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*Special Topic:* ***Forensic Examinations – Terms and Techniques***

# Laser Method of Micro-Composite Materials Synthesis for New Sensor Platforms of an "Electronic Tongue"

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## Abstract

The information on the chemical composition of biological food mixes cannot be adequately received and submitted by traditional chemical analysis methods. The Electronic Tongue technology, composed of non-selective sensors, offers a modern language of submitting information on the composition of mixes. Sensor data are mathematically processed and visualized in the form of spatial clusters on the plane of the component coordinates chosen. At the same time the materials used for the sensors play an important role, and the bimetallic microstructures provide distinction of their electrocatalytic responses to the mix composition analyzed. Whereas each separate response is not informative, the cumulative signal forms a mathematical image of the system. The authors offer a new laser method of synthesis of sensor-active micro-composite materials, solid-state bimetallic solid solutions and two-phase ones. Their microstructure is investigated by the methods of the X-ray phase, and electronic and microscopic analyses. The measurements in electrochemical cells show the presence of electrocatalytic activity and selectivity of new materials in relation to the human blood components. The method used allows to "draw" using a laser beam on the dielectric substrate of the micro-composite structure of a necessary configuration. The authors offer, according to the principles of the functioning of the electronic tongue, a new family of an electrocatalytically active nanomaterial that will allow to create new types of sensor matrixes and to expand the electronic tongue scopes.

**Keywords:** Electronic tongue; Electronic nose; Criminalistic analysis; Laser deposition; Analysis of physiological liquids; Micro-composites; Electrocatalytically active materials

## Abstract

Информация о химическом составе биологических смесей не может быть адекватно получена и представлена традиционными методами химического анализа. Технология "Электронный язык", состоящая из неизбирательных датчиков, – это современный язык представления состава смесей. Данные сенсора обрабатываются математически и визуализируются в виде пространственных кластеров на плоскости выбранных компонентных координат. Материалы, используемые для датчиков, играют важную роль, а различия биметаллической микроструктуры обеспечивают разные электрокаталитические отклики на анализируемый состав смеси. Каждый отдельный ответ не информативен, совокупный сигнал формирует математический образ системы. Предложен лазерный метод синтеза сенсорно-активных микрокомпозиционных материалов, твердотельных биметаллических твердых растворов и двухфазных. Их микроструктура исследована методами рентгенофазового, электронного и микроскопического анализов. Измерения в электрохимических ячейках показывают наличие электрокаталитической активности и селективности новых материалов по отношению к компонентам крови человека. Используемый метод позволяет "нарисовать" лазерным лучом на диэлектрической подложке микрокомпозитную структуру. Авторы предлагают новое семейство электрокаталитически активного наноматериала, которое позволит создавать новые типы сенсорных матриц и расширять возможности электронного языка.

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## INTRODUCTION

The history of chemical sensors since the early 20th century has been a story of searching selective sensors (Vlasov, 1997).

Selectivity is a cornerstone of chemical express analysis. The need for such sensors is huge in the analysis of flowing or stationary systems in the field conditions where there is no opportunity to conduct a test preparation, particularly in applied criminalistics. However, a very limited number of chemical sensors have been really selective so far. And by the late 20th century there appeared in the world of science a clarity of the fact that the infinite variation of sensor-active materials will not lead to the problem solution. In the late 1990s the new concept of sensor application, consisting in the creation of devices in the form of massif non-selective sensors with special mathematical data processing on the basis of methods for image-recognition, was offered (artificial neural networks, chemometrics, Bayes networks, etc.). This concept imitating human organs of touch was implemented when developing such analytical tools as the "electronic nose" (Gardner & Bartlett, 1994) and the "electronic tongue" which were introduced in sensor analysis in 1995 as a result of joint Russian-Italian researches (Vlasov, et al., 2005; Legin, et al., 1999).

The sensation of taste, for example, consists of five main criteria: acids, salinity, bitterness, sweet and the taste of L-glutamates ("umami"). Tasting food or drinks, people sense the taste with flavoring receptors located on the tongue. Each flavoring receptor reacts to several chemicals at the same time, thus analyzing the foodstuff. That is, the flavoring receptors exhibits low or, so-called, semi-selectivity, but not "rigid" or high selectivity. High selectivity means an unambiguous compliance of a signal of a receptor to one certain chemical. Microscopic flavoring receptors consist of approximately 50-100 cells each enabling the person to distinguish even the taste of small drops or food fragments. Research of the mechanism for the reception of flavoring substances how that the information on components of food mixes that is perceived by flavoring receptors is then transferred to taste nerves as a result of releasing neurotransmitters and, at last, reaches the taste area of the brain which processes a cumulative signal from the group of semi-selective receptors and forms a cumulative idea of the foodstuff taste.

