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Gas discharge electronic device based on the porous zeolite

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Microdischarges in spatially confined geometries, such as micropores of zeolite materials, present a promising method for the generation and maintenance of stable discharges up to atmospheric pressure. A zeolite plate with push contacts is placed in a chamber filled with air at a controllable pressure, and the current–voltage characteristics of the zeolite plate are measured as a function of the air pressure. The paper presents relatively new type of discharge in confined volumes – a micropores microdischarge in zeolite and describes their basic physical properties and mechanisms. Gas discharge light emission inside the microporous zeolites develop from the surface if the amplitude of the applied voltage reaches given threshold. Discharge inside the micropores of zeolite framework is produced by DC power supply and produce relatively cold microplasmas with high level of non-equilibrium with no instabilities. It is found that the gas in zeolite pores ionizes and, accordingly, the number of electrons in the pores grows. It is shown that such a zeolite plate used as a cathode in a planar gas discharge cell considerably reduces the ignition voltage of the gas discharge. The basic characteristics of the microdischarges, addressing the effects of the applied voltage, discharge power, length and diameter gas discharge gap are discussed.

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1. Introduction

Atmospheric pressure non-equilibrium micro-plasmas are very attractive for their various applications, such as surface modifications, ozone generation, environmental and biomedical processing. The advantage of the microplasmas is that they produce high densities of chemically active species at relatively low energy consumption without any vacuum equipment requirements. The atmospheric microplasmas can be generated by various types of electric discharges, usually dielectric or ferroelectric barrier discharges. They are typical with microdischarges, which form and propagate along the surface of the material and may spread into a wide surface discharge. The microdischarges are filaments of high current density with radius of hundreds μm and duration limited to few ns controlled by charge build up at the dielectric surface. The accumulated charge reduces the electric field to such extent that the current is extinguished. The microdischarges can be generated not only at the surfaces, but also inside spatially confined volumes, e.g. microcavities or micropores of various conductive and insulating materials. A number of such configurations and devices have been developed in the past decade. Among them, probably the most attention has been given to a micro-hollow cathode discharge [1-3] and a capillary plasma electrode discharge (CPED) [3-6]. A configuration similar to CPED employing nano- or microporous dielectric barrier on the electrodes instead of regular capillaries been reported recently [7–9]. Nonthermal plasma is characterized by its thermally nonequilibrium state and its high potential in various chemical reactions even at atmospheric pressure and room temperature. Non-thermal plasma has been investigated for various applications such as pollution control [10], ozone synthesis [11], surface treatment [12] and so on. Recently, some emerging fields are gaining attention in plasma applications such as carbon nanotube synthesis [13], nano-scale material synthesis [14], aerodynamic actuator [15] and bio-medical applications [16]. Atmospheric pressure nonthermal plasma has also been investigated for a wide range of pollution control applications during the last two decades.

Deliberate modification of electrode surfaces with zeolites has evoked considerable interest. Much debate has occurred recently concerning the nature of the electroactive moieties present at zeolite-modified electrodes and electrodemodified zeolites. Although the controversy continues, it appears that true bulk electron transport through the electronically insulating zeolites is unlikely [17]. That the redox chemistry is an interfacial phenomenon is possibly disappointing in terms of harnessing bulk zeolite shape selectivity in electrosynthetic or electrocatalytic strategies. Certainly, if the locus of the electron transfer was the intrazeolite surface, then the prospects for product and transition state shape selectivity would be good. The attractive zeolite characteristics that are liable to affect the electron transfer reactions at an electrode–solution interphase are (a) the size and shape selectivity due to the rigid structure made of pores and channels of molecular dimensions; (b) the cation-exchange capacity arising from the charge compensation of the negatively charged aluminosilicate lattice by mobile extraframework cations; and (c) the catalytic properties of both intrinsic and extrinsic sites of the micro-porous materials. This has led to the design, preparation, and use of various zeolite-modified electrodes. The zeolite frameworks are formed by chains of silicon and aluminum anionites. The framework has a negative charge because of its structure and this charge is compensated by water molecules and cations of alkali and alkali-earth metals weakly connected with it. Water can be removed by heating or evacuation of the zeolite, which has no effect on a rigid framework, its structure is virtually unchanged. The zeolite pores have the right forms. Connecting between each other through “windows”, they form the perforated channel chain. That’s why the zeolites can be considered as the object on which besides well-known phenomena (adsorption, ion-exchange phenomena), investigate the electron porous emission, electron multiplication and gas discharge in pores, dielectric and electric properties at pore saturation by different gases and liquids.

