Evaluation of magnetic properties of thin magneto-dielectric films deposited from beam plasma in medium vacuum

D Zolotukhin1, A Tyunkov and Y Yushkov
Tomsk State University of Control Systems and Radioelectronics, Tomsk, Russia

1E-mail: ZolotukhinDen@gmail.com

Abstract. By electron-beam evaporation of a solid state dielectrics (alumina ceramics) and a magnetic material (steel-3) in fore-vacuum, thin films of several μm, possessing both dielectric and magnetic properties, were deposited on a substrate. The work shows that the microstrip resonator method can be used to assess the presence of magnetic properties in films.

1. Introduction
Thin (with a thickness of several μm) magneto-dielectric coatings are actively used in high-frequency microelectronics due to their unique ability to ensure electrical insulation and exhibit magnetic properties with low eddy currents. In our previous work [1], we proposed a new method for producing such coatings. The method is based on three sequential stages: 1) electron-beam evaporation of a magnetic and then of a dielectric target; 2) ionization of metal vapor and the working gas; 3) deposition of coatings from the formed multi-component plasma on the substrate in medium vacuum [2]. The inductive method we used previously for estimation of magnetic properties of such coatings was uncertain due to its low sensitivity for diagnostics of thin films. This stimulated our further research on the methods aimed at detecting magnetic properties of magneto-dielectric coatings. In this study, we consider the method based on electromagnetic resonance in a microstrip resonator (MSR) [3, 4].

2. Experiment
Magneto-dielectric coatings were formed in the course of sequential electron-beam evaporation of steel-3 sample and alumina ceramic targets, followed by vapor deposition on the substrate at forevacuum pressures of the working gas (figure 1, a). After the vacuum chamber had been evacuated to the base pressure of 0.5 Pa, the working gas (oxygen, air, or helium) was injected into the chamber to an operating pressure of about 6 Pa, and a forevacuum plasma-cathode electron source [5] generated a continuous electron beam, focused to a diameter of about 5 mm, with an emission current of 100 mA. The evaporation of targets, first steel, then ceramics, was performed by a gradual increase in the electron beam energy from 4 to 12 keV. The overall coating deposition time varied from 20 to 40 minutes. In the deposited two-layer coating, the lower iron-containing layer is supposed to provide magnetic properties, while the outer layer based on alumina ceramic is responsible for dielectric properties of the coating, its thermal and chemical resistance, mechanical strength, wear and tear durability.
Figure 1. a) Experimental setup, b) Schematic diagram of irregular microstrip resonator.

Magnetic properties were analyzed by depositing coatings on rectangular alumina ceramic substrates, 0.94 mm thick, with dimensions of 7.8 mm × 4.9 mm, located at a distance of 5 cm from the evaporated target, and at an angle of about 30° to the beam axis. The coated substrates were placed inside an irregular microstrip resonator. The resonator (figure 1, b) consisted of two identical segments of microstrip lines (MSL) 1 placed on substrate 4 and connected by a segment of air strip line 2. The resonator connects to the transmission lines through communication capacities 3. A microwave ceramics with a high dielectric constant was used as a substrate of microstrip lines. Sample 5 is a parallelepiped with external dimensions matching the air gap formed by the segment of the asymmetric strip line. The MSL segments are located on metal plate 6. For certain frequencies, standing-waves with different half-wave numbers, commensurable with the resonator length (oscillation modes), are created [6]. The odd nodes in the middle of the resonator have antinodes of a high-frequency magnetic field and nodes of a high-frequency electric field. Conversely, the even modes in the middle of the resonator have antinodes of a high-frequency electric field and nodes of a high-frequency magnetic field. Additionally, in the corresponding antinodes of electric and magnetic fields, in the segment of the asymmetric microstrip line, the high-frequency fields are not only maximum in magnitude, but also uniform along the entire length of the measuring hole due to the large difference in the dielectric permeability of substrate 4 and the air gap. It is why the sample is placed in the region with the largest response of the electromagnetic field. Microwave ceramics with a high dielectric constant (T-150 with a dielectric constant of 150) is used as a substrate material in the segments of microstrip lines.

After placing the sample in the resonator cavity, the frequency dependencies of the reflection (S11) and transmission (S12) coefficients of the microwave signal were studied using a P2M-04 Micran network analyzer. The presence of frequency shifts was a sign of the presence of magnetic properties of the sample coatings.