It is almost impossible to measure the taste of products containing several hundreds of flavoring substance types by methods of traditional chemical analysis. In order to describe taste sensations, the person uses such concepts as "sweet", "not really sweet", "bitterish", "sweet-sour", "a little salty", "very salty", etc. Apart from analysis complexity, there are some interactions between different tastes and between flavoring substances. For example, coffee bitterness is suppressed with sugar addition (Trivedi, 2012; Adler, et al., 2000).

How to digitize sensations of the human tongue, and to make them suitable for quantitative chemical analysis? That is, to translate them into the language of analytic chemistry in order to be able to apply them in automatic production control, medicine, criminalistics, etc.? The "electronic tongue" microsensors are, first of all, created for these purposes.

Molecular and cellular biology research of taste perception was transformed, at first, to sensor technologies for identification and quantitative assessment of the taste of food mixes. It was made to lower a subjective factor in the work of tasters and expert juries.

However, the aim of the new field of analytical chemistry is not limited to the problem of taste recognition. The electronic tongue, proposed in 1995, is already positioned as the sensor used for analyzing any solution consisting of a complex structure by using massif nonspecific chemical sensors and image-recognition (Ghasemi-Varnamkhasti, 2010; Winquist, 2008).

The first patent for the taste sensor was filed in 1989 (Hayashi, 1990). It represented the system of multichannel electrodes using a lipidic and polymeric membrane as a primary converter. In order to create electronic tongues, further development of this science led to the commencement of using both other materials of primary converters, and various methods of chemical analysis. Vinkvist and Lundstr reported on a voltammetric electronic tongue in 1997, and, then, developed a hybrid electronic tongue, having united measurement technologies of potentiometrics, voltammetry and electric conductivity (Winquist, 2008; Winquist, et al., 2000).

A.V. Legin and his colleagues applied solid-state ion-selective electrodes on the basis of chalcogenide glass to the electronic tongue for the first time (Vlasov et al., 1994), and presented some examples of applying the system for analysis of foodstuff and the quality of drink, including wine and mineral water (Verrelli, et al., 2007; Legin, et al., 2003). For this, they processed the signals of primary and secondary converters by mathematical methods of chemometric analysis.

The difference of the electronic tongue from traditional mono-component sensors is that in its basis there is a new method of mathematical processing of a cumulative signal of groups of sensors. Each sensor which is part of the electronic tongue package cannot possess high rates of sensitivity and selectivity for the components measured, but the cumulative signal of such sensor families bears some additional information on the structure of the system which cannot be received using each sensor separately (Vlasov, et al., 2000).

The result received can be easily visualized in a 2D or 3D graphic model in the coordinate system chosen. That is, in this field we observe symbiosis of a visual image, digital model and verbal description of the system structure. It greatly simplifies a number of analytical and expert objectives as a visual image often simplifies verbal descriptions. In addition, a set of digital and visual data enables to model the following stage of human nervous system work: processing of the information store by the brain which functions are executed by highest level software. And, then, to turn to sending commands to actuation mechanisms and devices.

The modern Electronic Tongue is a new type of sensor platforms which are actively developed by leading research laboratories of the world. Similar sensor platforms received their names by analogy with natural prototypes – the human tongue and the human nose (the electronic nose for gases).

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In some works (Winquist, 2008; Winquist, et al., 2000) six types of metal electrodes are used for receiving voltammetric and potential responses, and chemometric analysis of principal components (PCA) is used for analyzing the data obtained.

Chemometrics is a synthetic discipline located at the interface of chemistry and mathematics. It enables to realize difficult algorithms of data processing, in particular, the results of multiple-response and multiple-factor experiments. In turn, with the development of chemometrics, producers of measuring devices started to actively create the equipment capable to perform measurements in the form of multidimensional datasets, but not that of a single digital file or value (Rodionova, 2006).

In chemometrics there are some widely known methods:

ANN (Artificial Neural Network),

DASCO (Discrete Analysis with Short Covariation Matrix),

INLR (Implicit Nonlinear Latent Regression),

PARAFAC (Parallel Factorial Analysis),

PAT (Process Analytical Testing),

PCA (Principal Components Analysis),

PCR (Principal Components Regression),

PLS (Projection to Latent Structure), etc.

Chemometric methods are already implemented into expert and criminalistic practice. For example, in judicial and technical environmental assessment (Johnson, & Ehrlich, 2002) and judicial technical assessment of documents (Gorshkova, et al., 2020). In the latter work, the dates of the actual performance of document details are transformed to the drawings of spatial clusters on the plane of the chosen component coordinates. In the absence of such representations, experts would face a difficult problem of verbal description of the results obtained to unaccustomed listeners. Demonstrating a visual picture of chemometric clusters, for example, during a court session will help the judge understand the logic of the expert who drew a conclusion by the measurement results. So far, unfortunately, legal proceedings have not adapted such form of the argument yet and prefer to rely on verbal images and concepts, but, we believe, it is a question of the near future, since scientific and technical progress will force the parties to proceedings to master this "language".

