Devices and Methods for Measuring of the Ambient Air Dust. Short Review

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Abstract

The main characteristics of airborne micro/nanoparticles, their impact on human health and air quality standards are presented. International standards classify microparticles by size (PM10, PM2.5, PM1, UFP), establish maximum allowable concentrations and control methods. Particular attention is paid to carbon- and virus-containing microparticles control. To monitor the air environment in enclosed spaces and in transport, the portable sensors of micro-, nanoparticles are required with the ability to classify them by size and electrophysical characteristics.

Detection of microparticles includes the sorting of particles entering the sensor by size and material type, subsequent actual detection of particles of the same kind, with subsequent classification by size, electrical and morphological characteristics. Separation of nano- and microparticles by size before detection improves the sensitivity and selectivity of the detector both in size and material. The virtual impactor and dielectrophoresis method are considered for integration in a Lab-on-Chip type sensor. Detection of microparticles is performed by separating the dispersed phase from the aerosol followed by the analysis, or directly in the air flow. The classification of detection methods according to speed and functionality is given. Among the methods allowing detection of micrometer and submicrometer size particles, the most suitable for miniaturization and serial production of Lab-on-Chip sensors are the multi-wavelength photoelectric, MEMS, and capacitor elements.

The microelectromechanics, microfluidics and microoptics technologies make it possible to create portable sensor systems of the Lab-on-Chip type to detect particulates matter of micrometer and submicrometer size. A micro-, nanoparticles detector prototype based on alumina technology using MEMS elements for a compact Lab-on-Chip type sensor is presented. The proposed design for multifunctional portable detector of airborne micro/nanoparticles is prospective for industry, transport, medicine, public and residential buildings applications.

Keywords: microparticles, detector, Lab-on-Chip, alumina technology, prototype.

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Приборы и методы измерений запылённости окружающей воздушной среды. Краткий обзор

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Представлены основные характеристики переносимых воздухом микро/наночастиц, их влияние на здоровье человека и нормативы качества воздушной среды. Международные стандарты классифицируют микрочастицы по размеру (PM10, PM2,5, PM1, UFP), определяют предельно допустимые концентрации и методики их контроля. Особое внимание уделяется контролю углерод- и вирус-содержащих микрочастиц. Для мониторинга воздушной среды в закрытых помещениях, в транспорте требуются портативные датчики микро-, наночастиц с возможностями их классификации по размеру и электрофизическим характеристикам.

Детектирование микрочастиц включает сортировку попадающих в детектор микро/наночастиц по размеру и типу материала и собственно детектирование однотипных частиц с последующей классификацией по размеру, электрофизическим и морфологическим характеристикам. Разделение нано- и микрочастиц по размеру перед детектированием повышает чувствительность и селективность детектора как по размерам, так и по материалу. Для интеграции в сенсоре *Lab-on-Chip* типа рассмотрены методы виртуального импактора и диэлектрофореза. Детектирование микрочастиц осуществляется с выделением дисперсной фазы из аэрозоля с последующим анализом либо непосредственно в воздушном потоке. Приведена классификация методов детектирования по быстродействию и функциональным возможностям. Среди методов детектирования частиц микронных и субмикронных размеров наиболее пригодны для миниатюризации и серийного изготовления Lab-on-Chip сенсоров мультиволновые фотоэлектрические, МЭМС, конденсаторные элементы.

Технологии микроэлектромеханики, микрофлюидики и микрооптики позволяют создавать портативные сенсорные системы типа *Lab-on-Chip* для детектирования твёрдых частиц микронного и субмикронного размера. Представлен прототип детектора микро-, наночастиц на основе алюмооксидной технологии с использованием МЭМС элементов для компактного сенсора *Lab-on-Chip* типа. Предлагаемая конструкция многофункционального портативного детектора микро/наночастиц воздушной (газовой) среды перспективна для применения в промышленности, транспорте, медицине, общественных и жилых помещениях.

Ключевые слова: микрочастицы, детектор, лаборатория на чипе, алюмооксидная технология, прототип.

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Introduction

The improvement of technologies and methods to control harmful emissions into the atmosphere has not yet led to a global reduction in the total mass of emissions of the substances, primarily aerosols – the air-dispersed systems of microparticles in the form of dust, smoke, fog. The most common hazards are industrial dust of various nature and natural sources – dust storms, volcanic eruptions, etc. Microparticles in air environment disrupt functioning of equipment, deteriorate products quality, cause occupational acute and chronic diseases of respiratory system, circulatory system, eyes, and skin [1]. When penetrating deep into the lungs, they can deliver toxins, bacteria and viruses into the body.