The high emissive characteristics of nanotubes and pores provide the basis for a new class of electron emitters with extremely low supply voltage and power consumption [18,19]. Investigations into the emissive properties of different nanomaterials indicate that they are promising as field emitters. The unique emissive characteristics of carbon nano-tubes (CNTs) render them effective electrode coatings in gas discharge devices. Field-emission-cathode gas-discharge luminescent lamps are widely used for the background illumination of liquid crystal screens.

In this work, we report for the first time to our knowledge, direct observations of a steady gas discharge in through nanopores of natural zeolite. The current–voltage characteristics of the zeolite plate were taken at different residual pressures and the gas discharge light emission (GDLE) from the pores was simultaneously detected. It is found that, with such a zeolite plate used as a cathode in a planar gas-discharge cell, the ignition voltage of the discharge uniformly distributed over the electrode surface considerably drops.

2. Experimental

Natural zeolite was taken as a porous object. Zeolites are nonstoichiometric compounds, the compositions of which vary over wide limits and produce a series of solid solutions. More than 40 structural types of natural zeolites are known today, of which mordenite and clinoptilolite (used in this work) are the most abundant. The value of zeolites is that they all have an open-work aluminosilicate frame consisting of a set of voids and channels. Their sizes (windows) are large enough (0.26–0.27 nm) for molecules and ions of most organic and inorganic compounds to penetrate into them. The frameworks of zeolites are formed by silicon and aluminum animonites. Owing to its structure, the framework has a negative charge, which is neutrallized by the cations of alkali elements, alkaline-earth elements, and water molecules that are present in the voids and channels of the framework, which are loosely bonded to it. The water can be removed by heating or vacuumizing the zeolite. These procedures do not influence the rigid framework: its structure remains almost unchanged. The object of investigation was monoclinic clinoptilolite with the space symmetry group $C2/m$; unit cell parameters a = 1.761 nm, b = 1.780 nm, and c = 0.741 nm; and β =115.2°. In experiments, 20.0×10.0×1.2 mm zeolite plates were cut from a large block of clinoptilolite (natural zeolite), the chemical compositions of which was the following (X_ray analysis data): Al_2O_3, 11.360; SiO_2, 67.840; Na_2O, 1.250; MgO, 0.490; P_2O_5, 0.110; SO_3, 0.030, K_2O, 3.010; CaO, 0.290; TiO_2, 0.080; MnO, 0.078; Fe_2O_3, 1.190; and KJ, 11.640 at %.

The dimension of zeolite plate is 20×10 mm^2 and thickness is 1,2 mm. The plane-parallel zeolite plate ground on both sides was placed in cells of two types: (i) the plate was sandwiched in flat metallic electrodes, one of which (anode) is a semi-transparent conductive SnO_2 layer on a glass disk and the other (cathode) is a polished metallic Cu disk, or (ii) the plate was separated from the anode by a insulating mica sheet with a circular aperture at its centre providing a 40 μm thin gas gap between the zeolite plate and the anode. Typical diameter of the gas discharge gap area is 9 mm. Both electrodes completely covered the zeolite plate. The cell was placed in a chamber equipped with windows for visual observation and recording of the GDLE, electrical leads, and an evacuation outlet. The experiments have been carried out on the system set-up shown schematically in Fig. 1.

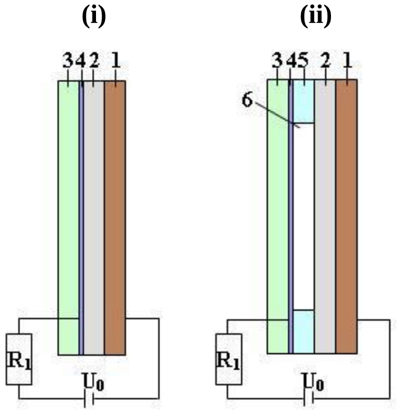

Fig. 1. Scheme of the gas discharge cell: (i) 1- metallic contact; 2- zeolite plate; 3- flat glass disk; 4- semi-transparent conductive SnO_2 contact. (ii) 1- metallic contact; 2- zeolite plate; 3- flat glass disk; 4- semi-transparent conductive SnO_2 contact; 5- insulating mica sheet; 6-gas discharge gap.

