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RESEARCH NOTE

AC Electrophoresis; Deposition of Ceramic Nanaoparticles on In-plane Electrodes at Low Frequencies

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ABSTRACT

In this paper, deposition of ceramic nanoparticles on in-plane electrodes under the influence of AC electric fields is investigated. Particles are dispersed in a non-aqueous suspension and the frequency range is between 0.01 Hz ot 10 kHz. Analysis of the particle response to the applied field is a difficult task due to the mutual effect of electric- and hydrodynamic force which are present in the system. In this work, however, we show the possibility of dividing the frequency range into four domains with four distinct governing mechanisms. Possible mechanisms are suggested and dominant forces are determined for each domain. In situ optical microscopy observations as well as numerical calculations are used for three dimensional visualization of nanoparticles´ movement dispersed in liquid medium. New applications such as micro-patterning and sorting ceramic particles are introduced for the first time.

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1. INTRODUCTION

Electrophoreic deposition (EPD) is used for preparation of free-standing objects and colloidal coatings [1]. In classical EPD process, direct current (DC) electric field is applied. Recently, application of modulated electric fields such as pulsed direct current (PDC) and alternating current (AC) are reviewd as innovations in EPD [2, 3]. It is shown that applying modulated electric fields can decrease electrolytic decomposition of water. As a result, formation of bubble-free ceramic coatings at high voltages from aqueous suspensions is possible [4-7]. It is important to note that in most of these researches, assymetric AC electric fields is used since it is belived that in symmetric AC electric fields charged ceramic particles oscillate around a fixed point [1]. Consequently, net movement of particle is zero and no deposition is formed on electrode surface. Despite the fact that symmetric AC electric field is not preferred for deposition of ceramic nanoparticles, these fields can be used for deposition of nanoparticles on in-plane electrodes. For this method, applications such as thick film deposition [8], gas sensor fabrication [9], micro-patterning [10] and particle separation [11] are introduced and tested successfully. In fact, our previous works demonstrate that using symmetric AC electric field facilitates manipulation of nanoparticles [8-11]. Besides, expensive methods of fabrication of miniaturized electronic parts can be replaced by the proposed method.

Manipulating ceramic nanoparticles in non-aqueous liquids by applying AC electric field at low frequencies is investigated in this work. Experiments as well as computations are used to explain some phenomena not reported before. Consequently, applications such as simultaneous deposition of charged ceramic particles on both electrodes, electrohydrodynamic focusing of nanaoparticles in the gap, separation of particles of different shapes and chain formation in ceramics are introduced for the first time. In addition, our attention is focused on low frequency range (below 10 kHz) which is not studied before.

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2. EXPERIMENTAL

In order to make the desired electrodes, a gold layer was coated on a glass substrate with painting technique using gold paste (Degussa, GZ117). Then, the assembly was fired at 600 °C for 1 h. Finally, Laser radiation was used to separate the gold layer into two parts leaving two in-plane electrodes with a gap distance of about 120 μm. The dilute (< 1 g/L) suspension was prepared for deposition process in three steps. First, the desired amount of ceramic nanopowder was added to non-aqueous medium. In the second step, the complex was ultrasonicated for 15 min. Finally, the suspension was kept intact for one hour to ensure its stability for deposition process. The equipments used for deposition process include a function generator (RIGOL DG1022), a voltage amplifier (hp 6826A) and a digital storage oscilloscope (Instek GDS-2204). An optical microscope was used to capture microscopic images. In addition, to investigate the deposition pattern, a scanning electron microscope (Hitachi.S4160) was used. Experimental set-up and gold electrodes are explained in [8-11].

3. RESULT AND DISCUSSION

Four frequency ranges are introduced by analyzing videos captured during particle movement. In addition, SEM images of deposition patterns formed at different frequencies (from 0.01 Hz to 10KHz) confirms this classification. Dominant mechanisms and applications in each frequency range are described in the following section.