Airborne particulate matter (PM) varies greatly in chemical composition, morphology and size. A number of international rules and standards for PM control have been developed that define the maximum allowable concentration of solid PM and methods to control them. The International Organization for Standardization ISO distinguishes several fractions of PM by size – PM10, PM2.5, PM1, Ultrafine particles [2]. In particular, PM10 is defined as “solid particles passing through an inlet of a specified size with an efficiency of 50 % at an aero-dynamic diameter of 10 µm” [3]. Ultrafine particles smaller than 0.1 µm are referred to as PM0.1.

Permissible levels of airborne microparticles of the PM10, PM2.5 classes are standardized by the World Health Organization [4], European Union [5], US Environmental Protection Agency [6], Ministry of Environmental Protection of the PRC [7] and sanitary regulations of the Russian Federation[1]. Special attention is paid to airborne micro-, nanoparticles of carbon in the form of black carbon [8], brown carbon [9] and soot (carbon black), the aggregates of which do not break down in the lungs into smaller or primary particles of ~ 10 nm size [10].

It is also important to control specifically the airborne viruses and bacteria of 20–500 nm size [11, 12]. Pathogenic viruses are viable in air, forming complexes with other microparticles [13] and drifting for a long time at a low sedimentation rate – from 3.1×10^{-3} m·s^{-1} for particles 10 µm in size to 3.5×10^{-5} m·s^{-1} for ~ 1 µm size particles [14]. In particular, it has been established that SARS-CoV-2 viruses remain suspended in enclosed spaces for a long time [15].

Thus, the control of atmospheric air dust content is an urgent production, hygiene and engineering problem. In particular, for everyday monitoring of the air environment in enclosed spaces, in transport, where PM concentration is increased [16], the portable individual sensors of micro-, nanoparticles being able to classify them by size and material are required.

The task of creating microparticle detectors includes two aspects:

– sorting of micro/nanoparticles entering the detector by size and material,

– proper detection of particles of the same type and their classification by size, etc.

Technologies and methods of microelectromechanics, microfluidics, and microoptics make it possible to create portable Lab-on-Chip type sensor systems to detect PM dust particles. This is the subject of the present brief review. Particular attention is paid to the features of detecting of nanometer size PM0.1 and Ultrafine particles. The aim of the study was to develop a multifunctional portable detector of airborne micro/nanoparticles based on microelectromechanical systems (MEMS) and alumina technology for industry, transport, medicine, public and residential buildings.

Micro/nanoparticles detection

General classification

Gas analyzers are used to control environmental pollution with gas and vaporous harmful substances; dust meters are used to measure concentration of dispersed pollutants. To get reliable data on the air pollution level, it is necessary to possess the reliable analytical techniques. The effectiveness of a technique is evaluated by a number of indicators:

– selectivity and accuracy;

– reproducibility;

– sensitivity;

– detection limits;

– express analysis ability.

The techniques to measure dust content in air could be divided into the two groups:

[1] Sanitary Rules and Norms of the Russian Federation 1.2.3685-21. Hygienic Standards and Requirements to Ensure the Safety and (or) Harmlessness of Environmental Factors for Humans. Chief State Sanitary Doctor of the Russian Federation. Decree of January 28, 2021, No. 2.

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– with separating the dispersed phase: weight (gravimetric), counting (conimetric), radioisotope, photometric types;

– without separating the dispersed phase: optical, photoelectrical, acoustical, electrical ones.

A number of measurement techniques (see below) have been proposed to monitor compliance with the above standards. The measurement can be direct, as observation of effects arising from direct interaction with particles (for example, photons absorption/scattering), or indirect, as observation of secondary effects allowing conclusions to be drawn about the aerosol concentration (see Table for direct techniques). The collection techniques require the particles to be deposited, for example, on a filter for a sufficient period of time to measure them, while online real-time (or near-real) systems determine concentration of suspended PMs continuously.

The measuring instruments provide data on mass concentration, density, sizes, composition, or other particle properties, for example, their ability to absorb/scatter optical or other radiation. The mass concentration is more relevant to health safety monitoring, while optical absorption is most interesting for climate studies as a decisive factor in global warming. The particle size could be directly obtained from a signal, or using additional techniques, such as initial separation of particles in size at entrance, or their ionizing followed by separation with high electric fields, etc.