The pressure in the chamber was monitored by a digital manometer attached to a pumping system and was kept at a certain constant value during the whole course of measurement. The assessment of the emission spectrum was then based on analysis of the GDLE (330 – 440 nm), recorded through a transparent anode. The GDLE was observed through the semi-transparent anode and registered by an intensified CCD camera. A potential difference of up to 1 kV was applied between the electrodes of the cell. The current voltage characteristics were recorded by gradually varying the voltage at the rate of 3 Vs^{-1}. The maximum sensitivity for the current axis was 10^{-9} Acm^{-1}, and for the voltage axis it was around 0.5 Vcm^{-1}. The entire experimental study of this work was performed in ambient air from atmospheric to 10^{-2} Torr pressures and the measurements were carried out at room temperature.

3. Results and discussion

As is well known, zeolites of silicoaluminate, have negatively charged frameworks with ion ex-changeable sites; hence, they tend to favor adsorption of positively charged ions. There are two ways to move charged particles in a plasma space: drift by an electric field and diffusion by the concentration gradients described in a drift diffusion equation with several assumptions [20].

$$\acute{\Gamma}_p = \mathrm{sgn}(q_p)\, \mu_p E n_p - D_p \nabla n, \qquad (1)$$

where $\acute{\Gamma}$ is the flux of particle p, E is the electric field, q_p is the particle charge, μ_p is the mobility, and D_p is the diffusion coefficient. Assuming all the samples have a similar electric field between the electrodes, the diffusion of charged particles into the nanosized pores of the zeolite crystal can cause a large change in the displacement current.

To test the assumption that the steady gas discharge can be initiated in the pores of a zeolite plate, we considered a system, in which the anode is separated from zeolite plate by an insulating mica sheet with a circular aperture at its centre providing a 40 μm thin gas gap between the zeolite plate and the anode. Fig. 2 shows the current–voltage characteristic (CVC) of the zeolite plate with push electrodes for different air pressures in the cell. The characteristic was taken as follows. After the working pressure in the cell had reached a desired value, different constant voltages were applied to the cell and, after the voltage had reached a steady value, the current was measured. Curves *1–3* were taken as pressures of 60, 45 and 200 Torr, respectively. They exhibit an initial linear portion, which turns to near-exponential with increasing voltage. Curve *4* was taken at a cell pressure of 0.1 Torr.

It should be noted that Shikhaliev [21] proposed a model of field enhanced self sustained electron emission in porous dielectrics, which finds application in IR imaging devices [22]. In the works cited, the electron emission from pores and nanotubes is attributed either to the effect of field enhancement near the top of the nanotube or to the avalanche multiplication of charge carriers due to the impact ionization of the nanotube walls. Another view of the mechanism underlying the electron emission from pores was put forward by Tatarinova [23], who observed a current in the vacuum gap that was emitted from the cathode with a porous surface and from getters. The CVC was linear, and the current depended on the gas saturation of the getters and pores on the cathode surface. As the applied voltage rose, a self-sustained gas discharge was initiated and the linear CVC became exponential. It was hypothesized that the resonance desorption of the gas in the presence of water, oxygen, or nitrogen molecules is the only physical phenomenon that can be responsible for the emission. Negative ions recombine on the surface of pores, liberating an electron, the energy of which (several eV) suffices to maintain the resonance desorption of the gas. According to this hypothesis, a gas discharge is ignited in surface pores. When the electric field "sags" into the volume of a cathode pore, the resonance desorption of the gas produces a gaseous medium in it. With a rise in the voltage, the gas ionizes and starts glowing, thus supporting the idea that a gas discharge can be initiated in gas-filled pores on the surface of the negative electrode.