The most frequently used method of mathematical processing of the electronic tongue signal is a chemometric method of principal components (PCA) (Malinowsky & Howery, 1980; Tauler & de Juan, 2006).

One of the main requirements to "electronic tongue" elements is their extreme smalless. Even dozens of various sensors assembled in a "package" are to allow analyzing the microquantities of substances. It is relevant in medicine and criminalistics, first of all, where it is often necessary to analyze trace quantities of some substances, e.g., blood, in the express mode (Petersen, 1996).

Another important requirement is good conductivity. Sensor materials, conducting badly, create additional problems when designing electronic schemes and devices and add a measurement error (Smikhovskaia, et al., 2019).

The best results are shown with hetero-phase micro-composite materials. Homo-phase metal deposits possess little lower indicators of sensitivity and selectivity than materials consisting of two and more phases. It is shown most distinctly for the manganese systems capable to work as an "electronic nose" (Fig. 1) (Baranauskaite, et al., 2019).

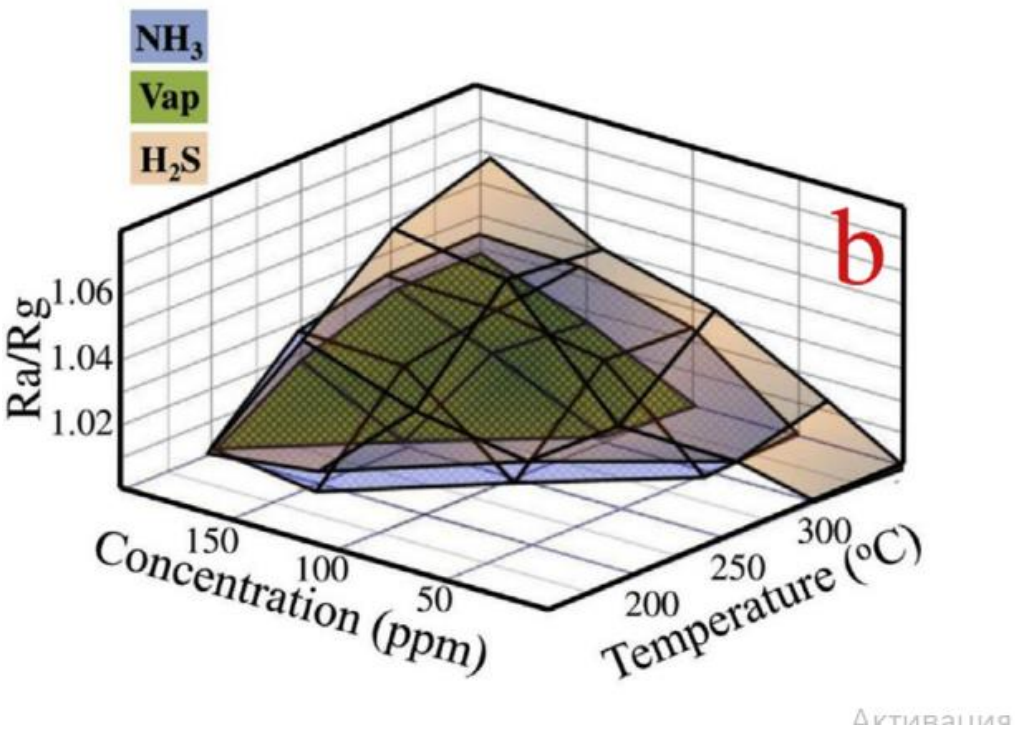


**Figure. 1.** The components of a cumulative signal of the molybdenum-oxidic sensor in gas mix of ammonia, hydrogen sulfide and vapors of acetic acid (Baranauskaite, et al., 2019).

Developing new methods to produce new micro- and nanocomposite materials is very important to solve the above-mentioned problem. It is known that composites have unique electric and electrocatalytic properties which makes them promising for creating various electrochemical sensors. There is a set of methods which can be applied to the production of such materials, however many of them have essential shortcomings related to using expensive reagents and difficult procedures of synthesis. The method of Laser-Induced Chemical Liquid-Phase Deposition (LCLD) has none of these shortcomings. Here, metal restoration comes from the localized volume of solution of electrolyte in the focus of a laser beam. It leads to formation of micro-dimensional metal tracks and clusters with an advanced surface on the surface of semiconductors and insulators of various types (Kochemirovskaya et al., 2020).

