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Title: „Development of technology for coating copper tracks and wires with graphene layers for applications in electronics"

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Summary

The aim of the industrial doctoral thesis is to build systems for graphene synthesis on copper surfaces and optimize synthesis conditions to efficiently cover the surfaces of copper wires and paths with graphene, thus protecting them from corrosion and electron migration-related destruction processes.

The research began with the development, construction, and optimization of two systems for graphene synthesis using chemical vapor deposition (CVD). The first system was designed to enable continuous coverage of copper wires of various diameters, while the second periodic system was aimed at enabling both low-temperature synthesis for covering copper paths and high-temperature synthesis for obtaining high-quality graphene. The construction and optimization of the systems constituted a coherent process aimed at obtaining high-quality coatings while maintaining the necessary guidelines for industrial implementation. In both systems, the research began with prototypical simple setups that only allowed for temperature control of the process and the composition and flow of commonly used reactive gases (methane, argon, and hydrogen).

Tests were performed on these prototypes to preliminarily identify the parameters necessary for producing a graphene coating, initially on copper wires and foils. The main process parameters were tested, including gas flow rate, gas mixture composition, process temperature, wire/foil heating time, synthesis duration, and preparation of copper wire/foil before the graphene synthesis process. Due to the observed limitations of the systems, both were modified by adding a gas receiver system enabling full control of reaction pressure and a radio frequency generator enabling plasma-assisted synthesis. Both systems were also modified by adding a input system for liquid hydrocarbons as carbon sources, allowing for synthesis using a non-flammable gas mixture of 95% Ar/5% H_2.

Comparative research was conducted to evaluate the influence of substrate preparation on the obtained graphene, including removal of post-production impurities from the copper surface (cleaning with organic solvents), reduction of roughness (electrochemical polishing, annealing), and removal of copper oxides (etching).

Two methods for evaluating the quality of the obtained graphene coatings were developed. The first is a rapid assessment of coverage directly on the copper surface, based on heating samples and observing the degree of oxidation under a light microscope, as well as SEM imaging of sample surfaces. The second is a complete characterization of quality requiring transfer onto a silicon substrate (Raman spectroscopy, AFM imaging) and/or TEM grid (TEM imaging) allowing for evaluation of layer thickness, defect density, and types of impurities.

To demonstrate the practical application of coatings obtained in the innovative continuous graphene synthesis system, comparative aging and high temperature resistance tests were performed. The tests were carried out on M1E grade commercial purity copper wires covered with CVD graphene, graphene oxide, and commercial coating, which were subjected to corrosion tests in a salt spray chamber. The degree of copper surface protection and changes in resistance at elevated temperature were evaluated. The research showed both the structural stability of graphene after anti-corrosion tests and similar or better resistance results at high temperatures compared to commercial coatings.