At frequencies below 1 Hz, it can be assumed that deposition is performed under DC condition since the period of time inwhich electrode polarity changes is long enough. However, in contrast to DC electric field, deposition can be observed on both electrodes when AC electric field is applied. Here, the electric force exerted on particles is oscillatory which means that they are moved to the counter electrode, following the electric field lines in the first half cycle. Then, by reversing electrode polarity in the next half cycle, they change direction and move toward the other electrode and the process is repeated. Consequently, a layer is deposited on both electrodes gradually; an observation which is not seen in DC–base setups. Another interesting feature is that when deposition is promoted on electrode surface, width of the deposition area is frequency-dependent. To explain the effect of applied frequency on deposition pattern , first, the shape of electric field lines which is produced by in-plane electrodes should be studied. If parallel, electric field is constant and uniform between two electrodes. However, in the case of in-plane electrodes, the field is not uniform with a maximum established near the edges. It means that in some regions above electrode surface, electric field is

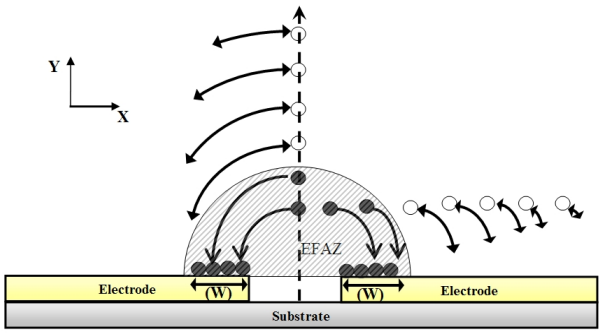

Figure 1. A schematic representation of nanoparticle deposition. Hatched area defines EFAZ

weak, unable to force particles to deposit on electrode surface. So, there exist particles that oscillate in the suspension. The situation is depicted in Figure 1 schematically. This figure shows that black particles deposit on the surface while white particles oscillate in the suspension. Right side of the figure shows particles located in one row at the same distance above the surface. For a constant elevation, and moving away from electrode edge the oscillation amplitude is decreased since electric field is weakened. The same situation holds for particles positioned at different heights as it is indicated on the left side. As the electric field has a decreasing tendency with elevation, the oscillation amplitude is reduced. Applied frequency produces the same effect. i.e., increasing frequency decreases the amplitude of oscillation which, in turn, impedes particles reach the electrode surface yielding to a narrower deposition area. Based on above explanations, a region above surface of in-plane electrodes called Electric Field Affected Zone (EFAZ) is defined where the field is strong enough to force particles to deposit on electrode surface [12, 13]. This zone –introduced by us for the first time- which is hatched in Figure 1 divides the domain into two sections; Inside EFAZ, nanoparticles can deposit on electrode surface, while beyond it, nanoparticles only oscillate in the domain. Dimensions of this region can be estimated by equating electric and drag forces in EFAZ. First, velocity of spherical particle, dispersed in non-aqueous medium, is obtained from Equation (1) [12, 13].

$$V = \frac{2E\varepsilon\xi}{3\eta} \quad (1)$$

In Equation (1), η is viscosity of suspension. ε is permittivity of medium. ξ is zeta-potential of particle in liquid, and finally, E is the applied electric field. Second, the maximum distance that a particle moves in one half-cycle (X) results from Equation (1) as below:

$$X = \frac{E\varepsilon\xi}{3\,f\eta} \quad (2)$$

where, f is the applied frequency. Equation (2), shows that the domain in front of electrode in which particles are affected by electric force is decreased by increasing frequency.

EFAZ for parallel and in-plane electrodes can be compared based on Equation (2). In parallel assembly, the distance between the electrodes is in the order of centimeters. However, this can be reduced to a few micrometers for in-plane electrodes. As a result, the latter may experience electric fields of higher strength as compared to parallel electrodes at the same voltage. Consequently, according to Equation (2), EFAZ -a zone in which particles are affected by oscillatory electric field and can deposit on electrode surface in one half-cycles- grows on the in-plane electrodes. It is important to note that electric field is not uniform. Thus, Equation (2) does not provide an exact value for the width of EFAZ. This equation simply implies that width of deposition is inversely proportional to the applied frequency. This statement is compatible with experimental observations.

In summary, although in AC electric field the electrophoretic (EP) force is oscillatory, our observations show that below 1 Hz, particles are moved through the liquid medium and deposit on electrode surface. So, the process is analogous to deposition under DC electric field. However, under AC conditions, deposition is performed on both electrodes and its area is dependent on EFAZ which, in turn, is proportional to the applied electric field and inversely proportional to the frequency.

Between 1 - 10 Hz, deposition is performed mostly inside the gap. A new type of fluid flow is observed that is not reported before and can not be explained by classical mechanisms of AC electrokinetics such as electrothermal or electroosmosis. Real-time video imaging during deposition process shows that this fluid flow is responsible for gap filling. Previously, we reported another mechanism for gap filling where electric field lines change direction toward center of the gap due to the surface conductance of glass substrate [14]. However, further studies reveal that this mechanism works near electrode edge where electric field is strong [10].