The article describes the authors' experience of creating a new type of voltammetric sensors that meet all requirements of platforms like the "electronic tongue" on the basis of metal one- and two-phase alloys of copper with various metals by method of laser synthesis. Their conductivity, sensitivity and selectivity to some components of human blood are tested, since it is supposed to be used for criminalistic purposes first of all.

## MATERIALS AND METHODS

The device for laser synthesis of numerous micro-dimensional, invisible to humans, sensor-sensitive bimetallic micro-tracks is presented in Fig. 2.

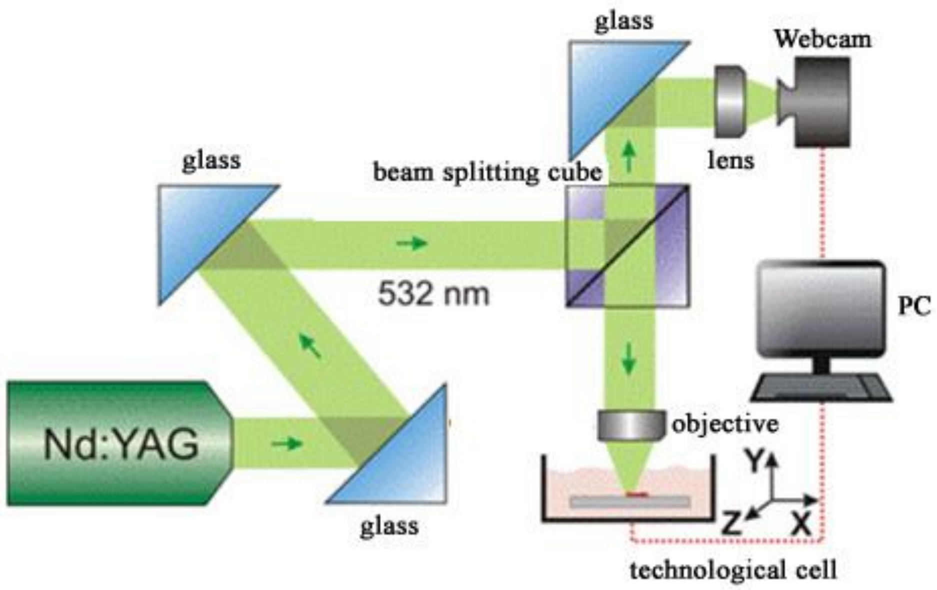


**Figure 2**. The technological scheme of laser synthesis of bimetallic micro-composites with high electrocatalytic activity

According to the scheme, the beam of the solid-state laser enters a system of collimating mirrors, then the beam-splitting cube, and further, via the transparent quartz glass of a cuvette and the solution on the dielectric substrate such that on the dielectric-solution border the beam is focused in a spot of 5-10 microns in diameter by means of the 8$^{th}$ fold lens on the interface. The beam reflected from the substrate passes, in reverse order, via the solution, quartz wall ditches, lens. Then, on the beam-splitting cube it splits in such a way that a part of the radiation reaches the webcam via the focusing system - where the webcam is used for observing the process of the deposition of metal in situ and on the monitor screen can be seen the process of focusing the initial beam on the substrate,. The dielectric and solution of electrolyte are placed on the motorized motion operated by the controller. In order to submit the operating commands with a personal computer, Standa Ltd software is used. The computer monitor is also reached by the information from the webcam by which the process is fixed in real time.

Laser radiation simultaneously activates the dielectric surface and accelerates the reaction of metal coating in the irradiated area. It occurs due to the increase of temperature in the local volume which is in focus of the laser beam in a consequence of two-photon processes (Kochemirovsky, 2014).

High intensity of focused radiation creates a local area of big temperature and concentration gradients, therefore the deposition has a localized nature: the width of the conducting tracks is in the interval of 50-250 microns. That is, it is possible to apply more than 150 sensor sensitive elements, 2 mm long each, on 1 sq.cm of the dielectric isolating material surface. Such geometrical parameters are optimum to create an electronic tongue.

The solutions used for laser deposition of micro-dimensional electrochemical sensors simultaneously contain the solutions of chlorides of copper and metals: cobalt, nickel, iron and zinc, xylitol reducer, organic sodium-potassium tartrate ligand and pH regulator. Compositions of solutions and concentration of components of basic solution are in Tab. 1.

**Table 1.** Composition of basic cupriferous solution for laser synthesis of bimetallic micro-composites

| Component | Concentration, M |
|---|---|
| Copper source: $CuCl_2$ | 0.01 |
| Ligand: sodium-potassium tartrate | 0.033 |
| Acidity regulator: NaOH | 0.1 |
| Reducer: xylitol | 0.075 |

The EDX analysis of the microstructure of samples was conducted by means of a scanning electronic microscope of Zeiss Supra 40VP with a field (Field Emission) cathode, column electronic GEMINI optics and completely oil-free vacuum system with an operating mode with low vacuum (VP).

