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Experimental setup for microwave plasma modification of dispersed materials at atmospheric pressure

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An experimental setup based on a waveguide-type microwave plasmatron has been developed to study plasma-assisted modification of dispersed materials (loose powders) in an electrodeless microwave discharge at atmospheric pressure in a gas flow. Devices (installation modules) have been developed for the formation of a vortex gas flow and controlled injection of dispersed particles into a microwave discharge. Preliminary experiments have been conducted on the treatment micron-sized SiO₂, MgO, and polytetrafluoroethylene (PTFE) particles in a microwave discharge in an argon flow. Particle analysis has been performed using scanning electron microscopy (SEM). In the case of oxide materials, it has been shown that microwave plasma treatment can lead to particle agglomeration and spheroidization. The experimental complex developed, combined with the use of various working gases, provides a versatile platform for plasma modification of dispersed particles across a wide range of sizes and material compositions.

Keywords: microwave plasmatron; atmospheric pressure microwave discharge; plasma modification; dispersed materials.

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1. Introduction

Modern technological advances in functional materials, nanotechnology, and powder metallurgy present researchers with the challenge of developing highly efficient and versatile methods for modifying the properties of dispersed materials. In this context, the use of plasma technologies is of particular interest, as they provide a wide range of thermodynamic and chemical conditions for influencing microparticles.

Plasma modification of loose powders enables changing their physicochemical properties, including the morphology, phase composition, crystalline lattice structure, as well

as the formation of functional coatings on particle surfaces. This opens the way to creating new materials with specified properties in high demand across various scientific and technical fields – from catalysts to composites. For example, plasma modification of powders reveals new possibilities of using it to develop new dispersed composite materials (DCM), where solid microparticles are used as a filler that imparts new properties to the matrix material. Modifying filler properties by means of plasma allows adjusting the chemical resistance, thermal stability, thermal and electrical conductivity, and other characteristics of DCM. One of the most promising directions in this area is the application of the microwave (MW) plasma characterized by

high energy density, non-equilibrium nature, and the ability to effectively control discharge parameters [1–10]. In addition to the physical impact (heating, evaporation, etc.), the microwave plasma provides an active chemical environment due to the presence of excited atoms and radicals, thus enabling changes in the surface structure as well as the deposition of various (oxide or nitride) coatings. It should be noted that despite the diversity of active particles generated in the plasma, their impact on materials is confined to a very thin surface layer, typically less than 1 μm thick. Thus, all effects of plasma material treatment are determined by a set of physicochemical processes initiated by the plasma's active particles in this thin surface layer. However, when treating such materials as micropowders, the modified layer can constitute a significant portion of the entire microparticle's volume.

At the current stage of development of this field, one of the key challenges remaining is to design the experimental setups that ensure reliable and reproducible mode for plasma treatment of particles with various sizes and chemical compositions. Electrodeless microwave discharges operating at the atmospheric pressure offer several advantages: simple implementation, stability, and absence of electrodes, which eliminates a source of erosion and contamination. They allow localizing the plasma formation region and act on dispersed materials in a targeted manner. Furthermore, the ability to use various gases (inert gases, nitrogen, air, etc.) allows for flexible adjustment of the plasma medium's composition depending on the required technological outcome. Nevertheless, the practical implementation of effective treatment of dispersed particles by gas-discharge plasma requires solving a number of technical challenges, including gas flow formation and controlled particle injection. To enhance modification efficiency, it is crucial to ensure uniform particle distribution within the discharge volume and to minimize aggregation or settling. A vortex gas flow combined with injection systems can serve as a solution to this task, enabling simultaneous particle transport and dispersion in the zone of maximum concentration of active plasma components.

This paper presents the experimental setup that implements an approach for plasma

modification of dispersed materials at the atmospheric pressure using a waveguide-type microwave plasmatron and devices for feeding the plasma-supporting gas mixed with the powder to be processed. Technical solutions are proposed for forming a stable vortex flow of the plasma-supporting gas and controlling particle dosing modes into the microwave discharge region to ensure effective plasma treatment. Particular attention is given to the versatility of the setup, enabling the treatment of a wide range of powder materials of diverse natures.

2. Microwave Plasmatron and Systems for Gas and Powder Delivery into the Microwave Discharge

The experimental setup was built based on the previously developed magnetron-based waveguide-type microwave plasmatron installed on the test bench for studying electrode-free atmospheric-pressure microwave discharge [11–14]. The microwave plasmatron consists of two units: the microwave generator and high-voltage power supply unit that permanently connected by a 2-m high-voltage cable bundle housed in a protective corrugated metallic conduit.