Table

Direct techniques to measure air/gasborne particulate matters

Technique	Output	Output delay	Time resolution
	Optical		
Aethalometry	black carbon mass	quasi-real-time	minutes
Deposit imaging	particle count, size	minutes	minutes
Direct imaging	particle count, size	real-time	minutes
Lidar	aerosol optical depth	real-time	minutes
Nephelometry	particle count, size	real-time	seconds
Optical particle counting/spectrometry	particle count, size	real-time	seconds
Photometry of soot particles	mass	real-time	seconds
Photo(opto)acoustic spectrometry	absorption	real-time	seconds
Spectropolarimetry	polarization degree	real-time	minutes
	Electrical		
Capacitance	particle count, size	real-time	seconds
Charge	particle count	real-time	seconds
Inductance	particle count, size	real-time	seconds
	Mechanical/Acoustical		
Film acoustic resonator	mass	real-time	minutes
Oscillatory microbalance	mass	real-time	seconds
Photo(opto)acoustic spectrometry	absorption	real-time	seconds
	Others		
Gravimetry	mass	days to weeks	hours
β-irradiation attenuation	mass	minutes	minutes

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The important factor affecting the particle size and overall concentration estimation is the air humidity. Microparticles act as condensation cores which leads to significant increase in effective size readings of up to ≈ 70 % relative humidity [17], for example, under optical measurements. On the other hand, this phenomenon is used to count the smallest-size nanoparticles [18]. Also, when collecting aerosol, the moisture accumulates in the filtering material affecting the measured mass [19], optical and electrical properties.

Consider the basic detection techniques of interest to create a compact Lab-on-Chip type device.

Gravimetry

This direct technique, despite its non-compactness and non-viability for Lab-on-Chip technology, should be mentioned as the established standard for PM measurements [20]. The technique is based on collecting particles by depositing them on a filter for a given time at a certain air flow rate, followed by their weighing and additional analysis. To collect a sufficient mass of microparticles, the sampler should be activated for a long time, so it shows average values and does not allow tracking short-term effects. To measure simultaneously PM10 and PM2.5, multiple samplers should be used with selective inputs for appropriate sizes.

β-irradiation attenuation

The β-irradiation attenuation measurement is based on absorption of beta radiation in solids to measure PM. However, the absorbed radiation is proportional only to the mass of the filtered substance and does not depend on its density, chemical composition, physical or optical properties. Typically, differential measurements are used, where the filter collects particles from the air flow, and the readings of two Geiger counters are compared, one of which is located before and one after the bleed air flow. The advantages of this technique include high accuracy, as well as shorter averaging time interval and delay compared to gravimetric measurements.

Acoustic techniques

In photoacoustic spectrometry [21], particles pass through a modulated laser beam. The illuminated particles heat up, releasing heat energy into the air. The heated air creates a sound wave whose frequency exactly matches the modulation frequency of the laser. The sound wave is recorded by a sensitive microphone, and its amplitude being proportional to the particles' absorptivity.

Noise is also used to estimate indirectly the amount of airborne microparticles, for instance, to monitor the black carbon and PM concentration through traffic noise [22].

Optical techniques

The main technique to detect PM1 – PM10 microparticles uses a simple light emitting diode (LED)/photodetector optical scheme (Figure 1). Variety of compact sensors uses broadband LED 1 or semiconductor laser irradiation. Photons, scattered by microparticles 2, are detected by a semiconductor photodiode 3 and registered as time-dependent patterns 4. Some techniques have been developed to improve the accuracy of measurements by optimizing the operating modes of the irradiation source, receiver, and data processing procedure. A number of companies on the market produce optical compact sensors.