The X-ray phase analysis of the synthesized structures was conducted with a Bruker "D2 Phaser" diffractometer with the copper anode and solid-state position sensitive detector of reflected LYNXEYE X-rays. The angle of diffraction was changed from 0 to 100 degrees.

The measurements of voltammetric characteristics of electrochemical cells were taken with a R-301 ELLINS potentiostat, in the background solution of 0.1 M $Na_2SO_4$. The length of the metal tracks synthesized by means of the laser-induced deposition method was 2 mm.

For the measurements, the cell working according to the three-electrode scheme was used.

As a contact with micro-tracks received on the surface of the glass-ceramic chip, the copper electrodes were put with the laser ablation method or conducting Kontaktol glue on the basis of silver.

## RESULTS

In traditional research of metal structures, synthesized by method of laser deposition, width-power dependence is used as a criterion for the determination of deposition speed. In this case, this parameter also receives a special importance because one of the aims of the research is to create the sensor platform of the electronic tongue and, consequently, we face the problem of maximum miniaturization of the sensor while simultaneously maintaining high conductivity.

There is copper, a basic metal for all sensor and active precipates studied and metals forming with copper various types of phase diagrams, cobalt (the diagram forming in a

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eutectic manner), nickel (unlimited solubility in a strong and liquid state), iron (the chart of eutectic type with wide concentration areas of solid solutions) and zinc. The similarity of the copper-iron and copper-cobalt charts consists in the eutectic nature of interaction, the distinction is in the presence of wide concentration areas of solid solutions of metals. The similarity of the copper-nickel and copper-zinc charts consists in available wide areas of chemical interaction in the form of solid solutions, but, simultaneously, in case of the copper-zinc chart there are chemical compounds of structures of CuZn, $Cu_5Zn_8$, $CuZn_3$ (Kochemirovskaya et al., 2020).

Fig. 3 represents graphic dependence of the deposited structures' width on the power of laser deposition.

It is apparent from all four graphs represented in Fig. 3 that the introduction of additives promotes miniaturization of the deposited structures, since, while increasing the power of laser radiation, the width grows much less when additives are introduced.

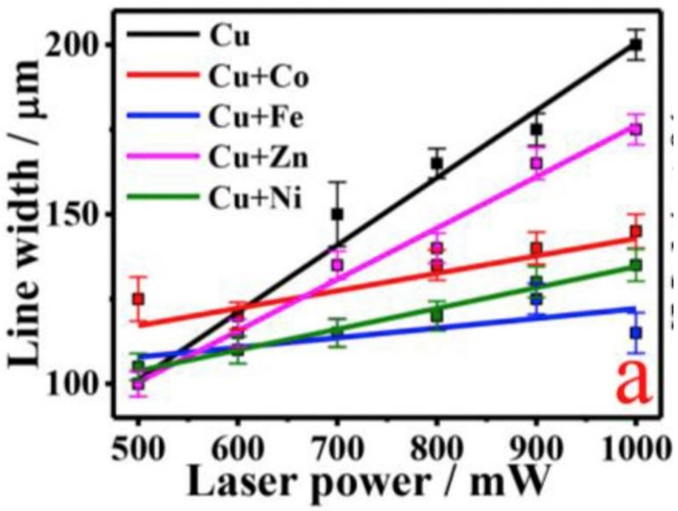


**Figure 3.** Linear approximations of dependences of width of the copper structures deposited from solutions with additives on the power of laser radiation of deposition (Smikhovskaia et al., 2018).

Miniaturization is easily achievable with the help of metals that interact with copper in a eutectic manner (particularly, cobalt and iron), i.e., potentially forming hetero-phase sensor sensitive materials of the electronic tongue. I.e., actually, the problem of reproducing the complex structure of the tongue receptors on the basis of available inorganic cheap materials is solved.

Nevertheless, as a result of metal additives, there is an insignificant increase in electrical resistance of precipitation received from solutions with the additives in comparison with purely copper structures.

The data of scanning electronic microscopy demonstrate that the precipitation consists of particles of 50-200 nm in size. The average size of the particles does not

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correspond to the concentration of additives or electrical resistance of 2-mm tracks (Fig. 4).