Preliminary observations by video imaging show that the system is subjected to a sudden impulse when the power is turned on at each frequency. When frequency is lowered to 1 Hz, the applied shock is repeated every 0.5 seconds. At higher frequencies, say, 1000 Hz, the system experiences the shock only once at the beginning moment. It implies that the electrostatic equilibrium of the system is disturbed whenever the power is turned on or off. Electrical neutrality is established when the ions are distributed homogeneously in the system [15]. In other words, after applying voltage to the electrodes, ion distribution in the liquid is affected. Most of the ions are attracted toward electrode edge where electric field strength is at maximum to keep the electroneutrality of the system. Consequently, migration of ions near the edge can lead to liquid flow. When the applied voltage is increased, ions far away from edges are directed toward electrode. As a result, this phenomenon is enhanced by increasing the voltage. This new type of fluid flow is important in various aspects. First, it is confined in a very small region in micro-scale range. Second, the intensity of fluid flow is adjustable by controlling process parameters like applied voltage and frequency as well as liquid properties such as conductivity and viscosity. In the present work, we used this electrohydrodynamic fluid flow to fill the gap between two electrodes by ceramic nanoparticles. Further investigations are required to develop more applications.

At Frequency range between 10 and 1000 Hz, nanoparticles are accumulated at a specified distance from electrode edge after deposition process. Figure 2 (a) shows a band of TiO2 nanoparticles collected at a defined distance from electrode edge. The applied voltage and frequency are 40 V and 1000 Hz, respectively. Video images reveal that particles can not deposit near the edges. Instead, they are moved away from the electrode edge toward an equilibrium position on top of the electrodes by an induced fluid flow. Characteristics of this type of flow are consistent with a phenomenon referred to as AC electroosmosis in non-uniform AC electric field.

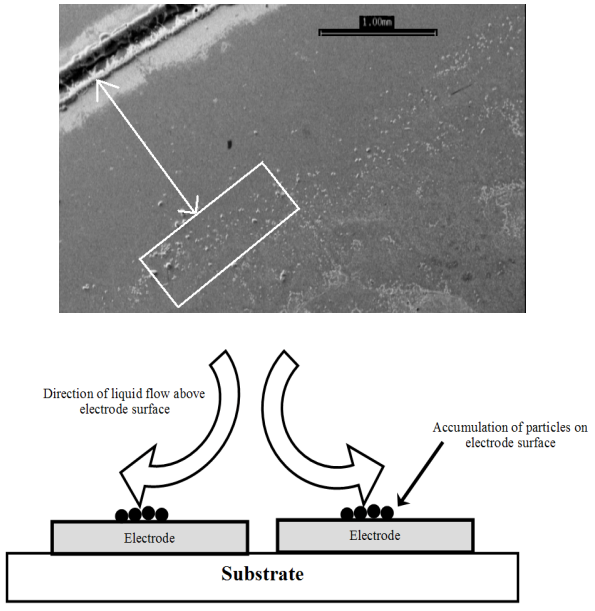

Figure 2. (a) SEM image of particle deposition on electrode surface. A band of TiO2 nanoparticles is formed at a specified distance from electrode edge. (b) A schematic representation of AC-elecroosmosis phenomenon. A vortex is formed on top of electrode and particles are accumulated on surface of electrodes.

First, it is a steady state fluid flow moving from the high strength field regions (electrode edge) onto the surface of the electrode [16]. Second, it is a function of experimental variables such as frequency, voltage and liquid conductivity [17]. The dependency of flow velocity to the process parameters was confirmed in our experimental observations. That is, by increasing the voltage and decreasing the frequency, the fluid flow intensity is enhanced. In AC-electroosmosis a vortex is formed above electrode edges as indicated in Figure 2(b). As a result, suspended nanoparticles which are moving toward electrode edges are pushed away by the effect of induced fluid flow. Induced liquid flow and collection of nanoparticles at a specified distance from electrode edge is shown in Figure 2(b) schematically. It can be seen that particles are collected on both surfaces in a symmetrical manner. This phenomenon confirms that AC electroosmosis is the dominant mechanism in this frequency range. 2D video images from top view help us to understand particle motion. However, a complete 3D visualization of particle motion requires a reliable 2D numerical model from side view.