Technical specifications of the microwave plasmatron:

- power supply voltage of the magnetron – 4.45 kV,
- anode current – 0.4–0.9 A,
- filament voltage – 3.15 V,
- filament current – 11 A,
- output power adjustment – 1.5 to 2.5 kW,
- level 3,
- power supply unit cooling – air-cooled,
- microwave unit cooling – water-cooled,
- min. 0.9 l/min,
- transformer overheat protection – thermal relays on cores,
- power supply unit dimensions (L×W×H) – 500×220×400 mm,
- microwave unit dimensions (L×W×H) – 370×170×270 mm,
- power supply unit weight – max. 27 kg,
- microwave unit weight – max. 6 kg.

The experimental setup also includes:

- microwave circulator with the operational power up to 5 kW,
- matched waveguide water loads,

– wide range of waveguide transmission line components.

A device for injecting dispersed particles into the microwave discharge region was developed and manufactured for the purpose of plasma modification of loose powders in atmospheric-pressure microwave plasma in the gas flow. When introducing powders into the discharge, several characteristics of atmospheric-pressure microwave discharge shall be considered. To maintain discharge stability and isolate it from the discharge chamber walls, various schemes for “swirling” the gas jet are typically used. However, in this case, when solid particles are introduced into the gas flow, they are centrifugally thrown from the central region

towards the chamber wall. The effectiveness of plasma impact on the particle surfaces is low. Figure 1 shows a scheme of the gas flow former, which allows constructing a microwave plasmatron where the tangential component of the gas flow velocity can be arbitrarily small. This is achieved by either rotating working gas supply tubes with a curved outlet or by using straight tubes. Discharge stability in this case is ensured due to the velocity jump and flow vorticity behind the cone's edge, which acts as a bluff body (poorly streamlined). Several versions of gas flow generators enabling the injection of dispersed powders into the plasma and featuring glass and ceramic hollow, as well as Teflon and combined designs, were manufactured and tested.

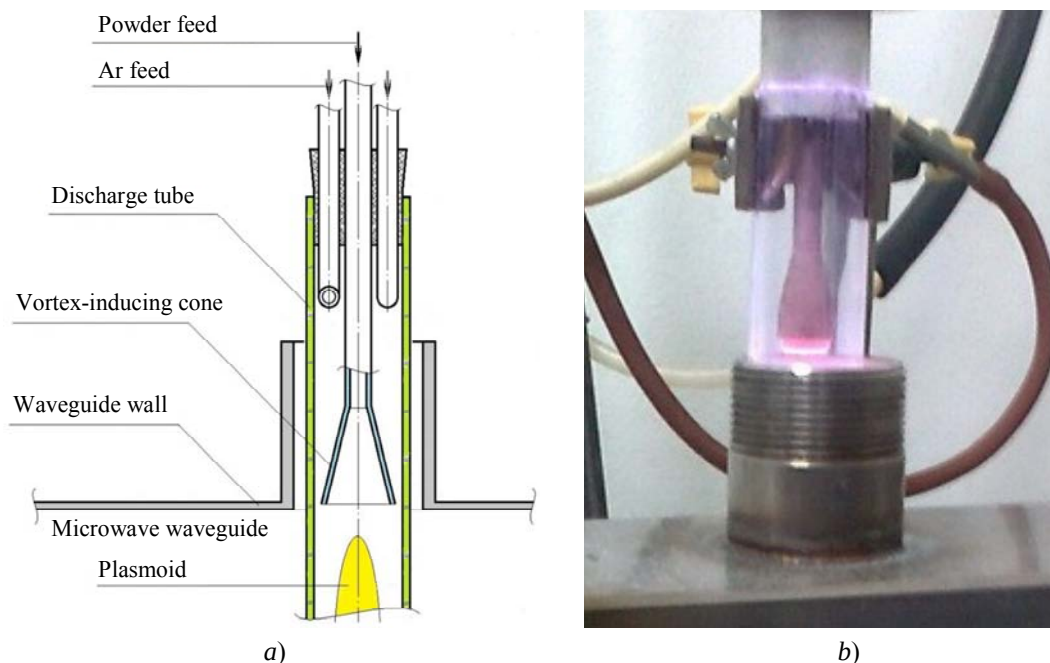


Fig. 1. Scheme of the gas flow former with a bluff body – a vortex-inducing cone (a) and its test prototype during a powder feeding experiment (b)

The next stage in developing the device for injecting micropowders into the plasma was to design a micro-dosing feeder. A pyramidal receiving storage hopper equipped with an adjustable pneumatic vibrator was designed and manufactured (Fig. 2a). The feeding single-flighted screw auger is driven by a motor, the speed of which can vary using a supply voltage frequency converter.

