Si-W alloy thin films deposition by magnetron co-sputtering

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Abstract. Results of experimental studies on Si-W coatings fabrication by magnetron co-sputtering from two sources for electrodynamic structures in millimeter and submillimeter range of electromagnetic waves are presented. Control over sheet resistivity of the resulting coatings by varying the power of a magnetron with a tungsten target is demonstrated. Dependence of complex permittivity of Si-W coatings upon dielectric substrates on chemical composition in the frequency range of 50-70 GHz is studied.

1. Introduction
Metamaterials for microwave vacuum electronics devices of millimeter and submillimeter wavelength ranges attracted a significant interest recently [1-4]. Metamaterials are artificially created media built of specially formed micro- and nanoscale structures that attain unique electromagnetic properties which cannot be found in any substances of natural origin. The most striking distinguishing feature of metamaterials is a feasibility of negative values of permittivity and permeability (either simultaneously or independently). Metamaterials on dielectric substrates were proposed [5] for use in vacuum microelectronic devices such as sheet electron beam traveling wave tubes [6-9]. Authors highlight the increase of the interaction impedance (by more than 50%), as well as the decrease of circuit attenuation (by more than 20%) in the slow-wave system [5]. Metamaterials were also proposed to be utilized in electromagnetic wave absorbers to suppress spurious excitations [10]. High temperature tolerant materials are preferred for vacuum microelectronic devices, which promotes searching for new production technologies utilizing heat-resistant alloys. These technologies should allow to control over electrophysical properties (primarily, conductivity) of resulting structures, since those affect the properties of the metamaterial as a whole.

Tungsten and silicon alloys (Si-W alloys) seem to be suitable option for the aforementioned heat-resistant alloys of controlled conductivity [12]. In addition, Si-W alloys are of interest for the formation of negative electrodes of lithium-ion batteries due to high capacity and increased stability [11].

2. Materials and Methods
The thin films of Si-W alloys were deposited on quartz substrates by simultaneous DC magnetron sputtering using NexDep setup (Angstrom Engineering, Canada) with two independently controlled magnetrons equipped with Si and W targets. Circular 6 mm thick targets with 76 mm diameter of silicon (99.999%) and tungsten (99.5%) were used (Girmet, Russia). Base chamber pressure was kept below 2×10⁻⁵ Torr, while working pressure was controlled at level of 2×10⁻³ Torr. Argon was utilized as
working gas, and the substrate was heated to 200 °C during the deposition. Chemical composition of the coatings was varied with tungsten magnetron sputtering power in a range of 22 to 106 W with 12 W steps resulting in a series of 5 samples prepared upon 1 mm thick quartz slides. Silicon target was sputtered at a power of 500 W in all cases.

The sheet resistivity of coatings was measured by the 4-point probe method. PWS2326 (Tektronix, USA) stabilized power supply unit, a F195 microammeter (Russia), a 27II multimeter (Fluke, USA), and a laboratory setup of tungsten probes located in line at a distance of 2 mm from one another were utilized. Voltage from power supply was applied to the external probes. Current through external probes and the voltage at the internal ones were recorded. The sheet resistivity was calculated according to the conventional formula [13].

The dielectric parameters of the coatings were measured in a free space [14-16] using a PNA N5227A vector network analyzer (Keysight Technologies, USA) and two V-band horn antennas (Ducommun Technologies Inc, ARH-1520-01) precisely mounted on optical rails together with sample holders. The principal scheme of the experimental setup is shown in figure 1.

![Figure 1. Principal scheme of experimental setup for dielectric properties measurements in a free space.](image)

The samples were placed equidistantly between the antenna horns. On the waveguide connectors of horn antennas, a full two-port TRL (Thru-Reflect-Line) type calibration was performed using a mechanical calibration kit. Then, the GRL (Gated-Reflect-Line) type calibration was performed using the time-domain measurements. The GRL calibration method converts a 2-port calibration of measurements using waveguide into a full 2-port calibration in free space. Following the calibration procedures, the S-parameters of the samples were measured, from which the dielectric constants values were calculated using the Keysight Technologies N1500A Materials Measurement Suite software.

3. Results and discussion
The deposited films adhered strongly to the quartz slides. The surface of the films was homogeneous, without cracks and voids. Sheet resistivity measurements results are presented in figure 2. Measured
static sheet resistivity decreased nonlinearly (still monotonously) from 540 kΩ/sq to 220 Ω/sq with tungsten sputtering power due to the increase of tungsten content in a resulting film.

Figure 2. Static sheet resistivity of Si-W coatings vs. tungsten magnetron sputtering power.

The effect of coating composition on both real and imaginary parts of dielectric constant was found (figure 3). The imaginary part of the dielectric constant increases with tungsten content in the entire range of frequencies measured. The real part has a more complex dependence with an extremum, which can be found in the entire range of tungsten content studied. The real part of dielectric constant as a function of tungsten source power and frequency is presented in figure 4 for the purpose of better visualization. An increase of the imaginary part of the dielectric constant with the tungsten content in the coatings can be attributed to an increase of the static electrical conductivity, which is well illustrated by figure 5.a. The dielectric loss tangent does not change significantly with the frequency for all samples (figure 5,b). At frequencies of 50 and 60 GHz, the average difference in the tangent values is 3.8%. At 70 GHz, the deviation is already much larger – dielectric loss tangent is on average 23% lower than at 50 GHz. The summary of dielectric properties is presented in Table 1 for frequencies of 50, 60 and 70 GHz.

Figure 3. Measured real (a) and imaginary (b) parts of the complex dielectric constant of quartz slides coated with Si-W coatings of various compositions.
Figure 4. Measured real part of the complex dielectric constant of quartz slides coated with Si-W coatings as a function of tungsten source power and frequency.

Table 1. The summary of dielectric properties study of Si-W coatings with varied tungsten content.

| Frequency, GHz | $\varepsilon'$ | $\varepsilon''$ | tg $\delta$ |
|----------------|-----------------|-----------------|---------------|
| 50             | 3.665-4.201     | 0.043-5.046     | 0.012-1.271   |
| 60             | 3.534-3.975     | 0.028-4.461     | 0.008-1.262   |
| 70             | 3.170-3.823     | 0.000-3.753     | 0.000-1.184   |

Figure 5. Measured imaginary part of the complex dielectric constant (a) and loss tangent (b) of the quartz slides coated with Si-W coatings.
4. Conclusion
The control over resistivity of fabricated coatings by magnetron co-sputtering of silicon and tungsten was demonstrated. The variation of sheet resistivity by more than 2400 fold in fabricated Si-W coatings was achieved. In turn, varying the resistivity allows to control over the complex dielectric constant of the resulting coatings in millimeter wavelength range which can be useful for synthesis of novel thin-film metamaterials (metasurfaces). Magnetron co-sputtering is suitable for simple and reproducible synthesis of refractory Si-W alloys of various compositions allows avoiding the high temperature processes. This significantly expands the choice of possible substrates and determines the prospects of the proposed methodology for creating 2-D electrodynamic structures and metasurfaces in millimeter and sub-millimeter ranges.

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