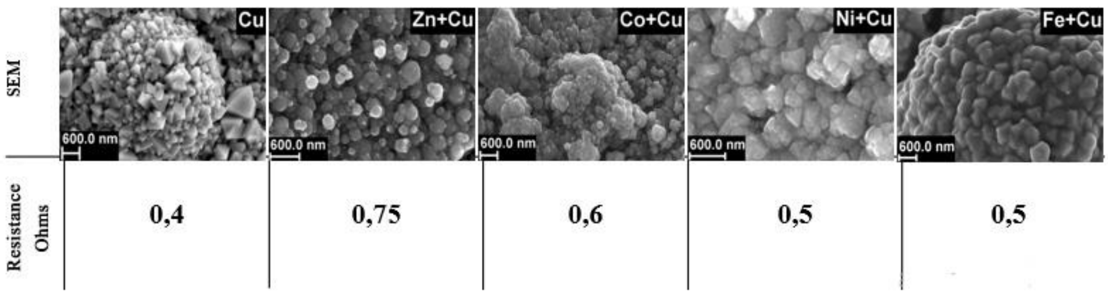


**Figure 4**. Laser induced coprecipitation of copper with nickel, cobalt, iron and zinc. Photos 1 and 4 from (Smikhovskaia, et al., 2018).

In Fig. 5, there are some X-ray diffractograms of the samples received from solutions by a laser method on the substrate of ST-50 glass-sitall. The individual peaks of zinc, iron, nickel are not revealed, which can be the result of codeposition of metals in the form of solid solutions. In case of the cobalt additive, there are individual peaks of the latter in the diffractograms.

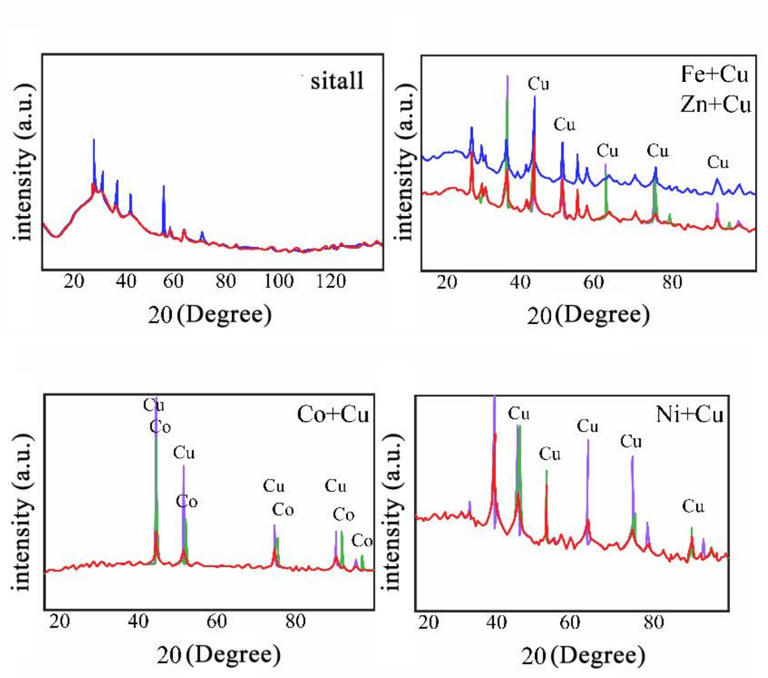


**Figure 5**. Diffractograms of sitall of precipitates obtained from solutions with additives $CoCl_2$, $FrCl_3$, $NiCl_2$, $ZnCl_2$ (figures 2 and 3 from Smikhovskaia, et al., 2018)

The differences of bimetallic track microstructures provide the distinction of their electrocatalytic responses to the composition of the mix analyzed. Each of these responses is separately a little informative for the quantitative chemical analysis of a complex mix. Together, they are capable to form a mathematical "image" of the system on the basis of a cumulative signal. The further algorithm of transforming this numerical matrix in visual and verbal images was briefly described in the previous sections.

The most promising types of contemporary electronic tongues are based on electrochemical principles (Legin, 2003; Verrelli, 2007; Vlasov et al, 1994; Winquist, 2008; Winquist et al., 2000). This is due to the simplicity of technical solutions when developing electronic schemes of reception and processing a signal of primary converters.

In order to test electrochemical properties of materials of hetero-phase microsensors, the structures received were investigated by the method of cyclic voltammetry. The quantitative characteristic of the proceeding reactions is the height and area of the peaks received, the qualitative one is the value of electrode reaction potential. In this case, the form of current curves is identical to purely copper electrodes and the electrodes received from solutions with the additives. The introduction of nickel, zinc and iron to the copper deposit leads to an insignificant shift of potentials to the positive area which data can be the consequence of forming solid solutions at the laser induced deposition of copper from the solutions containing mixes of metal chlorides which correlates with the X-ray phase analysis result.

