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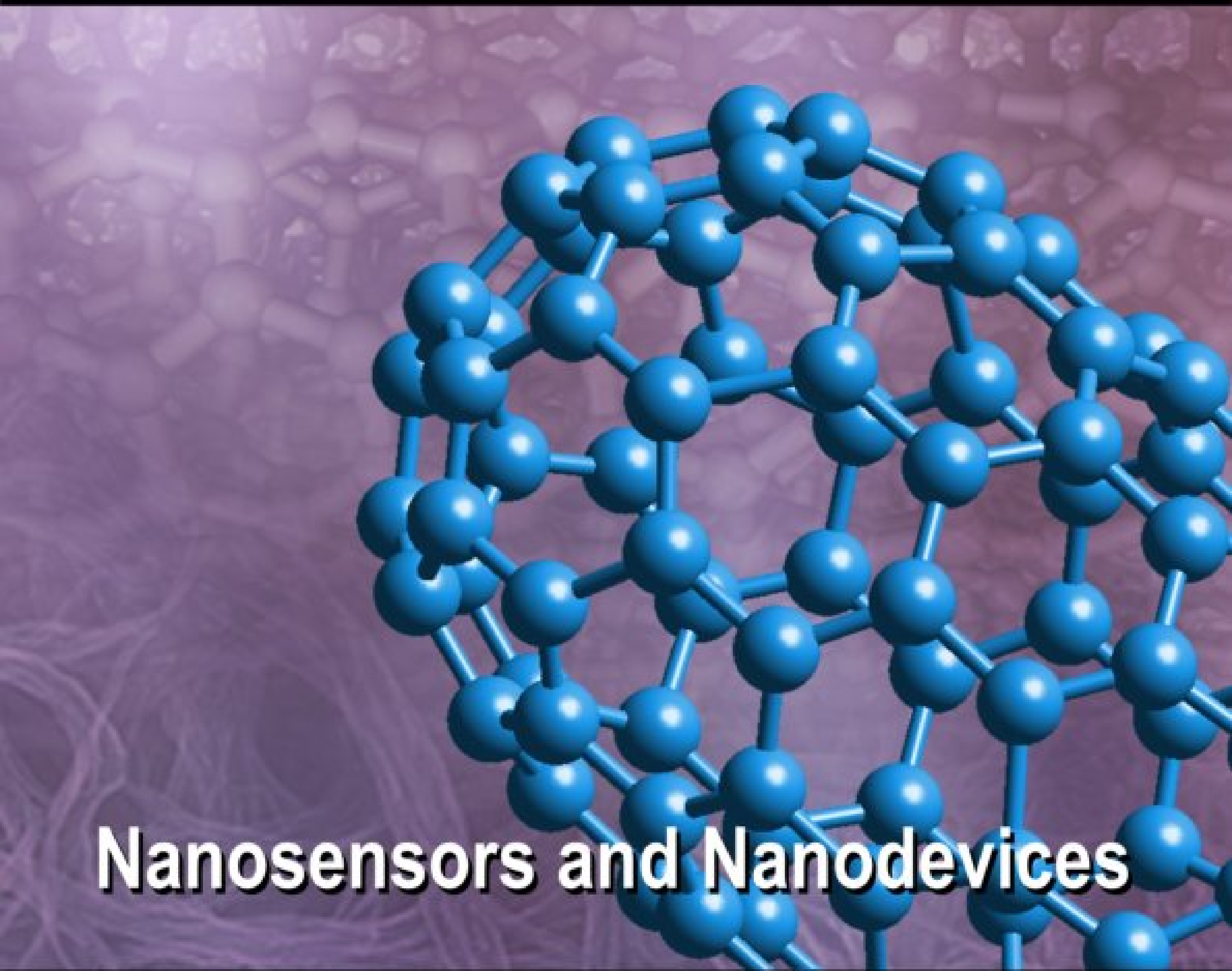

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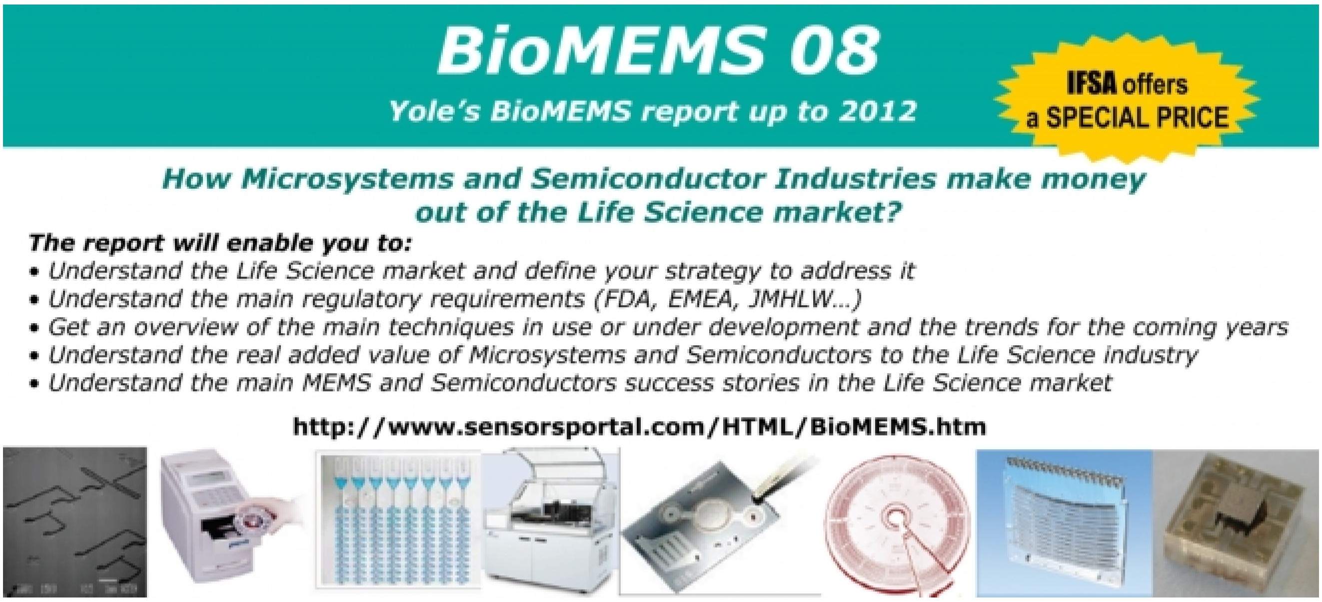

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Camera ready	March 20, 2011

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Camera ready	March 20, 2011

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Electromechanical TiO_2 Nanogenerators

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Abstract: We have developed a nanogenerator that is driven by mechanical forces to produce continuous direct-current output. The nanogenerator was fabricated with titanium dioxide nanoparticle arrays that were placed beneath a conducting electrode with a small gap. The force drives the electrode up and down to bend and/or vibrate the nanoparticles. A piezoelectric process converts mechanical energy into electricity. The electrode collects the output electricity from all of the nanoparticles. The approach presents an adaptable, mobile, and cost-effective technology for powering nanodevices by harvesting mechanical energy from the environment. *Copyright © 2010 IFSA.*

Keywords: Piezoelectric sensor, Electromechanical nanogenerator, Flexoelectricity in nanostructures.

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1. Introduction

The development of wireless nanodevices is of crucial importance to operate biosensors and environmental sensors, for the nanorobotics, for self-powered biomedical devices. Piezotronics is the field of application of semiconducting and piezoelectric properties, which leads to the fabrication of piezoelectric diodes, sensors and nanogenerators [1, 2]. The near future of research is the integration of multifunction devices in a single nanosystem having sensor, control, communication and actuator combined capacities. The piezoelectric effect at nanoscale has been proved experimentally in 2006 [1, 2] and theoretically in 2008 [3, 4] The challenge is to fabricate nanogenerators which harvest the energy of vibrations from the environment, producing electricity.

Recently, an approach for converting mechanical energy into electrical energy has been suggested by using piezoelectric zinc oxide (ZnO) nanowire arrays [1, 2]. The conversion of energy can be achieved by creating a Schottky junction between ZnO and a metallic corrugated zig zag electrode which realizes a rectifying contact with typical resistance of the order ~100 kΩ. The key aspect of such generators is the possibility of driving the top electrode up and down to bend the ZnO nanowires which create a voltage by their piezoelectric properties. This technique has been tested for ultrasound waves with typical output powers P~1 pW per mm^2 with a surface density of nanowires $\sim 10^{10}/cm^2$ corresponding to a diameter of nanowires ~40 nm. In order to become useful to operate a nanodevice composed of a single nanotube working typically at ~10 nW, nanogenerators of this kind need to be optimized. Possibilities of improvements are the use of patterned electrodes and/or the growth of highly uniform nanoarrays of oxide, matching the design of the electrode. It has been estimated that the performance can be improved by two to three orders of magnitude [2].

In this paper we report results of TiO_2 based nanogenerators. The output power of such nanogenerators is produced by piezoelectricity in the nanostructured TiO_2 particles due to the flexoelectric effect occurring in nanoparticles [3, 4].

2. Experimental

The experimental set up is illustrated in Fig. 1. An array of aligned TiO_2 nanostructures of a film deposited on a conducting substrate was covered by a conductive ITO/glass electrode. The ITO coating on glass enhanced the conductivity of the electrode and at the same time created a Schottky contact at the interface with TiO_2. The nanostructures of TiO_2 were obtained by a deposition protocol by a paste deposition of commercial TiO_2 powder (by Degussa) on identical ITO on glass followed by suitable sinterization procedure through heating overnight.

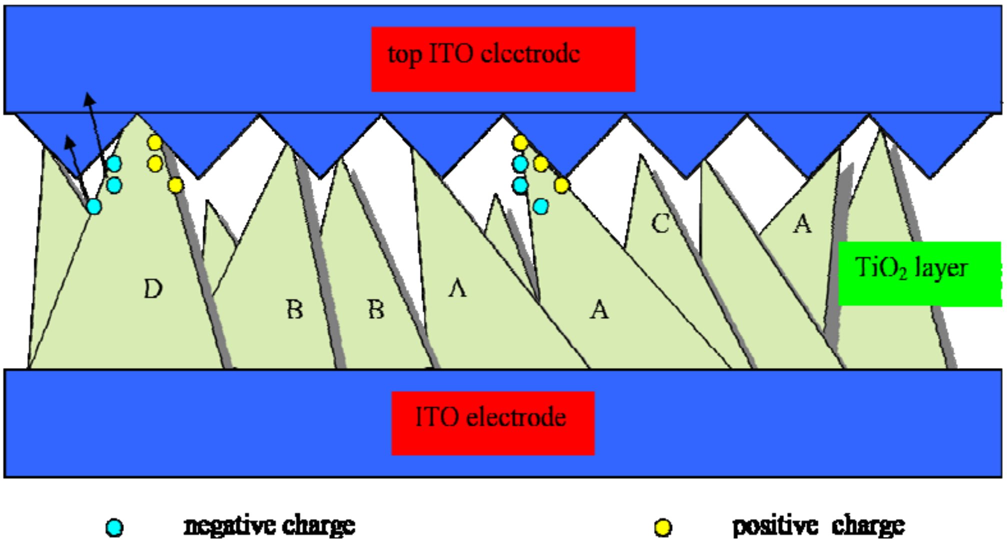

Fig. 1. Set-up of the generator. Nanoparticles are schematically described by cusps that can be bended by the top electrode.

Starting from partly already nanostructured commercial powders (Degussa), comprising a known proportion of anatase and rutile phases (70 %-30 %) [5] we increase the degree of separation of TiO_2 clusters by mechanical machining in acqueous phase, in organic phase or both and/or through sonication of a concentrated emulsion of TiO_2 which helps particle dispersion. Such emulsion is then applied to ITO/glass substrates (Optical Filters Ltd., England). The deposition method is a dr. Blade or spray deposition method. A sintering procedure is applied by heating the devices overnight at a temperature T=550°C. The final TiO_2 films present a uniform distribution at optical inspection with tightly sinterized nanograins. With this technique we obtain films with typical thickness 20 µm.

2.1. Characterizations

We have characterized the films by AFM microscopy. At the µm scale they are constituted by a disordered array in the xy plane with cusps. At larger resolution they exhibit a clear nanometric structure with nanocrystals having comparable dimension ~30 nm within the xy plane and in the z-direction (Figs. 2 and 3). If they were distributed compactly, a surface density 10^{12} cm^{-2} on the average would result.

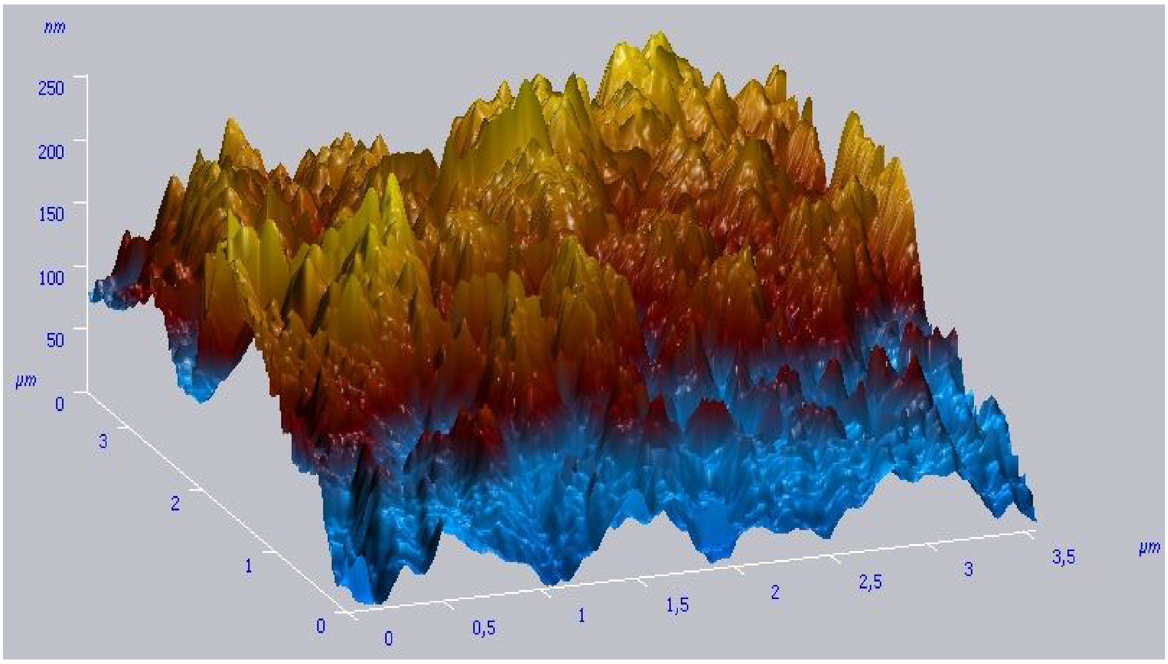

Fig. 2. AFM scan of TiO_2 films at the micron scale.

A similar nanostructure is revealed for the ITO electrode, with slightly smaller density 10^{10} cm^{-2} and diameter and height 100 nm and 5 nm respectively (Fig. 4.)

Each of the structures appears as nanocrystals distributed irregularly in size and orientation.

Typical dimensions of the devices are or order $5x5cm^2$. Various configurations of the devices have been considered. A typical configuration was obtained by placing the ITO electrode in contact with the TiO_2 array and manipulating it by a probe station. Pressure was applied on the sandwich through a calibrated gauge force of maximum value 50 N.

In another configuration the device was sealed to prevent infiltration of liquids; to improve the conductance of the devices an electrolyte was infiltered before sealing, using a procedure similar to the one adopted in the preparation of Graetzel's cells; a typical device of this kind had resistance of order ~100 kΩ. Further arrangements were also explored where the pressing of electrodes were obtained by mechanical stress at the borders of the devices.

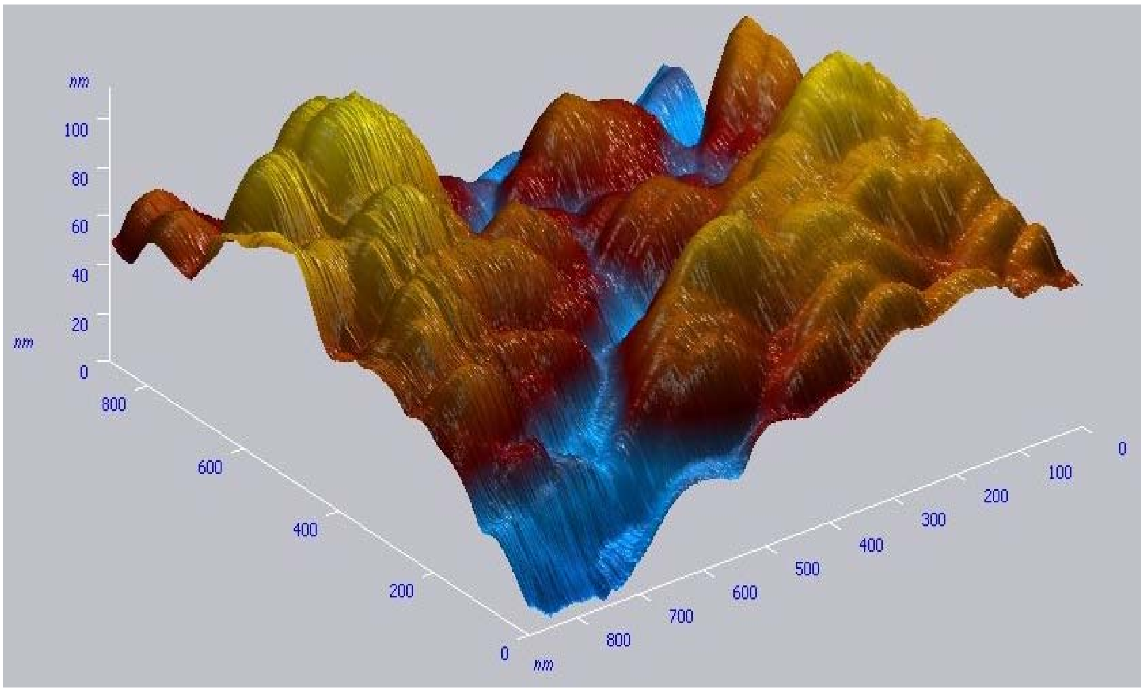

Fig. 3. AFM image of TiO_2 films at the nanometer scale.

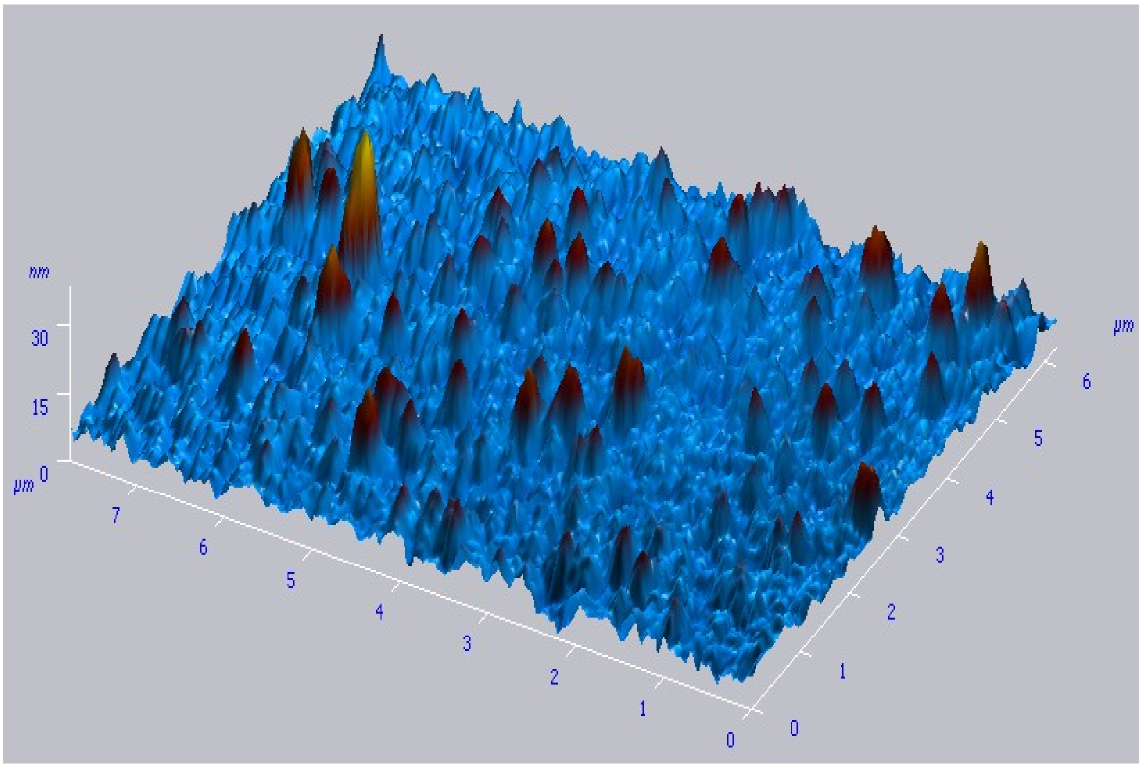

Fig. 4. AFM scan of ITO conducting films on glass. A nanometric structure is present.

2.2. ITO/glass Substrate

The commercial ITO/glass substrates (Optical Filters Ltd., England) have typical surface resistance 20 Ω and a resistivity $\rho = 20\ 10^{-4}$ Ωcm, such values being compatible with those reported in the literature [6].

When used as the substrate for deposition of the oxide, the ITO structure acts as a template to the arrangement of the oxide on it. Good adhesion is obtained for TiO_2 films indicating a good crystal structure matching. From the other hand, when ITO is used as the top electrode, its nanometric array structure is penetrated on contact by the smaller TiO_2 array.

3. The Principle of the Nanogenerator

The physical principle for creating charges in the nanoparticles is a coupling of piezoelectric and semiconducting properties [1]. The deflection of the nanoparticle by the electrode creates a strain field, with the outer surface being stretched (positive strain ε) and inner surface compressed (negative strain ε). This results in an electric field along the z direction inside the nanoparticle and on its surface trough the piezoelectric effect, parallel to the z-axis at the outer surface and anti-parallel to the z-axis at the inner surface. Across the nanoparticle, the electric potential distribution from the compressed to the stretched side surface is approximately between V-(negative) to V+ (positive). The electrode at the base of the nanoparticle is grounded.

The potential is created by the relative displacement of the Ti cations with respect to the O_2 anions in the crystal structure; thus, these ionic charges cannot freely move and cannot recombine without releasing the strain. The potential difference is maintained as long as the deformation is in place and no foreign free charges (such as from the ITO contact) are injected.

The created charges can undergo a discharge process. In the first step, the conductive electrode that induces the deformation is in contact with the stretched surface of positive potential (A-type nanoparticles in Fig. 1). It has a potential nearly zero, so that it is negatively biased with respect to TiO_2 for $\Delta V = 0 - V+ < 0$. The ITO-semiconductor interface in this case is a reverse-biased Schottky barrier and negligible current flows across the interface. In the second step, when the electrode is also in contact with the compressed side of the nanoparticle (D-type nanoparticels in Fig. 1.) this ITO – TiO_2 interface is positively biased for $\Delta V = 0 - V- > 0$. The interface in this case is a positively biased Schottky barrier, and it produces a sudden increase in the output electric current. The current is the result of ΔV driven flow of electrons from the semiconductor TiO_2 to the electrode. The flow of the free electrons through the nanoparticle to the electrode will neutralize the ionic charges distributed in the volume of the nanoparticle and thus reduce the magnitudes of the potentials V+ and V-.

3.1. The Concept of Introducing the Zigzag Electrode

When the top electrode has a configuration as shown in Fig. 1, by scan the electrode back and forth, we can reproduce the two processes discussed above. The electrode will exert a lateral force on the nanoparticles so by successive compression steps we can reproduce the two configurations discussed above and there will be generation of electricity. During the scan nanoparticles may either be bent and make contact with the electrode (A and D-type in Fig. 1.) or contact the electrode without being bent (B-type) or remain isolated without any contact (C- type). The B nanoparticles constitute a parallel resistance of the device (see discussion below), the C nanoparticles are an infinite resistance not entering in the process.

4. Results

TiO_2 generators exhibit a spontaneous voltage V~0.5 V, as measured by a high-impedance voltmeter, within their resistance range R~ 1MΩ, under the pressing conditions of the sandwich which ensure reasonable electric contact. In this state they change the voltage and resistance markedly as a function of pressure on the top electrode. The pressure is achieved and measured through the calibrated force gauge. Both static and dynamic pressure is applied so as to test the response of the device also to definite rates of changing force. The junctions are quite sensitive to small variations of the external force (typical rates 1N/s) with excursions of open circuit voltage V=1-10 mV. Static measurements are reported in Figs. 5 and 6.

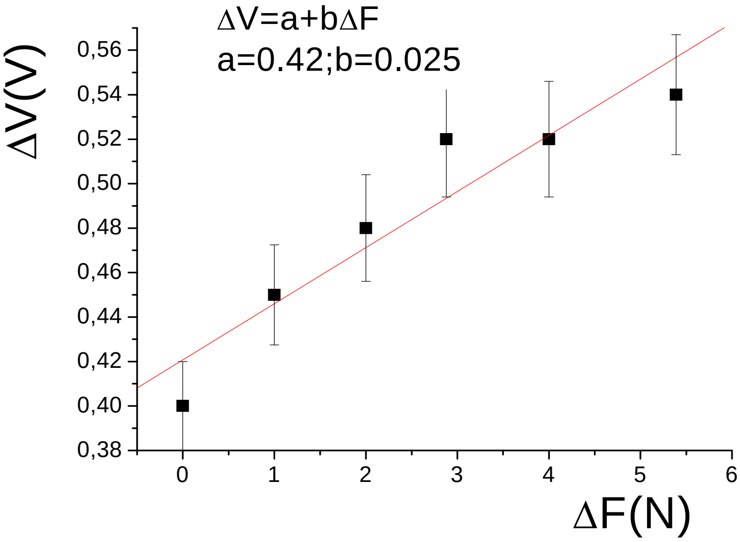

Fig. 5. Induced voltage change vs. applied force (static).

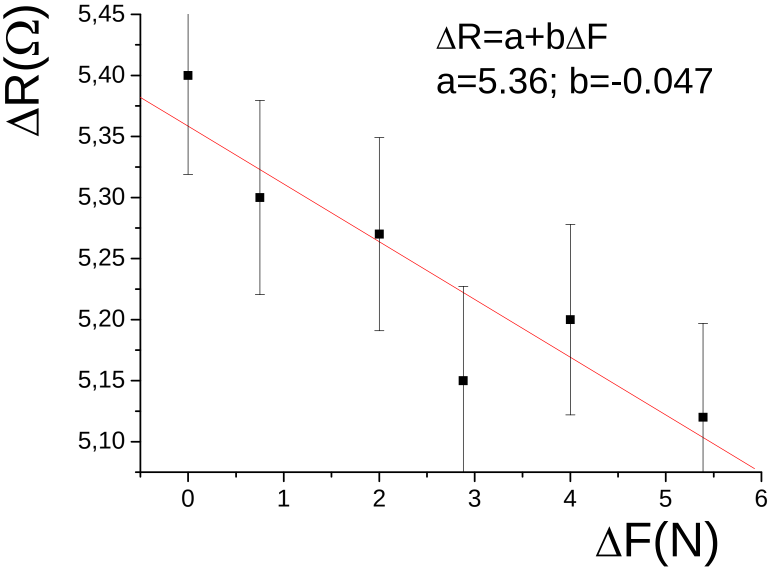

Fig. 6. Induced resistance change vs. applied force (static).

Large fluctuations (from sample to sample and in the same sample) appear in the voltage and the resistance, accompanied by time relaxation towards equilibrium as well as large hysteretic behaviour. Typically, times of order of several minutes can be required to achieve stabilized values. These fluctuations arise from the set-up of the junctions, and are determined by the pressing condition of the top electrode on the oxide film.

The lower contact on the ITO/glass substrate may be assumed to be ohmic, as indicated by the employed deposition method, which is a standard one in the literature [5].

The principal results obtained with such devices are summarized in Table 1. The geometrical parameters are reported in Table 2.

Tab. 1. Electric parameters of TiO_2 and ZnO devices.

	TiO_2 (this work)	ZnO (from ref. [2])
Resistance (Ω)	$5 \cdot 10^6$	$3.5 \cdot 10^3$
Voltage (V)	~ 0.5 (static)~ 10^{-3} (dynamic)	~ 0~ 10^{-3}
Current (A)	~$\cdot 10^{-7}$(static)~10^{-9}	~10^{-9}
Power (W)	~10^{-8}	10^{-12}

Table 2. Geometric parameters of device and film nanoparticles.

	TiO_2 (this work)	ZnO (from ref. [2])
Area (cm^2)	10	$2\ 10^{-2}$
Density cm^{-2}	10^{11}	10^9
Height (nanoparticle)	10 nm	1 µm
Diameter (nanoparticle)	10 nm	40 nm
Film thickness (µm)	20-30	

Since the voltage is increased and the resistance is decreased by the applied force ΔF, the voltage to resistance ratio, i.e. the internal current of the device, is an increasing function of ΔF and so is for the power .

The voltage from the high impedance voltmeter provided the measure of the current itself (see below).

The nanogenerators are equivalent to a voltage source V_s plus an internal resistance R_i and a resistance R_w , parallel to the portion that generates power [2]. As suggested in ref. [2] this resistance derives from nanostructures that are in contact with the electrode but cannot be bent or move freely; thus, they do not actively participate in the current generation, but they do provide a path for conducting current.

The capacitance of the system is ignored in the circuit in order to simplify the discussion about dc measurement. The capacitance of the junctions turns out to be of order of magnitude $C{\sim}10^{-10}$ F.

The short circuit current produced by the nanogenerator is $I_{sc} \approx V_s / R_i$. Since R_w was much smaller than the inner resistance of a voltage meter (ideally infinity), when the voltage was measured at open circuit, a loop was formed between the power generating portion of the system and R_w [2]. In this case, the current is $I_W = Vs/(R_i+R_w)$ and the measured voltage ΔV is that across the power-generating portion,

$$\Delta V \approx V_s R_w/(R_i + R_w) = (Vs/Ri) R_{eq} , \qquad (1)$$

where $R_{eq}=R_i R_w/(R_i+R_w)$ is the equivalent resistance of the device. This leads to a direct proportionality of ΔV to I_{sc}. Therefore, a measurement of ΔV and of the resistance allows a determination of I_{sc}.

5. Discussion

We can explain our results by piezoelectricity in nanoparticles produced by the flexoeloectric effect [3, 4].

Centro-symmetric dielectrics are not expected to polarize under mechanical strain. A non-uniform strain or the presence of strain gradients can however locally break inversion symmetry and induce polarization even in centro-symmetric crystals. This phenomenon is termed fexoelectrictiy [3, 4].

With reference to Fig. 1, at moderate pressure a nanoparticle is deformed and acquires positive (on the stretched side) and negative (on the compressed side) charges. The electric field due to the positive charges turns out to be opposite to the contact field at the Schottky barrier. At quite moderate force therefore, this field corresponds to a reverse bias and negligible current will flow at the junction. At forces of sufficient strength the contact will be through the two sides, so that there will be a current at the negatively biased side with a positive potential at the electrode. This current, which is much higher than the reverse current, will be driven by the potential ΔV- through the nanoparticle. On traversing the junction it will undergo a loop on continuing through the parallel resistance constituted by those particles that are in contact with the electrode without being bent. The potential ΔV- will be at around its maximum value Vs, and the resistance will be the TiO_2 resistance.

5.1. Order of Magnitude of the Piezoelectric Constant

The piezoelectric constant *d* can be defined by the equation

$$\Delta Q = d * \Delta F \qquad (2)$$

where ΔQ and ΔF are the piezoelectric induced charge and the applied stress.

We can derive the value of *d* from the experimental results on resistance. We note that the resistance is determined by all the nanoparticles. Thus, we can write for the conductance change due to the piezoelectricity process

$$\Delta (1/R) = \Delta G = \Delta n * q^2 \tau / m * S/L \qquad (3)$$

Here, S and L are the total area of the device and L its thickness , Δn the carrier density change, τ the relaxation time and m the mass of the carriers and q the electron charge. We can deduce Δn from the induced charge as

$$\Delta n = \Delta Q / qVc\ , \qquad (4)$$

where Vc is the volume of the device. We can thus establish the result

$$\Delta G / \Delta F = e\tau / m * d / L^2 \qquad (5)$$

So we can deduce *d* from the slope of the curve in Fig. 6. With typical values L=10μ and $\tau=10^{-13}$ s we find $d=1.05\ 10^{-11}$C/N.

The electric potential depends on a single nanoparticle. On referring to a simple geometry of the nanoparticle as a parallelepiped it can be written as

$$\Delta V = \delta Q * L / A\varepsilon_o \varepsilon_r\ , \qquad (6)$$

where A is the area of the base, L the height of the nanoparticle and δQ the charge on each nanoparticle. δQ will be

$$\delta Q = d^* \delta F\ , \tag{7}$$

where the force on each nanoparticle can be deduced form the total applied stress ΔF as $\delta F = \Delta F / M$, where M is the number of generating particles so that

$$\Delta V = d^* \Delta F^* L / M A \varepsilon_o \varepsilon_r\ . \tag{8}$$

The quantity $\Delta V/\Delta F$ can be deduced from the slope of experimental curve in Fig. 5. We find from this $\Delta V/\Delta F = 2.6\ 10^{-2}$ V/N. For L=10 nm, $A \sim L^2$ and $\varepsilon_r = 10$ we obtain d=2.3 10^{-11} C/N on assuming that $M = 10^9$.

These values of *d* compare quite well with the ones predicted for non-piezoelectric particles of similar dimensions as a result of the flexoelectric effect. In ref. [3, 4] the value d= 3.9 10^{-11} C/N is found for non piezoelectric nanoparticles at a particle dimension 8 nm.

From the value of *d* one finds the total charge on the array $\Delta Q = 10^{-11}\ \Delta F$, which corresponds to ~0.1 electron/particle, for F=1 N.

Table 3. Piezoelectric parameters of the TiO_2 device.

d coefficient (C/N)	$\sim 10^{-11}$
Charge (electron/particle)	~0.1
Voltage (mV)	1-500
Capacity (F)	$\sim 10^{-10}$
Quality factor	$\sim 10^{-3}$

5.2. Order of Magnitude of Distortion

According to ref. [7] the piezoelectric voltage can be expressed as

$$V\pm = 3T^* y_m / 4Ld \tag{9}$$

where T is the lateral dimension of the particle, L its length and y_{max} the maximum lateral deflection. From this formula we can estimate y_m. On using a typical value V=1V and assuming T ~L we find that $y_m = 10^{-11}$ m, which indicates a distorsion of some percent of the unit cell. A value of this order of magnitude has been reported in [8] as responsible for a ferroelectric instability in rutile crystals.

5.3. Time Response

The capacitance acts as a collector of the piezoelectric generated charge. This capacitance, connected with the device resistance is responsible for the current discharge, which will thus be characterized by a time constant of the order RC~0.1 ms. This number is similar to the one of ZnO nanowire devices [2].

This time can be inferred from energy conservation. The elastic energy introduced by the external force is of the order $\Delta E=\Delta F\ y_m$ so that it is given by $\sim\Delta E \sim 10^{-11}$ J for $y_m \sim 10^{-11}$m and $\Delta F=1$ N. Since the output power is $P\sim 5 \cdot 10^{-8}$ W, this output should be delivered in a time t such that $P\ t=\Delta E$, which gives $t\sim$ 0.1 ms.

5.4. Number of Current-generating Particles

This is an important parameter which determines a kind of quality factor. Since the total current is expected to arise from the generating particles, the maximum current will be obtained if all the particles generate current. This is not so, as can be deduced from the number of generating units $\sim 10^9$. The total planar density of nanoparticles on the surface of device is $N_{tot}\sim 10^{12}$ for an area of device 1 cm^2. Therefore the quality factor is 10^{-3}, i.e. there is one nanoparticle per thousand participating in the current generation, which could be increased by suitable optimization of the deposition of the TiO_2 film of the device.

6. Conclusions

The number of nanoparticles that was active for producing electricity was $N \sim 10^9$. As limited by the multiple contacts between the nanoparticles and the electrode in the present design, the large number of nanoparticles did not produce electricity because of their non-uniformity in height and distribution on the substrate surface; thus, the output current was rather small in the present design. These technical difficulties could be overcome by an optimized design to improve nanogenerator efficiency. For example, nanogenerator efficiency could be improved with the use of patterned electrodes to improve the lateral force on the nanoparticles, the patterned growth of high-quality uniform nanoparticle arrays matching the design of the electrode and an improved packaging technology to keep a precise control on the contacts between the electrode and the nanoparticle arrays. If the grown density of the generating nanoparticles could reach the limit of highly compacted nanoparticles $\sim 10^{12}$/cm^2, the output power per unit of area could be $\sim 10^{-5}$ W. Since the power used to operate a device fabricated with one nanostructure or nanotube is~10 nW [9-11] such nanogenerator could operate up to 1000 of such nanodevices, based on our current study.

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