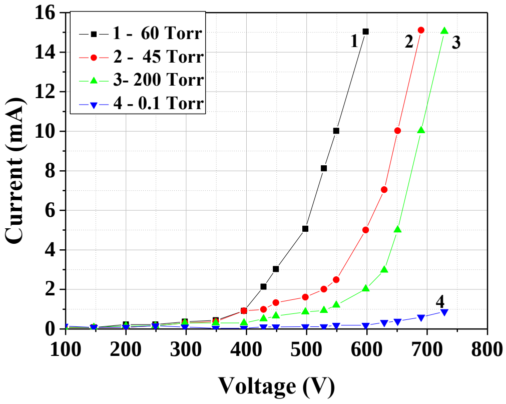

Fig. 2. Current I vs. voltage U for the zeolite plate at a residual air pressure.

At voltages corresponding to the linear–exponential transition, GDLE spots with an intensity growing with rising voltage appear on the side of the plate that is pressed against the positive transparent electrode (Fig. 3). Townsend discharge is the simplest type of gas discharge, where the effect of the self-consistent field is minimal. Hence, the study of its stability and the formation of spatially inhomogeneous two or 3-D structures can shed a light on the mechanism for such phenomena in more complicated systems [24]. Recently, Hensel *et al* studied microdischarge inside porous ceramics using an ICCD camera, and found a strong dependence of discharge characteristics on the pore size of the ceramics [25]. These visual observations provided intuitive insight into the understanding of the physico-chemical phenomena in the discharge plasmas.

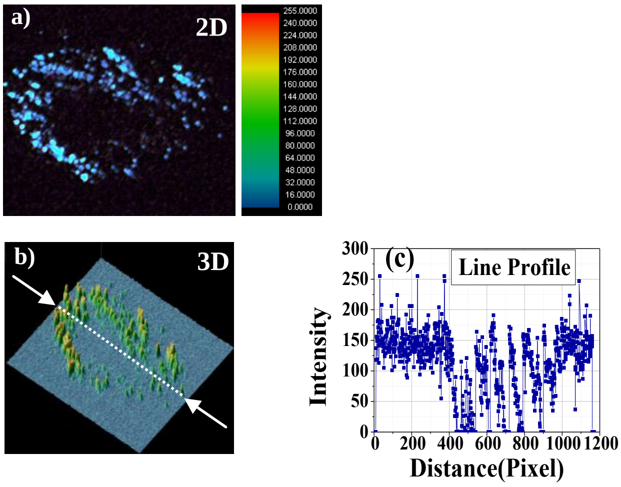

Fig. 3. 2D and 3D patterns of the discharge plasma from the pores of zeolite plate and variation of the GDLE intensity profile.

It should be noted that the diameters of the micro-discharges were not constant and changed with position. The measured diameter of the microdischarges was about 85–150 μm. The diameter did not depend on the applied voltage.

The pressure dependence of the ignition voltage shown in Fig. 4 (curve 1) obeys the Paschen law. At voltages $U > 650 – 800$ V, the current through the zeolite plate reaches several milliamperes and becomes unstable. A further increase in the voltage leads to the breakdown of the SnO_2 film where this electrode is in contact with the plate, and the surface of the glass is damaged. In the plate kept in an evacuated chamber (curve 4, Fig. 2), glowing spots were absent up to $U = 1$ kV. Thus, we determined optimal conditions for igniting a steady discharge in the air contained in the pores: a pressure falling into the interval 30–100 Torr and a voltage of 450 V. Note that, in the absence of the zeolite plate, when the width of the electrode spacing equalled the thickness of the plate the GDLE was not ignited up to U=1 kV. Fig. 5 is the image of a gas discharge in a narrow gap (d= 40 μm) between the transparent anode and zeolite cathode, and Fig. 4 (curve 2) shows the corresponding Paschen curve. The uniform light emission of the gas over the gas gap is seen. It is assumed that the electrons having been emitted from the nanopores of the zeolite cathode ionize the gas.

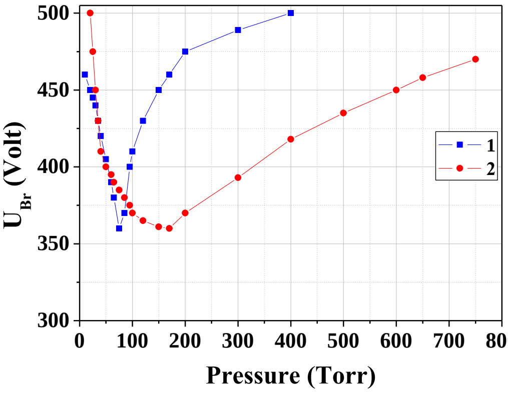

Fig. 4. Discharge ignition voltage U_{Br} vs. air pressure P in the (curve 1) pores of the zeolite and (curve 2) gas discharge gap with the zeolite cathode.