The area of the graph, limited to a voltammetric curve, characterizes a charge which proceeds via an electrode at the set concentration of the analyte studied. This parameter is responsible for sensitivity of the electrode. In Fig. 6 there are some voltammograms the microtracks received by a laser method from the solutions containing 0.01 M of $NiCl_2$, $CoCl_2$, $FeCl_3$, $ZnCl_2$ in the background solution of electrolyte of 0.1 M of $Na_2SO_4$ (pH=9.8). The addition solution for laser deposition of CoCl solution with concentration of 0.01 M, leads to forming the track with much higher analytical response than that of the same quantity of $NiCl_2$, $FeCl_3$ and $ZnCl_2$, besides, with these structures, the maximum miniaturization of a deposit is obtained.

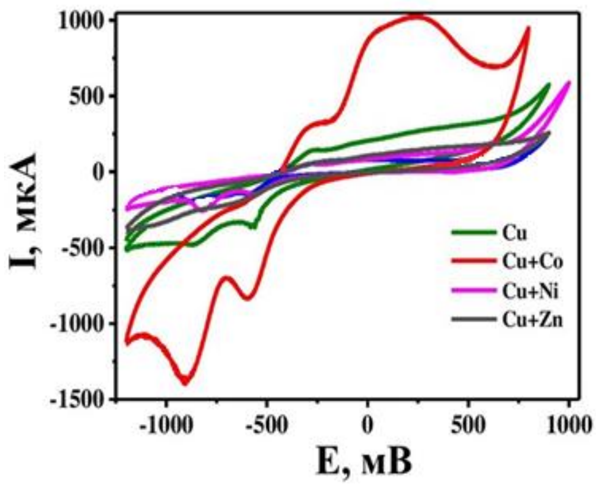


**Figure 6.** The voltammogram of the bimetallic microtracks received by the method of laser deposition (Smikhovskaia et al., 2018).

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It should be noted also that the control experiment on the laser deposition of a microtrack of pure cobalt and the research of its electric and sensor properties demonstrate that the structures synthesized are practically not conducting (the size of electrical resistance exceeds 10 MΩ), and it cannot be used as indicator electrodes in an electrochemical cell.

