above its center. The power supply time mode of the magnetrons was low-frequency voltage pulses with a
duty cycle of 10, following with the frequency of 20–30 kHz. Using the frequency selection, the
average discharge current was selected \( I = 0.3 \, \text{A} \). The magnetron discharge was stabilized by
voltage \( U = 1 \, \text{kV} \) to maximize the energy of the sputtered particles. Argon was used as a buffer
gas. Its pressure during the sputtering was \( 10^{-3} \, \text{mmHg} \), to achieve free (hammerless with the
atoms of the buffer gas) deposition of sputtered particles on the surface of the substrate. The buffer
gas was supplied directly to the target of the magnetron. The characteristic sputtering rate was
about 4 \, \text{nm/s}.

Using the Ntegra by NT-MDT atomic-force microscope (AFM), images of the surfaces of the inves-
tigated metal films were obtained (Fig. 3a–f). The obtained AFM-images show that the most homo-
geneous is the hafnium film sample. The Zr film has a microcrystalline structure. Diagonal fringes on
the images of areas 5x5 \, \mu m are explained by surface imperfections of fused silica microscope slides
with 1-mm thickness used as substrates for the film sputtering.

![Fig. 1. Spectral dependence of refractive index for thin films of Hf, Ti, and Zr.](image1)

![Fig. 2. Spectral dependence of extinction coefficient for thin films of Hf, Ti, and Zr.](image2)
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Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.
Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.dib.2019.104903.

References

[1] V.P. Korolkov, A.G. Sedukhin, D.A. Belousov, R.V. Shimansky, V.N. Khomutov, S.L. Mikerin, E.V. Spesivtsev, R.I. Kutz, Increasing the spatial resolution of direct laser writing of diffractive structures on thin films of titanium group metals, Proc. SPIE 11030, Holography: Adv. Mod. Trends VI 11030 (2019), 110300A, https://doi.org/10.1117/12.2520978.

[2] E.V. Spesivtsev, S.V. Rykhlitskii, V.A. Shvets, Development of methods and instruments for optical ellipsometry at the institute of semiconductor physics of the Siberian branch of the Russian academy of sciences, Optoelectron., Instrum. Data Process. 47 (5) (2011) 419–425, https://doi.org/10.3103/S8756699011050219.