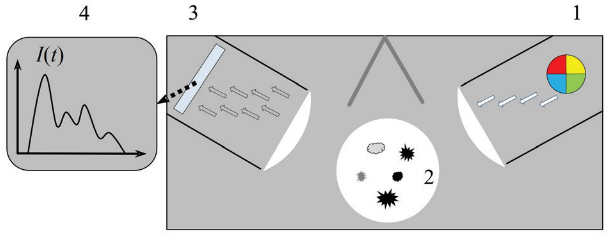

Figure 1 – Multi-wavelength optical sensing of airborne microparticles: 1 – light-emitting diodes; 2 – microparticles flow; 3 – photodetector; 4 – spectral/temporal data [23]

The minimum detected size of PMs cannot be significantly less than the optical beam wavelength. For laser illumination, the 0.4 to 3.3 μm radiation is used. Accordingly, the minimum radius of detected particles cannot be significantly less than ~ 100 nm. To improve the informativity of measurement, in particular, to estimate the size distribution of particles, the multi-wavelength laser systems are considered [24], as well as in the multiwave ethalometry technique [25]. The multi-wavelength detector [24] allows the analysis of microparticles by size. Optical feedback could stabilize the laser source measuring [26]. For instance, the optical particle counter of the Cubic Sensor and Instrument Co. [27] based on laser scattering technology could automatically identify and simultaneously output the number of particles in 6 channels including 0.3, 0.5, 1.0, 2.5, 5.0 and 10 μm size particles.

Microelectromechanical system

MEMS elements are used to detect and analyze the airborne micro-, nanoparticles [28, 29]. This approach makes it possible, in principle, to perform serial and batch production of devices using microelectronics technology to miniaturize and reduce the cost of sensors and systems. Most MEMS sensors are based on electric counting of particles. These sensors have no high sensitivity due to small volume and flow of detected air and corresponding small value of detected electric current.

The microbalances are the mechanical systems used to determine PM mass concentration. In an oscillating microbalance, the particles are deposited in a small conical glass tube. The tube' natural frequency is changed by additional mass of deposited particles, thus, the particles mass could be determined. Unfortunately, the microbalance monitoring is sensitive to mechanical noise and temperature fluctuations [30].

The MEMS resonator systems have been proposed, such as cavity film acoustic resonator [31] or surface acoustic wave resonator [32] which use the thermophoresis effects to deposit particles on a sensitive element and measure the resonant frequency change in real time.

Capacitive detectors

Capacitive detection is a rather new technique to detect microparticles where the capacitance change is measured (typically ~ 10 zF = 10^{-20} F) that occurs when a microparticle(s) enters the electric field between two electrodes, or when they deposited on the surface of a CMOS logic gate [33]. The capacitance change amplitude can be used to estimate particle(s) diameter [34]. This technique is under development and could enable detection of particles smaller than 1 μm [35].

Nanoparticles detection

Detection of microparticles of some tens of nanometer has its own features when the above methods are unsuitable in many cases. For the purposes of sensor miniaturization, the following solutions could be considered.

The known condensation type counter sensor use the technique of heterogeneous condensation of water vapors on nanoparticles [36]. The optical detection technique is used to count the formed micrometer-size water drops. The condensation type counter method is capable of detecting single nanoparticles [37] and possesses a high resolution in nanoparticle sizing when using a differential mobility analyzer nanoparticle selector [38]. The system includes a reservoir, a saturator, a condenser and a miniature optical drop detector.

In the electrical technique, the electrically charged nanoparticles in the device are counted by measuring the electric current in the circuit under their capturing (Figure 2).

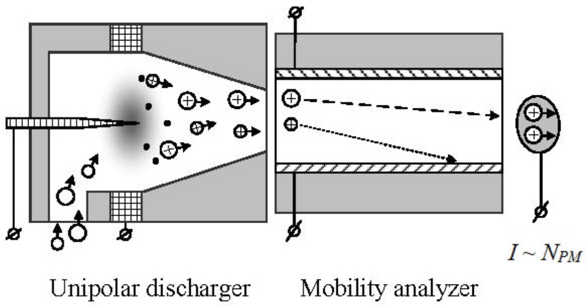

Figure 2 – Electrical discharge/microfluidic detection of nanoparticles [39]

Such devices are distinguished by high accuracy and selectivity. The devices require high-voltage unit to pre-charge the entering nanoparticles.

Sorting

Separation of nano- and microparticles prior to detection makes it possible to increase the sensitivity and selectivity of the detector, both in sizes and material thereof. Among the selection techniques, the two are most suitable for integration in a lab-on-chip device – virtual impactor and dielectrophoresis.

Virtual impactor

The aerial microjet chip classifies microparticles in size using an inertial separator – so-called *virtual impactor* (Figure 3).