The undesirable escape of the feed gas through the screw auger was prevented by

maintaining a sufficient powder layer thickness in the receiving hopper.

Three polydisperse powders were processed: SiO_2 and MgO oxides, and polytetrafluoroethylene (PTFE) particles. High-purity argon (99.998 %) was used as the plasma-forming gas. The gas flow rate during processing was 25 l/min for the oxides and 15 l/min for PTFE. The microwave power delivered to the discharge was approximately 1 kW.

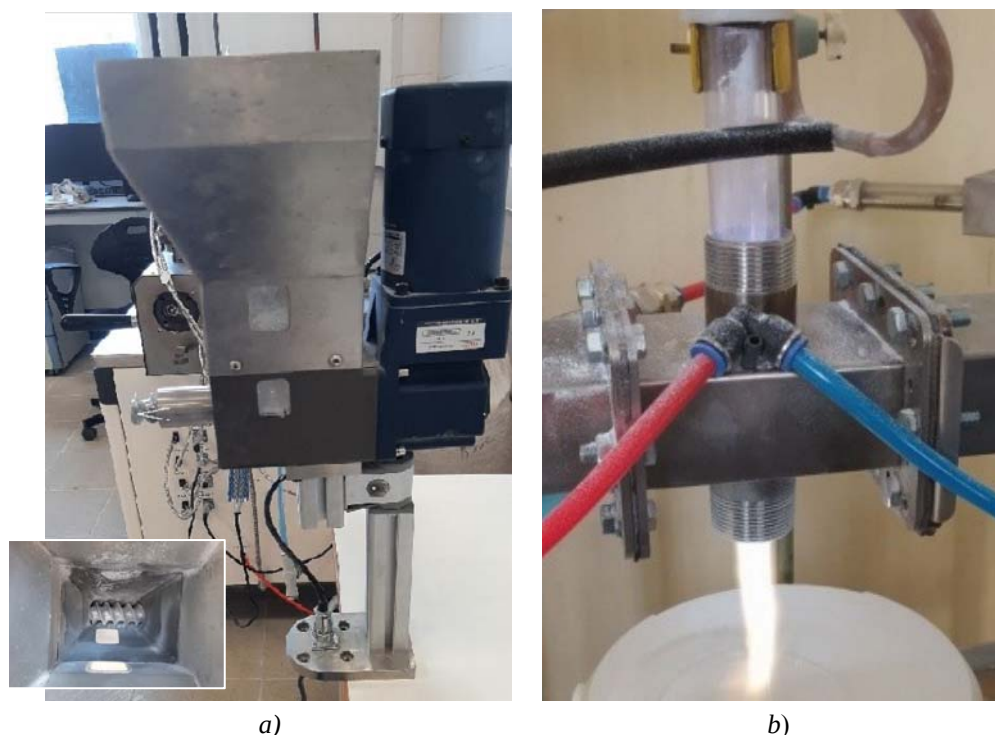


Fig. 2. Micro-dosing feeder for loose materials (a) and a photograph of the experiment on plasma treatment of particles in a microwave discharge within in the gas flow (b). The insert shows the screw auger at the hopper bottom (top view)

3. SEM Analysis of Treated Powder Microparticles

The morphology and elemental composition of the treated powders were analyzed using the scanning electron microscopy (SEM) method. Our research was performed using JEOL scanning electron microscope with the accelerating voltage of 0.5 to 20 kV. The electron-optical system of the microscope allows imaging with maximum magnification of $\times 100,000$. The spatial resolution with a 20 kV electron beam in high vacuum mode was 4 nm. The microscope is equipped with a highly efficient secondary electron detector and a compact, cost-effective energy-dispersion spectrometer enabling the spectral mapping and elemental analysis at individual points. Additionally, the microscope is fitted with a sputter coating device for pretreating of non-conductive samples prior to SEM scanning.