3. Results and discussion
The results of MSR investigations are shown in figures 2 and 3 below.

As seen from figure 2 (a), the frequency dependencies of the coefficients S11 and S12 do not exhibit any noticeable frequency shift or a decrease in the resonance peak amplitudes in a wide frequency range (300-1000 MHz) with a coated plate placed in the resonator. When depositing the two-layer “Fe-Al2O3” coating in oxygen, no noticeable shift of the resonance peaks of S11 and S12
occur either ($\Delta f = 0$), even for the narrow search range in the neighborhood of the resonance peak of the first oscillation mode with the antinode of the magnetic field (figure 2, b). This may be indicative of an excessive oxidation of the iron-containing layer in the oxygen plasma to the oxides that do not possess magnetic properties. Nevertheless, when forming the “Fe-Al$_2$O$_3$” coating with atmospheric air, which contains less oxygen (~ 21 %), injected into the chamber, a shift of 0.7 MHz of the resonance peak of the first mode of the magnetic field becomes noticeable (figure 3, a).

![Figure 2](image1.png)

**Figure 2.** a) Frequency dependencies of the coefficients S11 and S12 for an empty resonator cavity (solid lines), and with a plate with the two-layer “Fe-Al$_2$O$_3$” coating deposited in oxygen placed in the cavity (dot-dashed lines); b) same dependences near the first resonance peak. Oxygen pressure is 6 Pa.

![Figure 3](image2.png)

**Figure 3.** a) An enhanced view of the frequency dependencies of the coefficients S11 and S12 near the resonance peaks of the first mode of the magnetic field in an empty resonator (solid lines), and with a plate with the “Fe-Al$_2$O$_3$” coating deposited in air (a, dot-dashed lines) or helium (b, dot-dashed lines). Gas pressure is 6 Pa.

It follows from figure 3 that with a coated sample placed in the resonator, the peak of the first mode of the magnetic field is slightly shifted to the left and decreased in the amplitude. This shift indicates that the deposited coating has magnetic properties, i.e. non-zero magnetic permeability. If the “Fe-
Al$_2$O$_3$ coating is deposited in helium, the resonance peak shift increases by $\Delta f = 1$ Mhz. Thus, one may conclude that the shift of the resonance peaks (i.e. the magnetic properties of the coating) is in the reverse proportion to the quantity of oxygen in the plasma-forming gas; and to improve the coating magnetic properties, the deposition should occur in an inert gas. However, the need to solve the complex electrodynamics problem to find numerical values of the magnetic permeability, along with the smallness of the observed shift, speaks for the insufficient sensitivity of the MSR method as applied to the measurement of the magneto-dielectric coatings.

4. Conclusion

Using the microstrip resonance method, we have demonstrated the presence of magnetic properties in the coatings deposited by electron-beam evaporation of an iron and alumina target in the forevacuum range of pressure. The magnitude of the shift of the resonance peaks depends on the kind of gas and is maximal for helium, while for coatings obtained in oxygen, this shift is zero. At the same time, the small value of the shift, together with the need to solve the complex electrodynamics problem, is a limiting factor in applying the microstrip resonance method for characterization of thin-film magneto-dielectric coatings. This stimulates the study on applicability of the other research methods, for example, based on ferromagnetic resonance (FMR).

Acknowledgements

The work was supported by the Russian Foundation for Basic Research (grant # 20-08-00370 A) and by a Grant from the President of the Russian Federation (No. MK-154.2020.8). Authors are grateful to Dr. V A Zhuravlev (Tomsk State University) for characterization of magnetic properties of the samples.

References

[1] Zolotukhin D B, Klimov A A, Oks E M, Tyunkov A V, Yushkov Y G and Zenin A A 2021 Vacuum 184 109944
[2] Smith Y and Vein H 1962 Ferrites (Moscow: Foreign Literature Publishing House)
[3] Suslyaev V I, Kochetkova T D, Zhuravlev V A and Sudakov S V 2003 Instrum. Exp. Tech. 46 672-6
[4] Belyaev B A, Zhuravlev V A, Kirichenko V I, Suslyaev V I and Tyurnev V 1994 Investigation of electromagnetic parameters of bicomplex media at microwave frequencies using an irregular microstrip resonator (Krasnoyarsk: Institute of Physics SB RAS)
[5] Burdovitsin V A and Oks E M 2008 Laser Part. Beams 26(4) 619-35
[6] Gurevich A G and Melkov G A 1994 Magnetic waves and oscillations (Moscow: Fizmatlit)