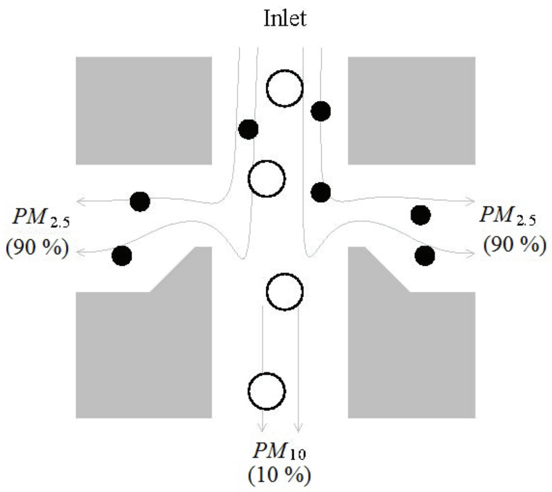

Figure 3 – Virtual impactor to select microparticles

Dielectrophoresis

The electric forces acting to micro-, nanoparticles under electrophoresis and dielectrophoresis (DEF) effects present the effective sensitive method to manipulate the particles when the device size lowers to Lab-on-Chip scale [40].

DEF is a selective electrokinetic effect including displacement of uncharged microparticles in inhomogeneous electric field due to interaction of their electrical dipole (induced or own) with electric field gradient. DEF forces action is defined by both the scale and electric properties of the particles and surrounding medium (gas or liquid). To create an inhomogeneous and non-stationary electric field in the DEF method, both the stationary and alternating electric fields are used, including additional constant bias. In particular, DEF method is successfully used to separate metallic and dielectric microparticles [41].

Lab-on-Chip MEMS detector of airborne nanoparticles

The above approaches are used in the MEMS detector under development where the micromembrane and capacitive measuring elements are used to gain higher sensitivity without pre-charging micro- , nanoparticles and with possibility to implement a complete measuring procedure in a single Lab-on-Chip device (Figure 4) [42].

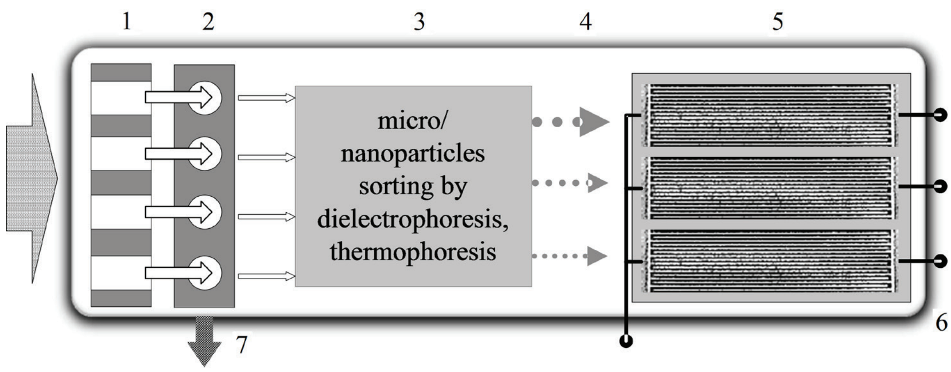

Figure 4 – MEMS nanoparticle detector: 1 – porous membrane; 2 – virtual impactor; 3 – separator; 4 – nanoparticles; 5 – capacitive detector; 6 – output signals; 7 – microparticles outlet (to optical detector)

The detector contains a thin alumina plate with periodic array of micro/nanometer size through holes 1 and virtual impactor 2 for size separation of the particles. The subsequent dielectrophoretic/thermophoretic sorting 3 with gradient electric/thermal fields allows to separate PMs in narrowed nanosize intervals providing their oriented movement 4, as well as to determine their amount using the anodic alumina based interdigitated electrode matrices 5 in every channel for subsequent capacitive measurements 6. The additional detection of microparticles 7 from the impactor 2 could be performed by optical counting technique.

Conclusion

The classification of airborne microparticles by main types and size is presented. The main techniques for separating and detecting airborne solid microparticles of micrometer and submicrometer size using optical, capacitive, microelectromechanical system and other sensing elements are considered. A prototype design of a micro-, nanoparticles detector based on alumina technology using microelectromechanical system elements for a compact Lab-on-Chip type sensor is presented. The prototype design contains mechanical filtering elements, dielectrophoretic/thermophoretic cells, and capacitive detecting elements with separate output signals to estimate the amount and size of nanoparticles, and to detect additionally the microparticles by an optical technique.

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