By direct observation of the gas discharge, it is established that the sources of the GDLE were the nanopores on the zeolite plate surface. In addition, we obtained direct experimental evidence for the gas discharge ignition in the nanopores of natural zeolite plate placed in a constant electric field, i.e. the multiplication of electrons by ionization of the gas in the pores. In other words, nanopores in zeolite are effective emitters of electrons. Fig. 5 shows GDLE in the gap with zeolite cathode. Two electrode plates were placed in parallel inside a vacuum chamber in a separation of 40 μm to test the plasma ignition. Very bright visible light was observed on the zeolite plate when the air plasma ignited between the electrodes shown in Fig. 5.

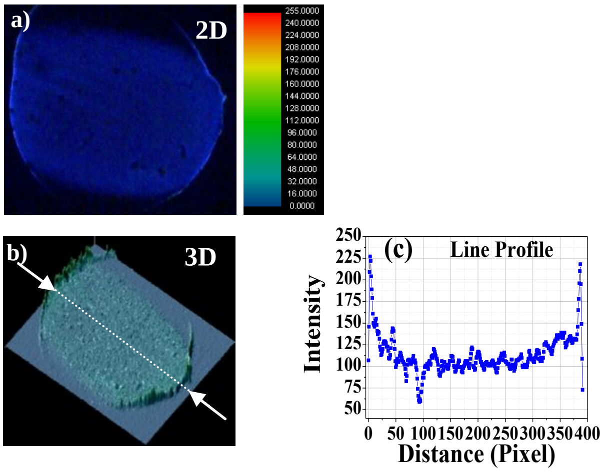

Fig. 5. 2D and 3D patterns of uniform GDLE in the gap with. the zeolite cathode and variation of the GDLE intensity profile.

From the optical emission spectroscopy, it is clear to see that the air plasma generated in the ultraviolet (UV) and visible (blue) range (330 – 440 nm). Unlike dc glow discharge generated by planar electrodes, zeolite acts as the needle-array electrode which eventually produces streamer discharge [26]. We found that there is no cathode dark space in front of the cathode plate. Therefore, the high density cathode glow regime merged and gives better visible light emission. The zeolite electrode also reduced the "operation voltage" which is defined as the minimum voltage to sustain the plasma after the ignition. However, the surface of the zeolite only slightly affects the current density and visible light intensity. It may due to the space charge in the plasma regime which limited the current. However, chamber pressure affects the plasma ignition voltage directly.

A comparison of our findings for type (ii) structure cell and the data obtained from type (i) structure cell shows that the presence of the gas discharge region does not significantly modify the character of the charge transport in our system (see Fig. 6). When zeolite cathode with linear CVCs is used in the type (ii) structure cell, a spatially uniform distribution of the current density stays stable up to fairly high values of *j*. Such uniform distribution is easily visualized from the distribution of the intensity of the GDLE. Two-dimensional (2D) and 3D pattern of GDLE in the cell has been presented in Fig. 5. At the indicated conditions, the low-current gas discharge does not exhibit any pattern formation by itself and is characterized by a low temporal and spatial noise [27].

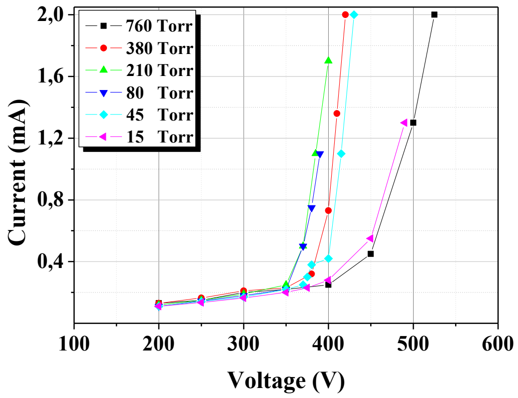

Fig. 6. CVCs of the gas discharge gap with the zeolite cathode with respect to pressure. The 40 μm thick discharge gap is filled with air.