In order to develop a mathematical model, electric field and bulk fluid velocity were obtained after solving partial equations numerically. Then, particle path was determined by integrating its equation of motion. All effective forces including Brownian, electric, drag and Buoyancy were considered in this equation. To determine bulk fluid flow due to the AC electroosmosis, fluid velocity on electrode surface was measured. For this purpose, movement of particles on electrode surface due to fluid flow was monitored. Then, particle velocity on electrode surface was measured through analyzing recordings. The measured fluid velocities against distance from electrode edge are compared for 100 Hz and 1000 Hz in Figure 3(a). In both frequencies, fluid velocity is decreased exponentially from electrode edge. However, it is higher at frequency of 100 Hz. This was expected in AC-electroosmosis since fluid velocity diminishes gradually at high frequencies. Experimentally obtained fluid velocity was applied to Navier-Stokes equation as boundary condition to obtain bulk fluid velocity. Trajectory prediction of a 100 nm particle above electrode edge is shown in Figure 3(b). The applied voltage and frequency are 40 V and 100 Hz, respectively. Nanoparticle is initially located 50 μm above electrode surface. After three cycles, particle which is oscillating due to the EP force is pushed away from electrode edge due to fluid flow. The effect of liquid flow on particle trajectory is also shown in Figure 3(b) (all other forces are ignored). Since bulk fluid velocity (due to AC electroosmosis) can be controlled by adjusting applied voltage and frequency, it can be used for transferring nanoparticles near electrode edge. Particles give a different response to the applied electric field and hydrodynamic forces based on their size and type. We took this advantage to sort ZnO particles of different shapes [11]. A mixture of ZnO nanorods and nanoparticles (synthesized via different routes) was prepared in acetone. After applying AC electric field to the suspension, it was observed that rods are collected on the edges while particles deposit on electrode surface. In this way, the method can be used for separation and/or purification of complex systems in a liquid medium.

In conclusion, observations and numerical calculations show that AC-electroosmosis is dominant in the frequency range between 10 and 10 KHz. AC-electroosmosis can be used for transporting nanoparticles dispersed in liquid medium. The idea of sorting nanoparticles was tested successfully in this frequency range.

At frequencies above 10 KHz, a phenomenon called chain formation was observed for ceramic nanoparticles such as ZnO, WO_3 and SnO_2. Figure 4 shows a schematic representation of particle alignment bridging two electrodes.

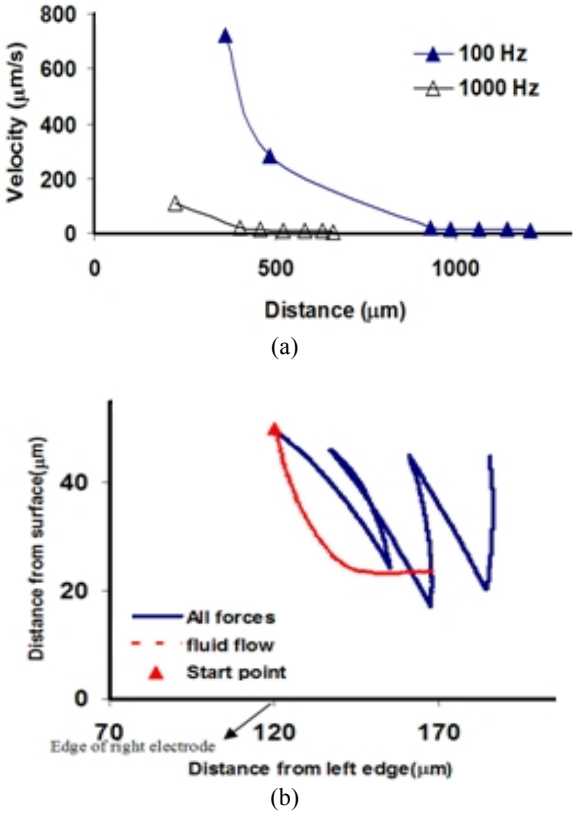

Figure 3. (a) Measured fluid velocity on electrode surface at frequency of 100 and 1000 Hz. The voltage is 40 V. (b) Trajectory prediction of a 100 nm particle located initially at the edge of right electrode at the height of 50 microns above electrode surface. The applied voltage and frequency are 50 V and 100 Hz, respectively

TABLE 1. Mechanisms, applications and observations in each frequency range.

Frequency range	Mechanism	Application	Observation and Remarks
< 1 Hz	Oscillatory EP force	Covering both electrodes at the same time	Nanoparticles cover surface of both electrodes. The area of deposition depends on EFAZ
1Hz to 10 Hz	Unsteady-state liquid flow	micro-patterning, Gap filling and gas sensor fabrication	The gap between two electrodes is filled due to unsteady-state liquid flow
10 Hz to 10KHz	AC-electroosmosis	Particle separation	Steady-state liquid flow pushes particles away from electrode edge
>10 KHz	Chain formation	Gap filling and gas sensor fabrication	Chains formed in the gap connect two electrodes and the gap is filled by nanoparticles.

