

Dear Editor and Reviewers!

Thank you for your time considering this manuscript. Here, is the point-by-point reply to all remarks and suggestions. All substantial changes are given within the new version of the manuscript and properly highlighted.

Sincerely yours,

On behalf of corresponding author

Arthur Dolmatov

Reviewer-1

The subject of the present study is very interesting and might be suitable for publication. The manuscript is submitted to the MDPI Sensors journal. Although the selected topic “Synthesis and Applications of Nanostructured Metals and Metal Oxides” is correct, the whole manuscript development of the subject might better focused on the suitability of the proposed materials for the applications as components of microwave devices, magnetic sensors or simply as a protecting coverings of biomedical components (see examples: Chiriac et al.; Microwire array for giant magnetoimpedance detection of magnetic particles for biosensor prototype. J. Magn. Magn. Mater. 2007, 311, 425–428 Safronov, et al. Polyacrylamide ferrogels with magnetite or strontium hexaferrite: Next step in the development of soft biomimetic matter for biosensor applications. Sensors 2018, 18, 257; Darton, N.J.; Ionescu, A.; Llandro, J. Magnetic Nanoparticles in Biosensing and Medicine; Cambridge University Press: Cambridge, UK, 2019; p. 279 and others).

Thanks for the important note. The articles you suggested helped to improve the introduction section and better reveal the topic of protective materials for magnetic sensors and biosensors, these articles are also mentioned in the text now.

The title should be re-thought as it does not explain the type of the material under consideration (microparticles) and quite popular but not accurate expression “electrodynamic performance” should be also clarified – what exactly was studied and understood?

Thank you for your comment. The title of the article has been changed - now it is easier to understand that the coated particles of carbonyl iron were studied in terms of thermal and electrodynamic properties in the microwave frequency range.

Yes, there is a tradition to call these particles in the initial state as “pure iron”, however it is most likely they are core/shell type particles covered by the iron oxide and therefore the whole story become more complex as the FeOx/SiO₂ shell may contribute to the electrodynamics in a different way.

This is an important note. Initially, we started from pure particles of carbonyl iron. Previously, using gamma-resonance spectroscopy, it was shown that no oxidation of iron occurs during the deposition of SiO₂ (<https://doi.org/10.1016/j.jallcom.2017.02.250>). At the same time, it is known that the intrinsic oxide layer of the surface area of iron particles does not exceed a few nanometers (even in the case of iron nanoparticles <https://doi.org/10.1021/ja900353f>). Therefore, the effect on the microwave can be ignored. Appropriate comments and explanations have been added to the article under Magneto static measurements section.

As the microwave studies are very important, at least brief description of the main point must be provided apart from ref. 38, which does not give a complete feeling of the device also.

Thank you. We enriched this section with more information on the procedure of microwave measurements.

The concept of iterations is given in very confusing way – the size distribution should be given for 800 particles of each type. The word “iterations” is confusing, the size cannot be changed by the repetition of measurements for which they typically use concept of iterations.

Thank you for your comment. Perhaps the word iteration is not the best, but it fully reflects the meaning of the operation - the repetition of deposition of SiO₂ on iron particles with air drying between operations. The size distribution histogram is not a repetition of the same measurements, these are the sizes of uncoated iron particles, particles with one two and three iterations of SiO₂ deposition

Full description of “iteration” is now given in lines 115-123: Since the maximum thickness of the shell is limited to 200 nm in one-stage Stöber synthesis, multi-iterative deposition technique was studied. One “iteration” here was full cycle of deposition including TEOS hydrolysis and drying in air. Each “iteration” increases thickness of the shell on the same particles already covered with SiO₂. To simplify, the first iteration transforms Fe -> “Fe@SiO₂”; the second iteration gives “(Fe@SiO₂)@SiO₂” and the third iteration results in “((Fe@SiO₂)@SiO₂)@SiO₂” product. In this study, pure carbonyl iron was studied and compared with powders, underwent 1, 2 and 3 iterations.

Synchronous thermal analysis results are very interesting but the reason for the peak displacement toward the higher temperatures requires additional explanation.

Thank you for your comment. Iron oxidization occurs with the complex formation of short-lifetime iron oxides magnetite (Fe₃O₄) and hematite (Fe₂O₃) [10.3390/s22083086, 10.1007/s10973-013-3456-x]. The heating flow of pure iron particles in presence of oxygen at linear heating demonstrates the exothermic reaction peak (see Fig. 10a). Protective coatings based on silica prevent direct contact between material and oxidizing environment, display improved thermal stability behavior that does not depends on the choice of the source precursor of Si [10.1016/j.ceramint.2018.11.013]. For instance, the onset temperature of lithium-rich layered oxides coated with SiO₂ utilizing a sol–gel process shifts from 217.84 for uncoated material to 245.78 C [10.1007/s10854-020-04481-6]. Also, nanocomposite AlCr(Si)N protective coatings with increasing Si-content prevent oxidization [10.1016/j.jmst.2021.04.065]. The addition of the coating on a substrate surface increases the onset of oxidation from 1100 up to 1260 C and protects from further oxidation. Herein, the presence of SiO₂-coating on the carbonyl iron prevents of diffusion of oxygen oxidation from the coating surface to the iron core during the early stages of oxidation [10.1007/s10854-020-04481-6, 10.1016/j.jmst.2021.04.065]. That displacement of the peak toward the higher temperatures (see Fig.10 b-d), enhance thermal stability and, also, hydrophobicity [10.1016/j.electacta.2016.10.099] and corrosion resistance [10.1016/j.jallcom.2017.02.250]. Additional clarifications are now included in the text

All figure must either show error bars or contain some kind of the experimental accuracy evaluation in the main text (see figure 8).

Thank you for your comment. Figure 8 now has error bars, all other figures contain measurement accuracy information in the main text.

Work is very interesting but it requires some polishing, style correction and better description to be useful for the other researches and multidisciplinary audience of Sensors journal.

Thank you for your comments, they made the article more understandable and accessible. The text was revised based on the recommendations of all reviewers.