The results obtained can be summarized as follows.

(i) The current in the CVC of the zeolite plate sharply grows at certain pressure-dependent voltage U_{ign}. At pressures below 0.1 Torr, the current is almost independent of the pressure.

(ii) At high feeding voltages, the GDLE of the nanopores is seen through the transparent anode.

(iii) The GDLE wavelength, $\lambda = 0.3 - 0.4$ μm, corresponds to a normal gas discharge in the given medium (air). Although the intensity of the GDLE was controlled by the operating current and voltage, the operation voltage cannot be too high to avoid high energy ion bombardment on the cathode which would eventually damages the transparent anode layer [28].

(iv) The pressure dependence of the ignition voltage obeys the Paschen law. The reduction of the electrode separation enhanced the electric field which reduced ignition voltage further. By optimization of the gas pressure, a minimum ignition voltage and operation voltage was obtained. Due to the small gap between the electrodes, higher pressure was required to exceed the Paschen minimum.

(v) In the discharge gap, where the zeolite plate serves as a cathode, a steady gas discharge arises, which uniformly covers the electrode.

Based on the above results, it can be concluded that a steady gas discharge can be initiated in the pores of a zeolite plate. Since nanopores in a zeolite plate go through, they contain a gas with a small amount of electrons and ions in the absence of an electric field. These carriers provide a steady through current (the linear portion of the CVC). At fields causing the exponential run of the CVC, the amount of electrons and ions grows and a gas discharge arises. Its GDLE from the pores emerging on the zeolite plate surface pressed against the transparent anode is observed. With an increase in the field, so does the gas-discharge steady current, which eventually turns into a breakdown current. The experimental data suggest that the amount of current carriers increases through the ionization of the gas contained in the pores rather than through the ionization of the pore wall material, because the current disappears at low pressures. It should be noted that the described model, where the zeolite plate play a role of the virtual cathode is involved for an explanation of the gas discharge ignition in the nanopores of zeolite placed in a constant electric field that it takes place in. Further measurements are on going, to further characterize the electronic and ionic conductivities and their interplay, and the transport properties in the zeolite-like materials. More detailed studies on the optical spectroscopy, gas composition and the correlation between the different parameters are currently in progress and will be reported elsewhere.

4. Conclusions

Thus, we obtained for the first time to our knowledge direct experimental evidence for the gas discharge ignition in the nanopores of natural zeolite placed in a constant electric field. In addition, it was found that zeolite can be applied as a cathode material that reduces the discharge ignition voltage in a vacuum gap by several hundreds of volts; in other words, nanopores in zeolite are effective emitters of electrons. In a gas -discharge cell where a zeolite plate is used as a cathode (i.e. (ii) type cell), the electrons emitted by it ionize the gas in the gas discharge gap, causing a GDLE throughout the gap. It can therefore be supposed that cheap natural zeolite will be effective in low-power gas discharge devices and flat cathode-luminescent light sources. We propose a design of flat panel plasma light source without additional optical components which has a simple configuration to achieve the low power consumption and uniform light emission.

The unique electronic properties of nanopores zeolite make them also good candidate materials in the electronic industry, so that silicon-based technologies could be replaced or supplemented by nanopores zeolite-like materials. The possible applications of zeolite nanotubes range from electronics, field-emission displays, energy storage devices, functional fillers in composites, and biomedical applications. These have attracted both industrial and academic interest. However, due to the gap between basic research and the requirements of real-world applications, small nanotubes are facing technical difficulties such as dispersion, chirality selection, and handling. Therefore, it is important to smoothly and efficiently transfer the basic findings to industries to commercialize the novel technologies rapidly.

Thus, our result can be used for the spatial stabilization of the discharge plasma of the cell to optimize the parameters for the development of low-power microdischarge devices where a stable homogeneous discharge is required. In summary, we believe that gas discharge cell (GDC) with zeolite electrode comprising usual semiconductor gas discharge system [29-32] have significantly improved performance in terms of dc breakdown voltage, nondestructive behaviour, and reliability over the current commercial GDCs.

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