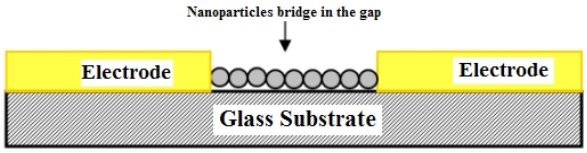

Figure 4. Schematic representation of chain formation in the gap.

Here, the uniform electric field between electrodes induces a dipole within each particle entered to the gap space. Dipoles interact with the external field as well as themselves producing a dipole-dipole interaction [18]. On the other hand, charged ceramic particles interact with each other based on Coulomb rule. As a result, there exist dipole-dipole, charge-charge and charge-dipole interactions. In addition, when particles come close to each other Van der Waals interaction cannot be neglected. These interactions determine the final shape of clusters formed in the gap [18]. Once AC electric field is applied, above mentioned interactions come up and lead to formation of chains between electrodes. When the electric field turns off, chains are deconstructed and nanoparticles are re-dispersed in the suspension. This process is reversible in initial stages. However, if the voltage is applied for a long time (in the order of several minutes) deposition process takes place and electrodes get connected via formed chains. By removing the applied voltage and pulling electrodes out of the suspension the chains are not ruined. Formation of nanoparticle chains bridging the electrodes introduces the process as a promising one for gas sensor fabrication. Sensors fabricated by this method were tested successfully [19]. It is concluded that at frequencies above 10 KHz, ceramic nanoparticles fill the gap between two electrodes. However, the mechanism of gap filling is completely different from the one observed at frequencies between 1 Hz and 10 Hz where nanoparticles fill the gap by unsteady state liquid flow induced near electrode edge due to charging-discharging effect of electrodes. At higher frequencies (above 10 KHz), chain formation is the dominant mechanism.

Table 1 summarizes our findings on AC electrophoretic deposition of ceramic nanoparticles dispersed in non-aqueous medium. The applied frequency range is categorized into four domains based on our observations. Dominant mechanism is presented for each frequency range. Finally, potential applications for AC electric field are introduced in the last column of the table. Thus, AC electric field is an appropriate method for manipulating ceramic nanoparticles, and it can be used for fabrication of miniature ceramic parts.

4. CONCLUSION

Application of symmetric AC electric field for deposition of ceramic nanoparticles on in-plane electrodes in a wide range of frequency (0.01 - 10 kHz) was reviewed. This frequency range was divided into four subcategories according to governing mechanisms determined from video images as well as mathematical models. Below 1 Hz oscillatory Coulomb forces in EFAZ promote particle deposition. Between 1 Hz and 10 Hz, a new type of fluid flow was identified. This electrohydrodynamic fluid flow focuses nanoparticles in the gap space between electrodes. At frequencies between 10 Hz and 1000 Hz, the combined effect of induced fluid flow (AC electroosmosis) and electric forces can be used for sorting nanoparticles. Above 1000 Hz induced dipoles in each particle promote chain formation aligning them in the direction of applied electric field. Finally, applications such as thick film deposition on two-electrode assembly, gas sensor fabrication, sorting particles based on their shape and forming aligned chain of particles were introduced as achievements of this study.

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چکیده

در این مقاله نشست نانوذرات سرامیکی بر روی الکترودهای هم صفحه تحت میدان الکتریکی متناوب بررسی شده است. ذرات در محیط غیرآبی پراکنده سازی شده و فرکانس بین ۰.۰۱ تا ۱۰ کیلوهرتز می باشد. تحلیل نحوه پاسخ ذرات به میدان الکتریکی به دلیل اثرات هم زمان نیروهای الکتریکی و هیدرودینامیکی که در سیستم حضور دارند امر دشواری محسوب می شود. در این تحقیق نشان داده شده است که محدوده فرکانس موردبررسی را بر اساس مکانیزمی که عمل می کند می توان به چهار قسمت تقسیم کرد. مکانیزم های احتمالی و نیروهای غالب برای هر یک از این چهار ناحیه تعیین شده است. از تصویربرداری میکروسکوپی درجا و همچنین محاسبات عددی برای قابل رویت کردن سه بعدی نحوه حرکت نانوذرات معلق در سیال استفاده شده است. کاربردهای جدیدی مانند الگودهی در ابعاد میکرومتری و طبقه بندی ذرات سرامیکی برای نخستین بار با استفاده از این تکنیک معرفی شده است.

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