The results of electron microscopy for MgO powder particles at $\times 10,000$ magnification are shown in Fig. 3. Before being treated with the plasma most particles are seen to have a "flake-like" appearance of a highly asymmetric,

fragmented (scaly or needle-like) shape, less than $1\ \mu\text{m}$ thick and several microns long. After treatment, the powder contains larger, symmetric, rounded particles approximately $10\ \mu\text{m}$ and larger in size. Thus, it can be stated that the microwave plasma treatment results in agglomeration of MgO particles through sintering and subsequent spheroidization.

For the SiO_2 powder changed its particle size distribution after plasma treatment. The reference powder (before treatment) consisted of roughly spherical particles featuring a coarse fraction of $10\text{--}50\ \mu\text{m}$, "sprinkled" with smaller particles by an order of magnitude. As a result of plasma treatment, this smaller fraction became predominant. The particles were observed to acquire a more symmetric and smooth (rounded) shape, while the degree of "sprinkling" decreased. In this case, we can also speak of particle spheroidization in the microwave discharge plasma, which seems to be caused by melting of particle surfaces.

As for PTFE particles, the analysis conducted did not reveal significant changes in their morphology as a result of exposure to the microwave discharge under the applied treatment

modes. This is likely due to the high chemical inertness of PTFE, its low surface energy, and thermal stability, which hinder the effective interaction with plasma's active components at the given discharge parameters. This result

emphasizes the need to optimize the treatment modes (for instance, by increasing microwave power, extending the treatment time, or using reactive gases) to achieve noticeable plasma effects on PTFE.

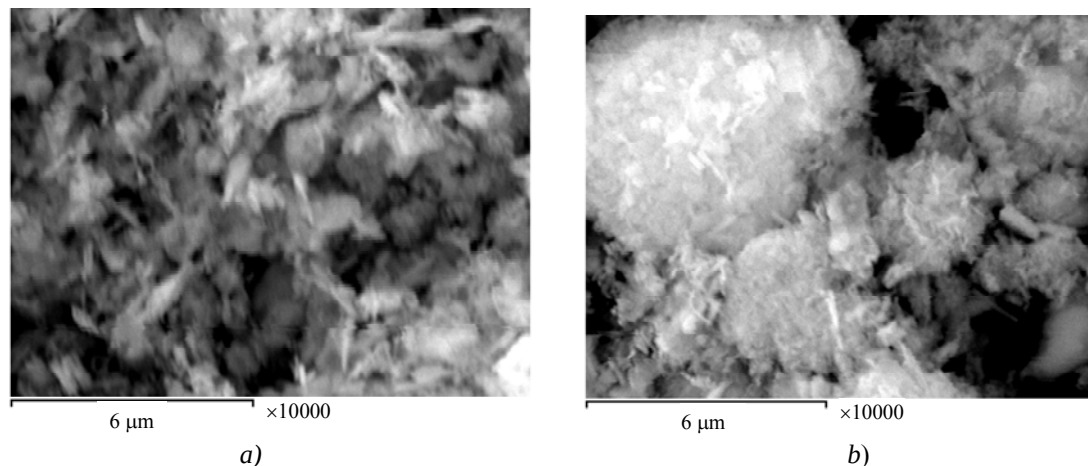


Fig. 3. SEM photos of MgO particles at $\times 10,000$ magnification before (a) and after (b) microwave plasma treatment

4. Conclusion

The experimental setup for studying the microwave plasma modification of dispersed materials, which is described in this paper, was designed on the basis of the existing test bench equipped with a previously developed waveguide-type microwave plasmatron that was supplemented with specially designed modules for feeding the working gas and dispersed particles into an electrode-free atmospheric-pressure microwave discharge – a vortex gas flow former, a micro-dosing feeder, a screw auger, and a mixer of powders with working gas.

Micron particles of three materials were treated with plasma: SiO₂ and MgO oxides, and

PTFE particles. Analysis of particle morphology by the scanning electron microscopy for the oxides revealed that plasma treatment results in particle agglomeration (which is more pronounced for MgO) and spheroidization thereof due to melting. Although the treatment of PTFE particles did not yield a substantial effect, selecting optimal processing parameters (microwave power, treatment duration, etc.) and plasma-supporting gases to achieve this effect is the goal of further research.

The designed experimental setup is highly multi-purpose and represents the in-demand tool for developing microwave plasma methods for obtaining new dispersed, materials with special functional properties, including composites.

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