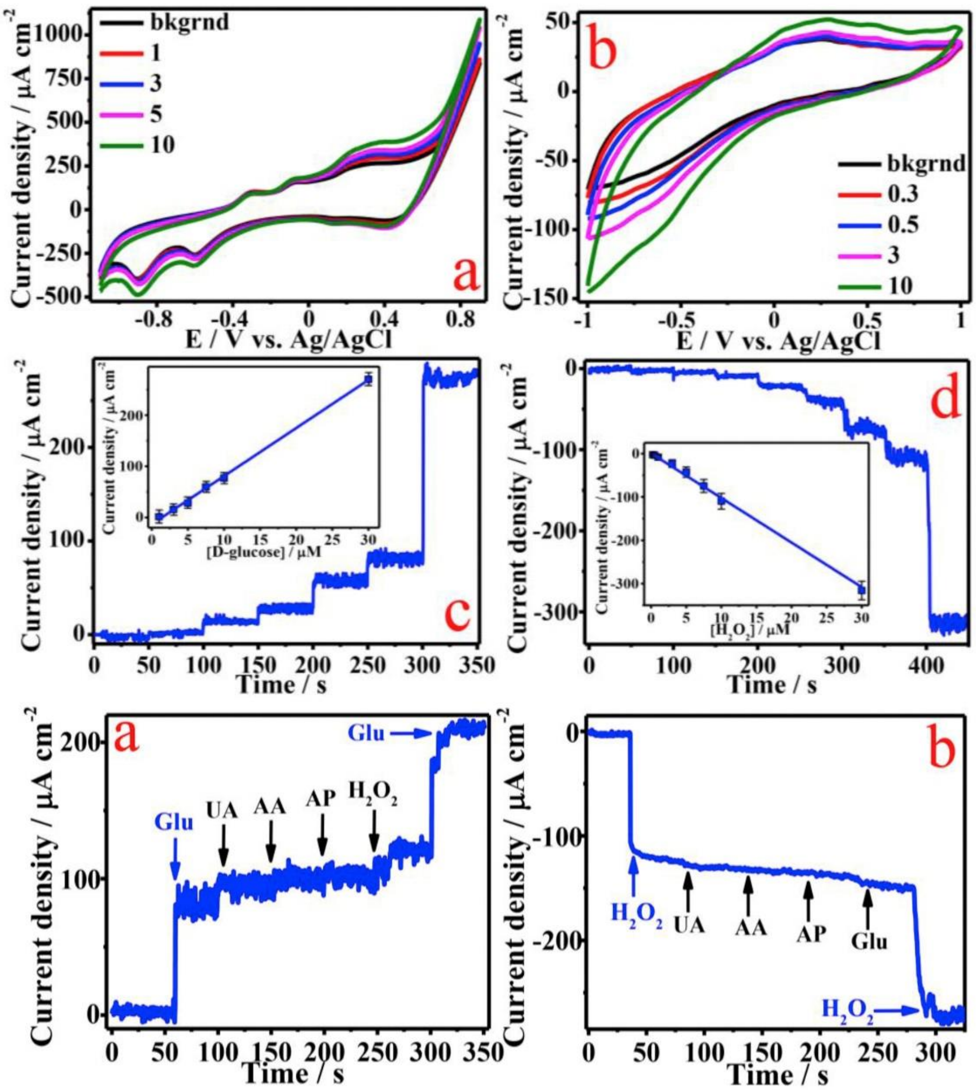


**Figure 7.** The cyclic voltammograms and chronoammograms of a copper-cobalt, microtrack in hydrogen peroxide solutions (a) and the glucose (b), and also in a complex mix containing, together with $H_2O_2$, glucose (GLU), uric acid (UA), ascorbic acid (AA), acetaminophenol (AP) in a background electrolyte (Smikhovskaia et al., 2018).

The results obtained can be explained, having assumed that the deposits - except for the one containing cobalt - synthesized by the method of the laser induced deposition, represents solid solutions on the basis of copper. In this case, the impurity metal atoms are built in a crystal lattice of a copper deposit, increasing its density at the simultaneous increase of electrical resistance.

The characteristic difference of the binary chart of "copper-cobalt" from those of Cu-Ni, Cu-Fe and Cu-Zn is that both metals interact at the eutectic type, that is, they are not inclined to interconnect and have, unlike the chart of Cu-Fe, extremely insignificant areas of solid solutions (Lyakishev et al., 1990).

Besides cobalt, silver possesses a similar phase diagram in the interaction with copper. Nevertheless, in case of laser induced deposition of silver and laser induced codeposition of copper with silver, chloride-anion cannot be used and replaced with nitrate-anion, because of the insolubility of silver chloride in water.

Some experiments on joint (Cu+Ag), and also consecutive deposition of silver on copper (Cu\Ag) were conducted. This manner of deposition excludes the possibility of metals interacting according to the crystallization fields of the phase diagram and, according to the physical and chemical characteristics, it is closer to a mechanical way of forming micro-composites. Copper-cobalt microtracks obtained by consecutive deposition have high sensor activity (Fig. 7). Copper-cobalt microtracks demonstrate the same results.

## DISCUSSION

The detailed study of interrelations of the processes unfolding near the focus of a laser beam on a dielectric surface in the solution for laser deposition confirm the systems approach adopted here - and so do the properties of bimetallic deposits. This systems approach aims for the improvement of the technology of laser synthesis of materials for electronic tongue micro-components in the form of polymetallic micro- and nanostructured systems having high conductivity, electrocatalytic activity and other useful properties.

This work describes the development of a promising sensor platform to create an "electronic tongue" on a microchip basis with a set of electrically and catalytically active microtracks forming - as a result of interaction with the liquid environment - a dataset which can be processed by methods of PCA image-recognition.

The main advantage of the technology offered is an opportunity to simply, not expensively, and ecologically sustainable, realize a "package" of differing sensors in the dimensions close to the human tongue’s taste clusters. Each separate sensor of such a "tongue" is not visible to the human eye - just as little as a group of taste receptors of a biological prototype. Certainly, there are already alternative technologies of creating micro-sensors of similar smallness but these technologies are connected with the production of expensive precision templates (Saei et al., 2013). The method offered here is unconventional. It consists in "drawing" by a laser beam on the dielectric substrate of a micro-composite structure of a necessary configuration. Particularly, for example, it is possible to reproduce, close to the nature, a 3D model of the human tongue possessing functions similar to the tongue. Also, in contrast to some alternative technologies, the production process does not involve the formation of toxic waste. The method offered is waste-free. The synthesis of microtracks is conducted until full expenditure of reactants, and the realized sensors can be repeatedly dissolved in acid and sent to a new production cycle.

The result obtained is well coordinated with the trends of materials science of the electronic tongue that were outlined above (Legin et al, 2003; Verrelli et al., 2007; Vlasov et al., 1994; Winquist, 2008; Winquist et al., 2000). In these works, special attention was paid to original nonspecific solid-state chemical sensors. In total, the matrix consists of 0 to 45 sensors. The matrix composition is being changed during the experiments depending on stability of the sensor and/or cross sensitivity. All the measurements are taken by electrochemical methods. Data processing is conducted with the use of analysis of PCA main components and various types of artificial neural networks: return distribution, self-organizing card (Kohonen network), etc. Various types of special computer software are used. However, the principles of the device design and performance are identical to all applications. It is a mainframe of conductive sensors on a chemically inert substrate or in an inert case.

According to these principles, the new family of electrocatalytically active nanomaterials enables the creation of new types of matrixes for an electronic tongue and to expand their scope.

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