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HIGH FREQUENCY ELASTIC PROPERTIES OF NITRIDE NANOWIRES-BASED STRUCTURES

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Abstract. The present research aims at studying the elastic properties of III-Nitride nanowires (NWs) embedded in HSQ matrix, capable to efficiently convert the mechanical energy into electricity. We report on elastic properties of such novel nano-engineered functional materials studied by using “transient grating” acoustic experiment.

1. Introduction

Flexible piezo-electric nanowires are today a topic of intense research, motivated by their numerous economically relevant applications (e.g., rollable displays, wearable intelligent electronics, piezo-generators, and so forth). Nitride nanowires show remarkable mechanical and optoelectronic properties stemming from their high aspect-ratio. They are mechanically flexible and can stand high deformations without plastic relaxation. Thanks to this promising performance, polymer-embedded nanowires offer an elegant solution to create a piezo-generator material, which converses the environment energy to electric one.

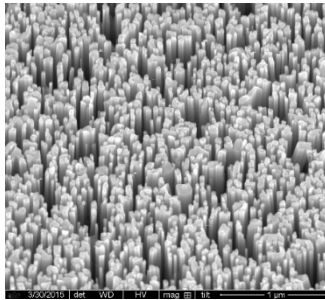
A large number of works are devoted to study optoelectronic properties, there have been few research works on elastic properties of nanowires. A few researches are mainly focused on the study of mechanic-electric energy conversion at kHz domain. The most of environment vibrations covers a kHz scale frequency spectrum, and the transformation of ambient kHz noise into the electric power is a promising way to create any self-power micro-devices. Nevertheless, related on the nanosize, the mechanical resonances are expected at frequencies within a range stretching from a few hundreds of MHz up to several GHz.

The current work proposes to explore ultrasonic elastic properties of nitride nanowires at MHz and GHz frequency range. The interest of this research is fundamental: the mechanical behavior of materials at the nanoscale is different from that at the macroscopic scale due to the increasing ratio of the surface to the volume. The systematic study of elastic properties leads to an accurate understanding of the deformation of nanowires allowing thereby for the prediction of the piezoelectric behavior, the enhancement of the energy conversion and in return to an improved design of the nanowires-bases structures.



2. Growth of GaN nanowires.

Self-assembled GaN NWs were grown by Plasma-Assisted Molecular Beam Epitaxy (PA-MBE) on a thin n-doped AlN buffer layer [1] deposited on n-type doped Si (111) substrate (the NW morphology is illustrated in Fig. 1).



The growth was performed at 760°C, under N-rich conditions. NWs have a cylindrical shape with a hexagonal cross section delimited by {10-10} planes [2]. They are characterized by height and diameter of the order of $1 \mu\text{m} \pm 120 \text{ nm}$ and $45 \text{ nm} \pm 20 \text{ nm}$, respectively and by a density of the order of 10^8 NWs/cm².

Figure 1. Carpet of Nitride Nanowires (SEM photo).

3. Fabrication and characterization of elastic properties of piezogenerators.

3.1. Fabrication.

The sample consists in piezoelectric GaN nanowires grown on a thick silicon substrate. The NW arrays are encapsulated into spin-on glass (HSQ), which is annealed at 400°C. Then the NW tops are uncovered by doing reactive ion etching and the Schottky contact is deposited, though a shadow-mask, on the NW top side. Finally, an ohmic contact is directly deposited on the substrate (Figure 3).

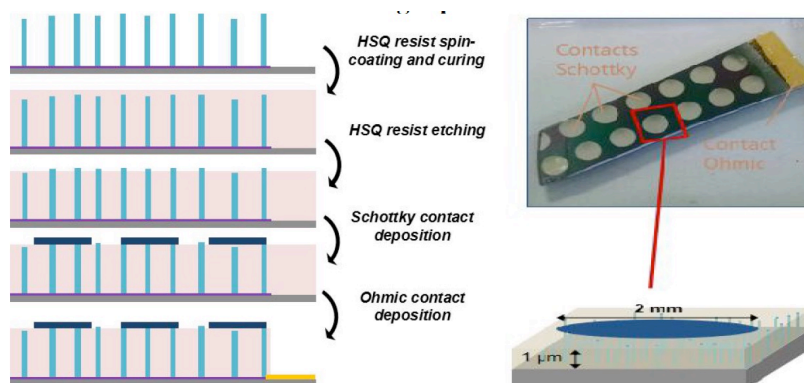


Figure 2. Process steps to realize the piezogenerator (left) and the picture of sample (right).

3.2. Experimental set-up.

Experimental techniques well suited to measure the sound velocity in composite systems, and in turn the dispersion, are not that many. When the device includes piezoelectric elements, electromechanical transducers and network analyzers can be used for characterization but with the disadvantage of a poor tuning of the frequency. The non-contact technique that we describe here allows for characterizing the elastic properties of almost any kind of materials, transparent or opaque, ordered or disordered, piezoelectric or not... in a frequency range that stretches from a few tens of MHz up to several GHz.

Basically, the so-called Transient Grating Method [3-5] is a four waves mixing technique which consists in exciting the sample into vibration by illuminating its surface with two IR light pulses ($\lambda_{\text{exc}}=1064\text{nm}$; 30ps in duration). These IR light pulses interfere to produce interference fringes with a period of Λ . As a consequence of photoelastic processes, standing elastic waves with wavelength of Λ and angular

frequency of Ω are thus created within the illuminated area. The wavelength Λ can be easily tuned by setting the angle between the two incident beams.

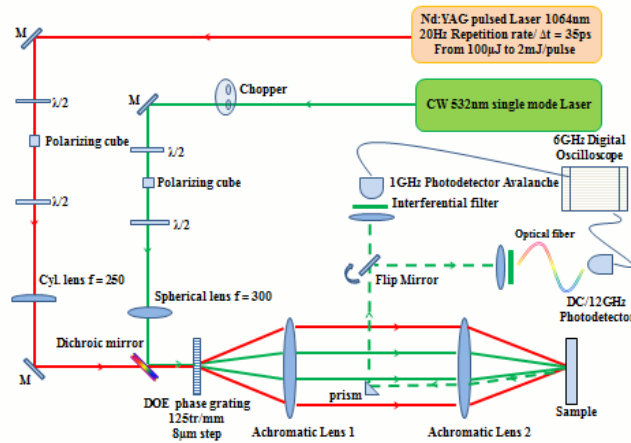


Figure 3. