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An improved AFM cross-sectional method for piezoelectric nanostructures properties investigation: application to GaN nanowires

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Abstract

We present an improved atomic force microscopy (AFM) method to study the piezoelectric properties of nanostructures. An AFM tip is used to deform a free-standing piezoelectric nanowire. The deflection of the nanowire induces an electric potential via the piezoelectric effect, which is measured by the AFM coating tip. During the manipulation, the applied force, the forcing location and the nanowire's deflection are precisely known and under strict control. We show the measurements carried out on intrinsic GaN and n-doped GaN–AlN–GaN nanowires by using our method. The measured electric potential, as high as 200 mV for n-doped GaN–AlN–GaN nanowire and 150 mV for intrinsic GaN nanowire, have been obtained, these values are higher than theoretical calculations. Our investigation method is exceptionally useful to thoroughly examine and completely understand the piezoelectric phenomena of nanostructures. Our experimental observations intuitively reveal the great potential of piezoelectric nanostructures for converting mechanical energy into electricity. The piezoelectric properties of nanostructures, which are demonstrated in detail in this paper, represent a promising approach to fabricating cost-effective nano-generators and highly sensitive self-powered NEMS sensors.

1. Introduction

With the current growing interest in NEMS applications and developments, the self-powering of NEMS devices becomes a more and more popular issue and is a key challenge for novel nanotechnologies. The piezoelectric effect can be employed

to convert mechanical energy into electrical energy, and vice versa. It represents a promising approach for nano-generators or self-powering nano-devices. Many structures of different piezoelectric materials can be used as electromechanical transducers. However, piezoelectric nanowires seem to be the most suitable candidates because of their high length-to-diameter ratio, allowing remarkable strain sensitivity. Therefore, the electromechanical properties investigation and

⁵ These authors contributed equally to this work.

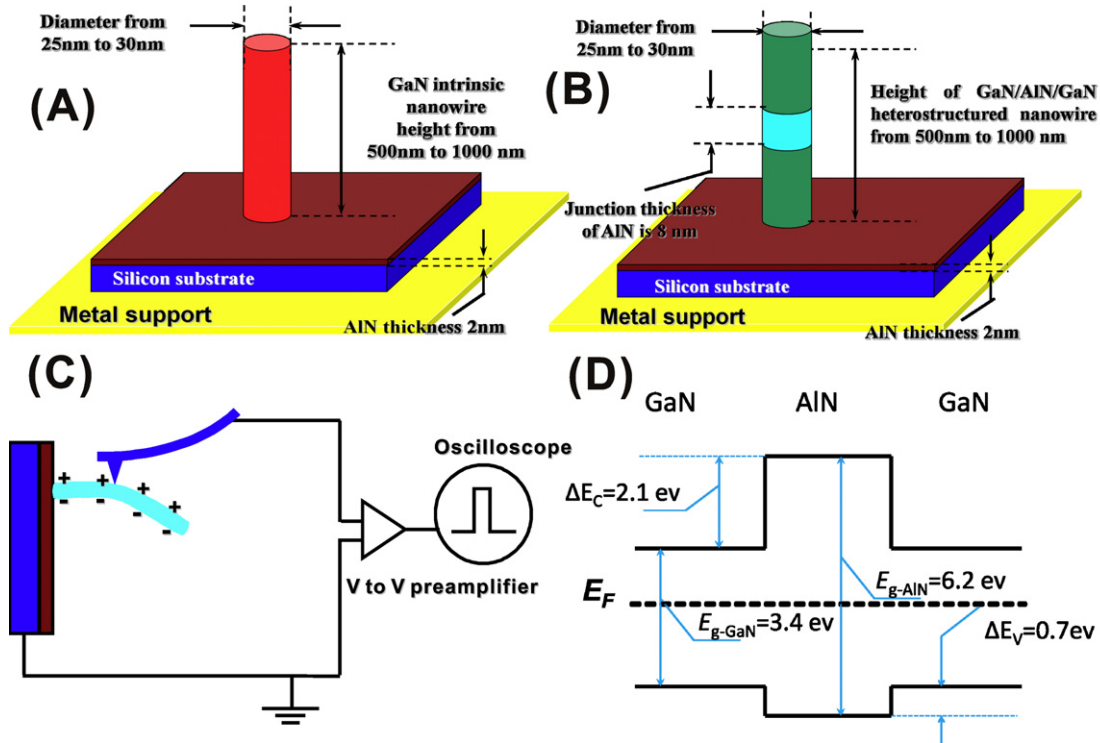


Figure 1. (A) Schematic illustration of GaN intrinsic nanowire dimensions and the sample structure. (B) Schematic illustration of GaN/AlN/GaN heterostructured nanowire dimensions and the sample structure. (C) Experimental configuration, the gain of the amplifier is 10. (D) The band diagram of an n-type GaN–AlN–GaN nanostructure.

characterization of piezoelectric nanostructures have assumed even greater importance owing to the potential large-scale applications of self-powering nano-devices in the near future.

To investigate piezoelectric free-standing nanostructures, appropriate tools as well as convenient methods are required. Atomic force microscopy (AFM) is a classic technique for nanoscale manipulation and nanostructure characterization. Wang *et al* provided an approach to study the piezoelectric properties of nanostructures using AFM [1]. In their work, they demonstrated the distribution of the spontaneous electric potential in a nanowire [1, 2], indicated the impact of the Schottky barrier and estimated that the energy-conversion efficiency of a nanowire-based piezoelectric power generator is between 17% and 30%. However, following the new trends in the development of nanotechnology, a more precise control of the tip–sample interaction and a detailed analysis of the relationship between the applied external force and self-generated electric potential are needed to completely understand the piezoelectric properties of nano-materials because of their great potential for fabricating high-output nano-generators [3, 4]. In this paper, we primarily propose an innovative method to investigate the electromechanical properties of such nano-objects via AFM. As in previous works on this subject, we use the bending of a nanowire by an AFM tip. The deformation of the nanostructure induces an electric potential at the surface of the nanostructure. Both deformation and electric potential can be measured and used to investigate and accurately understand the piezoelectric properties of nanostructures. Our method allows us to select

the appropriate object to be studied, to know its state before and after the measurement, and to precisely control the force applied.

2. Experimental details

Two types of GaN nanowire samples are studied in this work: intrinsic GaN nanowires and Si doped (n-type) GaN nanowires with AlN insertion in the middle of the wire. These GaN nanowires were grown by molecular beam epitaxy (MBE) on Si [111] substrates, their diameters were 25–30 nm and they had a density of approximately 250 nanowires μm^{-2} . Prior to GaN nanowire growth, a 2 nm thick AlN buffer layer was directly deposited to improve the nanowire orientation [5]. Thus, the nanowires can vertically grow along the c -axis ([0001] orientation). In the case of n-type doped GaN/AlN/GaN nanostructure heterostructures, we added Si impurities during the GaN growth. Afterward, an 8 nm-AlN barrier was deposited, followed by the growth of Si-doped GaN. The structures of our intrinsic GaN and heterojunction GaN/AlN/GaN samples are shown in figures 1(A), (B) and 2.

The experiments are carried out at room temperature and normal atmosphere pressure in a quasi-dark box by using AFM (with a high performance controller Digital Instruments Dimension 3100 from Veeco)⁶. Our measurement system is schematically shown in figure 1(C). The sample is vertically

⁶ VEECO Dimension 3100 atomic force microscope with 3 axial (x, y, z) close-loops. Please see supplementary data files (available at stacks.iop.org/Nano/22/105704/mmedia).

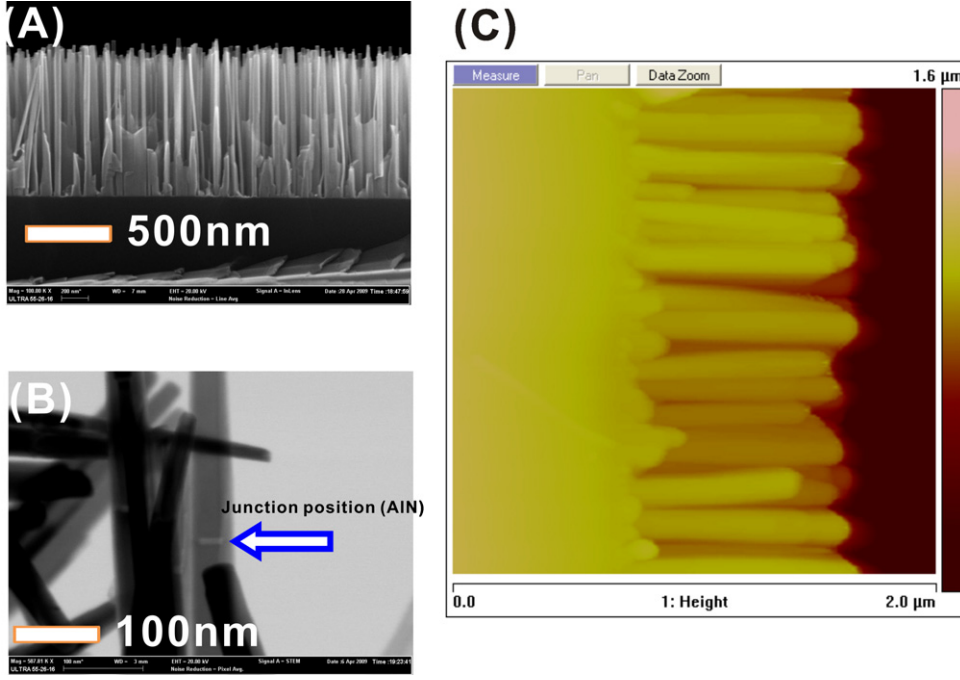


Figure 2. Scanning electron microscopy image of (A) GaN intrinsic nanowires and (B) scanning transmission electron microscopy image of AlN insertion in n-type GaN nanowires (C) AFM topographic image.

fixed on a metal support by Ag-gel to get an ohmic contact⁷. Once an electric potential appears, a current passes through the circuit, which consists of the AFM tip, the measurement setup, the chuck and the metallic support. The total resistance is the sum of the GaN nanostructure's resistance and the contact resistance. Due to the anticipated high tip-sample contact resistance, a high input impedance preamplifier is required to improve the voltage measurement accuracy. After having confirmed no detectable RC discharge effect in our amplifier (less than 1 mV)⁸, a differential amplifier with 100 MΩ input impedance is used. Furthermore, the values of measured electric potential, which are presented in the following, are already divided by 10 (the gain of amplifier). In addition, we present the band diagram of the GaN-AlN-GaN nanostructure, as shown in figure 1(D).

The method that we have developed includes two major steps: image scanning and force manipulation with AFM. The purpose of the first step is to get a topographic image of the sample. A tapping mode scan provides high-resolution images such as topography and phase mode imaging. The suitable nanostructure can be selected by recourse to these images. In the second step, changing from the tapping mode to force manipulation, the conductive AFM tip is used to apply a local force and deform the free-standing nanowire at a selected forcing location, as shown in figure 1(C). The 'force curve' of the AFM cantilever allows a precise control of the applied force, which can be calculated by equation (1).

$$F = K_c d. \quad (1)$$

⁷ Silicon substrate is highly doped. We use the silver paste to connect the silicon substrate and metal supporter to obtain the Ohmic contact.

⁸ The illustration of the extremely low noise level and no detectable RC discharge process can be found in our supplementary data file (available at stacks.iop.org/Nano/22/105704/mmedia).

Here F , K_c , and d are respectively the force applied to the nanostructure, the cantilever spring constant, and the deflection of the cantilever. The mechanical properties of the nanostructure can be characterized by measuring its deflection according to the slope variation of the AFM force curve [6–8]. Finally, the strain induces an electric field inside the nanostructure that produces a piezoelectric potential.

The electric potential is maximal at the surface of the nanowire: positive at the tensile side and negative at the compressive side [1, 2, 9, 10]. The relationship between the force applied to the nanowire and the measured potential can thus be established. After force curve measurement, a tapping mode scan is performed to make sure that the tested nanowires were not broken during the manipulation. Importantly, the applied stress can be controlled well by two AFM force curve parameters: Z -start position, Z ramp size and Z -velocity. Z -start position and Z ramp size are employed to control the maximum value of the applied force and Z -velocity is used to control the force duration.

3. Results and discussion

Having observed the horizontal deformation measurement method [1] in contact mode, we intend to improve our investigation method by considering the following possibilities:

- The manipulation process could be divided into two steps: an imaging step and a manipulation step to know the object situation before and after manipulation.
- The tip-sample force should be strictly controlled by the AFM closed-loop feedback system.

- We attempt to establish a convenient and precise control process for the application of the external force to avoid too many random tip-sample collisions during the manipulation.
- Using the slope variation of the AFM cantilever deflection, more information could possibly be obtained to completely analyze the piezoelectric properties of the nanostructure.

To understand the electrical behavior of the GaN nanowires, the tip-nanowire contact should be analyzed. When the nanowire is deformed, the tip contacts its stretched side, where the tensile strain generates a positive electric potential [1, 2, 9, 10]. The electron affinity of GaN is 4.1 eV and its band gap is 3.39 eV [11]. The AFM tip is heavily n-type doped and coated with a Pt-Ir alloy. The exact value of the Pt-Ir work function is unknown but can be estimated to be of the order of 5.5 eV [12]. Therefore, we should have a Schottky contact for intrinsic GaN nanowires and for n-type GaN nanowires [12]⁹. Consequently, on the stretched side of the nanowire, the Pt-Ir and n-type GaN interface may act as a reversed-biased Schottky diode, and little current can pass when no strain is applied [1, 2, 11, 12]. However, in our experimental configuration, we believe that the preamplifier with low noise level and without detectable RC discharge effect (less than ± 0.4 mV) can help us to overcome this limitation (see footnote 8).

Also, as mentioned previously, the duration or the velocity (AFM force operation parameter) of the force applied can be controlled well. Under the impact of gradually increasing force, the conduction band edge of the GaN nanowire may be changed due to the combined results of the high location piezoelectric potential and of the mechanical stress [13–15]. The shift of the conduction band and nano-contact effect may effectively lower the Schottky barrier [13–17]. Accordingly, a positive spontaneous electric potential can be successfully detected and measured on the nanowire’s stretched side when the nanowire deflection (applied force is larger than 200 nN)¹⁰ and positive piezoelectric potential is large enough.

Due to the impact of the Schottky barrier and the contact resistance, the measured value is lower than the real value of the spontaneous electric potential at the surface of the nanowires. Since the Schottky barrier height depends on the metal coating of the tip, in our study, several metal coatings such as Pt-Cr, Pt-Ir, and doped diamond have been tested. The Pt-Ir/Pt-Cr coating can help us to measure the highest potential. All the experimental results presented in the following are obtained by using Pt-Ir/Pt-Cr coating tips (EFM conductive AFM probes from Nanoscience or Veeco).

⁹ The I - V measurement and the schematic energy-band diagram of tip-nanowire are shown in the supplementary data (available at stacks.iop.org/Nano/22/105704/mmedia).

¹⁰ We have found a threshold value of the applied force in the cut-off region when the piezoelectric potential can be measured. This value can be as large as 200 nN. We conducted many measurements from September 2009 to December 2010. All of our experimental results are consistent, reproducible and repeatable with or without an amplifier (in this case, no external voltage is applied in any stage of experiment). Please see the supplementary data (available at stacks.iop.org/Nano/22/105704/mmedia).

The force applied to a single nanowire and the electric potential that is obtained via the piezoelectric effect are plotted as a function of time. As shown in figures 3(A) and (B), the electric potential decreases monotonically as the applied force decreases for both types of GaN nanowire samples (in other words the potential is an increasing function of force). Indeed, under a dynamic force impact, the nanowire is deformed. The potential is induced by the deformation and is proportional to it. In figures 3(C) and (D) the rate of increase and decrease of the force are progressively diminished. These results indicate the long lifetime, good stability and high reproducibility of the self-generated piezoelectric potential. Under the same experimental condition (such as under the impact of the same force, the same forcing location and the same AFM tip), we found that the measured electric potential of intrinsic GaN nanowires is 1.7×10^{-3} V ($F = 250$ nN) and that of n-GaN/AlN/GaN nanostructures is 2.0×10^{-1} V ($F = 250$ nN), as shown in figures 4(A) and (B). These results demonstrate that n-doped GaN/AlN/GaN nanostructures might have better piezoelectric properties than intrinsic GaN nanowires.

Our method exhibits a good reliability during piezoelectric measurement on the basis of all these experimental observations (see footnote 10). Firstly, the topographic image and the auxiliary mode image (such as the phase mode image) provide information about the nanostructure such as its dimensions and its position before and after the ‘force curve’ manipulation. Importantly, these images permit us to choose a relevant nanowire to measure. Moreover, because the tip-sample interaction force is less than 1nN in the tapping mode, the nanowires cannot be damaged during the scan step. Secondly, switching to the ‘force curve’ measurement mode, the applied force, the application time and the forcing location can be precisely controlled, altered and known, as can be seen in figures 3 and 4. Finally, the force curves on different forcing locations can be used to calculate the nanowire deflection, which can help us to study the mechanical properties of the nanostructure being measured (figure 5).

To analyze the piezoelectric properties of the nanostructures, the relation between the force and the spontaneous electric potential should be understood. First of all, we define the function Λ as follows:

$$\Lambda(\kappa_0, \kappa_{\perp}, e_{15}, e_{31}, e_{33}, \nu) = \frac{1}{\kappa_0 + \kappa_{\perp}} [e_{33} - 2(1 + \nu)e_{15} - 2\nu e_{31}]. \quad (2)$$

According to [1] and [2], we have equation (3)

$$\varphi = \frac{1}{\pi} \Lambda \frac{F}{Er} \quad (3)$$

where φ is the maximum spontaneous electric potential at the nanowire surface, ε_{\max} is the maximum mechanical strain in the nanowire, κ_0 and κ_{\perp} are dielectric constants (their values can be found in [9]), the values of the piezoelectric constants of the intrinsic GaN film: e_{33} , e_{31} and e_{15} are respectively 0.73 C m^{-2} , -0.49 C m^{-2} and -0.49 C m^{-2} [18]. ν is the Poisson ratio. E is the Young modulus and r is the radius of the nanowire.

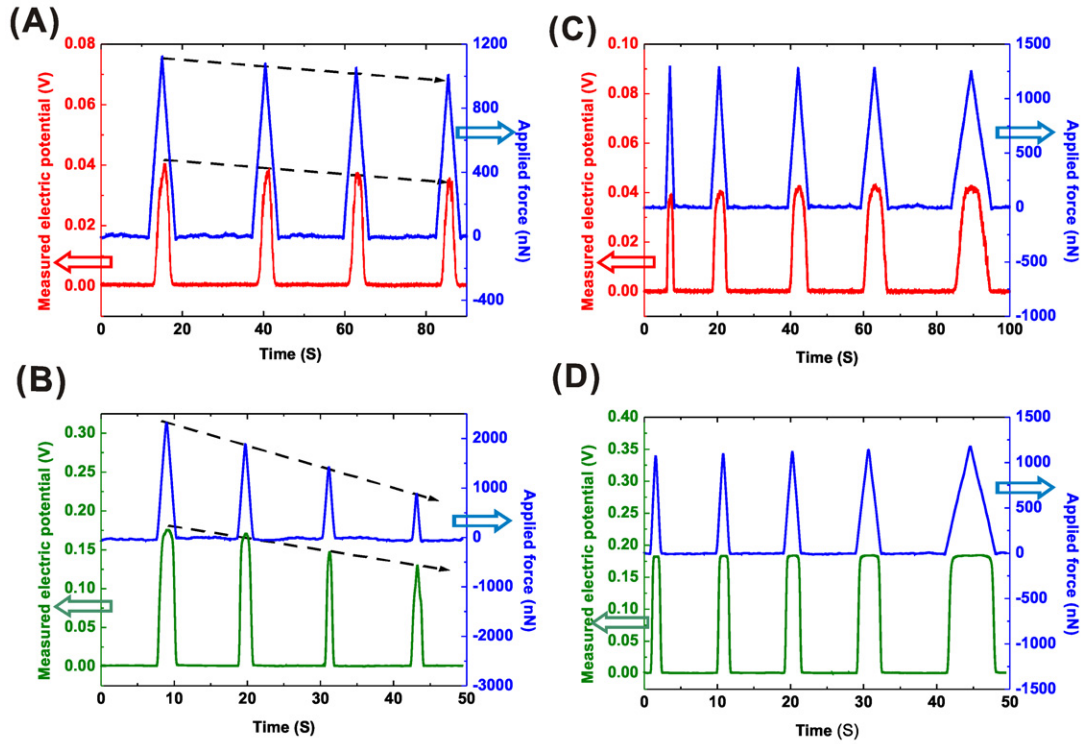


Figure 3. Spontaneous electric potential measurement: (A) on GaN nanowire (B) on n-doped GaN/AlN nanowire, the spontaneous electric potential decreases with decreasing applied force. Period control of spontaneous electric potential: (C) GaN nanowire measurement, (the force curve scan rate is decreasing: 0.5, 0.4, 0.3, 0.2 and 0.1 Hz); (D) GaN/AlN nanowire measurement (the force curve scan rate is decreasing: 0.5, 0.4, 0.3, 0.2 and 0.1 Hz).

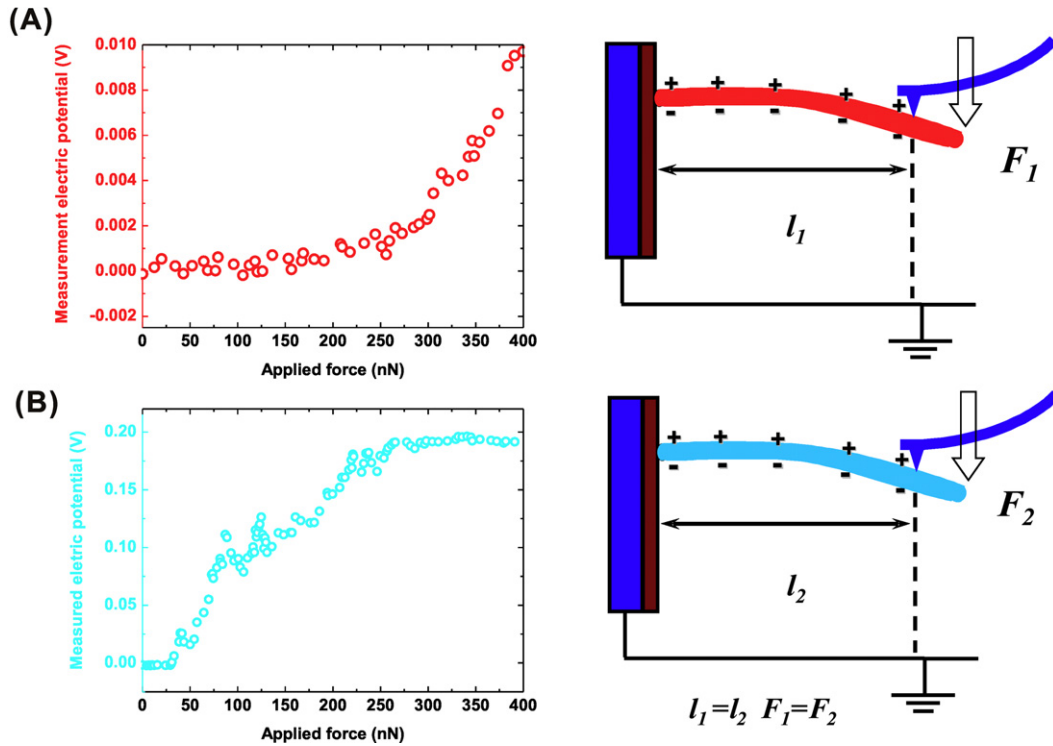


Figure 4. Measured electric potential of (A) intrinsic GaN nanowire; (B) n-doped GaN/AlN/GaN nanostructure as a function of applied force under the same experimental conditions.

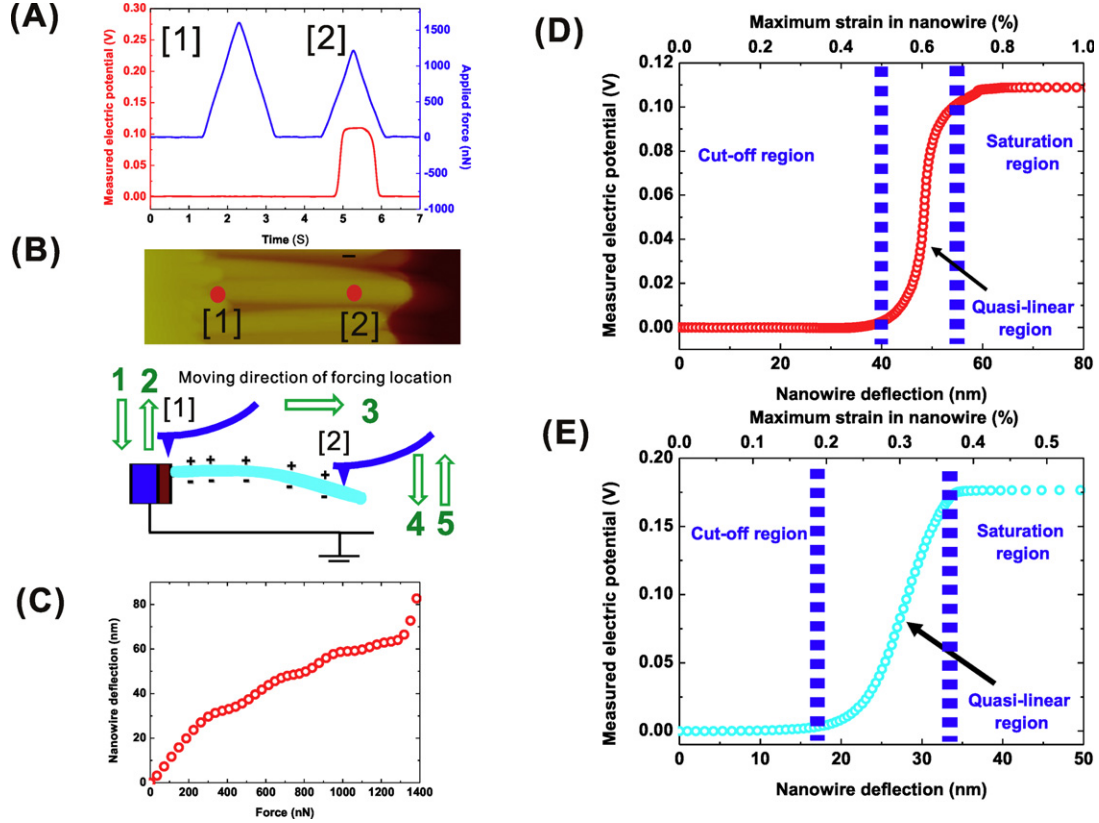


Figure 5. (A) Force curve measurement at different forcing locations on a GaN intrinsic nanowire; (B) the force curve manipulation process and forcing location illustrated on a nanowire; (C) the deflections of one GaN intrinsic nanowire as a function of the applied force; (D) corresponding measured electric potential as a function of the GaN intrinsic nanowire deflection at the forcing location [2]; (E) measured electric potential as a function of the GaN/AlN/GaN nanostructure deflection.

The spontaneous electric potential can also be expressed as a function of the nanowire deflection, and given by equation (4)

$$\varphi = \frac{3}{4} \Lambda \frac{r^3}{l^3} m_{\max} \quad (4)$$

where l is the distance between the base of the nanowire and the forcing location. The variable m_{\max} represents the maximal deflection of the nanowire where the force is applied.

We set and use the same ‘force curve’ parameters for all forcing locations, such as Z-start position, Z-ramp size and Z-velocity, to apply a force to the nanostructure. Following the system instructions we have configured, the AFM tip begins to approach the sample surface from an original pre-position. Once the tip touches the sample surface, a force curve manipulation can be applied by the AFM tip at the forcing location 1 on the substrate. In such a case, the slope of the cantilever deflection is maximal compared with the other forcing location. After finishing the first manipulation, the AFM control system will move the tip from the first pre-position to a new one. During this procedure, the tip will move at a safe height without touching the selected nanowire. The AFM control system repeats the same force curve operation at forcing location 2 (shown in figure 5(B)). Due to the nanowire deflection, the slope of the cantilever deflection is reduced. As stated previously, in this way the nanowire deflection can be

calculated by using the slope variation of the force curve on different forcing locations, as shown in figures 5(A)–(C).

Figures 5(D) and (E) show the relation between the measured electric potential as a function of nanowire deflection. The potential-deflection curve can be distinctly divided into three regions as follows: cut-off, quasi-linear and saturation regions. In the cut-off region, because of the Schottky barrier restriction and the bad contact induced by the weak force at the beginning of manipulation, we cannot measure the electric potential even if the nanowire becomes deformed. The nanowire deflection is too small to change the band structure in this region. As can be seen in figure 5(D), the cut-off region extends to 40 nm nanowire deflection (the applied force should be 200 nN at least for an intrinsic GaN nanowire (see footnote 10)). With the gradual increase of nanowire deflection, the mechanical stress becomes large enough to decrease the Schottky barrier height or eliminate this barrier. Consequently, the self-generated electric potential can be measured and is found to increase proportionally to the nanowire deflection, as described by equation (3). This is the quasi-linear region, for the range of nanowire deflection from 40 to 55 nm. Finally, when the nanowire deflection becomes very large, the measured electric potential is saturated because the induced internal electric field cannot continue to increase. We think that the reason for this is probably the saturated

rotation of electric dipoles [19]¹¹. Shown in figure 5(D), is the piezoelectric potential saturation region when the nanowire deflection is larger than 55 nm.

Furthermore, as can be seen in figure 5(E), the three regions stated previously can also be clearly identified on the deflection-potential curve of the n-doped GaN/AlN/GaN nanostructure. In comparison with the intrinsic GaN nanowire, n-type doping cannot avoid the Schottky barrier restriction either. Therefore, a n-doped GaN/AlN/GaN nanostructure also needs an initial deflection to minimize the impact of the Schottky barrier.

Additionally, we compared the measured values of spontaneous electric potential with the theoretical values. Substituting the piezoelectric constants of intrinsic GaN film into equation (3), the spontaneous electric potential can be calculated as a function of nanowire deflection. For a nanowire deflection of 55 nm, the calculated value of the induced electric potential is around 7.6×10^{-3} V. This value is almost 15 times lower than the measured electric potential (1.1×10^{-1} V, induced by a 55 nm deflection) of a GaN intrinsic nanowire ($l = 598$ nm $r = 15$ nm), as shown in figure 5(D). On the other hand, for a nanowire deflection of 35 nm, the theoretical value of the electric potential is 4×10^{-3} V. The corresponding electric potential (induced by 35 nm nanowire deflection) measured for a n-doped GaN/AlN/GaN nanostructure ($l = 639$ nm, $r = 15$ nm) can be as high as 1.8×10^{-1} V, as shown in figure 5(E). This result is almost 45 times larger than the theoretical prediction.

According to our experimental results and equation (3), the drastic increase of the function Λ at the nanoscale could be a possible cause of: (I) the difference between calculation and experiment in these two cases; (II) the difference between the electric potential saturated value in GaN intrinsic nanowires and in GaN/AlN/GaN nanostructures.

The function Λ is proportional to the piezoelectric constant (e) and Poisson modulus (ν), and is inversely proportional to the dielectric constants (κ_0, κ_{\perp}). Although further investigation is needed for detailed analysis, we estimate that the reason, i.e. the significant increase of the function Λ , seems to be: (I) the intrinsic GaN piezoelectric constants or dielectric constant could exhibit a strong size-dependent behavior. The size effect and free boundary [20] might result in the significant improvement of the piezoelectric properties¹² or the reduction of dielectric constant. This result agrees with our experimental observations (see footnote 12) and the conclusion of [21]. (II) The n-type doping could cause a decrease of the dielectric constant, an increase of piezoelectric constant or possibly change the sensitivity of the photoelectric effect of the n-doped GaN nanowire¹³.

¹¹ Please see the figure in the supplementary data (available at stacks.iop.org/Nano/22/105704/mmedia). If the applied force is less than 1000 nN, we do not find a saturation region for intrinsic GaN nanowire. Consequently, we suspect that, under the impact of large force, the saturated rotation of electric dipoles induces the piezoelectric potential saturation.

¹² The piezo-potential of the nanowire is inversely proportional to the cube of its length-to-diameter ratio. The size effect measurement of GaN and ZnO nanowire can be found in the supplementary data files (available at stacks.iop.org/Nano/22/105704/mmedia).

¹³ The doping effect is still under further investigation. However, for GaN nanowires, n-type doping cannot improve the photo-voltage effect. Please see our supplementary data (available at stacks.iop.org/Nano/22/105704/mmedia).

Accordingly, a higher electric potential can be measured on the n-doped GaN/AlN/GaN nanostructure compared with intrinsic GaN nanowire.

With the consideration of all these experimental results, a number of competitive advantages of our method may be summarized as follows:

- (1) This method allows more reliable and repeatable potential measurements because the image scanning and piezoelectric measurement steps are independent.
- (2) It is possible to select, localize, and observe a tested nanowire by relying on topographic tapping mode scans before and after the force curve manipulation.
- (3) We can accurately control the mechanical stress applied to the nanowire and precisely measure the nanowire's deformation and the induced potential according to the force curve. Thus the relationship between the spontaneous electric potential of the nanowire and the applied stress can be investigated.
- (4) Our method can help us to overcome the Schottky-barrier-limited behavior and is sufficient to allow the measurement of a positive self-generated electric potential at the stretched side of the nanostructure.

To successfully measure the spontaneous electric potential of the piezoelectric nanostructure, we think that two critical factors should be carefully analyzed: the type of tip–nanowire contact, whether Ohmic or Schottky contact; and the value of nanowire deflection.

In the case of Ohmic contact, it seems easy to measure the self-generated electric potential on the compressive side even if the nanowire deflection is small, such as the experimental results of [1]. On the other hand, owing to the Schottky barrier, the piezo-charges can accumulate at the stretched side of the nanowire [1–4]. Once this barrier is lowered by the combined effects of the piezoelectric potential and strain in the nanowire, it is also possible to successfully measure a positive piezoelectric potential in the case of Schottky contact.

4. Conclusion

We develop an innovative AFM method with precise control of the applied force, the forcing location and the nanowire deflection to investigate the piezoelectric properties of free-standing nanostructures. By using our improved investigation method, we demonstrate the relation between the measured piezoelectric potential and nanowire deflection. We discover the following phenomena: the Schottky barrier can be considerably lowered or totally eliminated by tensile mechanical stress and a positive piezoelectric potential; the measured electric potential is saturated under the impact of a large nanowire deflection, higher saturated piezoelectric potentials are found in comparison to theoretical calculations; the maximum electric potential of intrinsic GaN nanowires is different from that of n-doped GaN–AlN–GaN nanostructures. The causes of the saturation of the piezoelectric potential and its difference in both types of GaN nanowire are still under further investigations and will be discussed in the future.

Our experimental results are exceptionally useful to thoroughly examine and understand the piezoelectric properties of nanostructures. The piezoelectric properties of nanostructures, which are introduced in detail in this paper, represent a promising approach for harvesting mechanical energy and further reveal the great potential to fabricate cost-effective nano-generators or highly sensitive self-powered NEMS sensors.

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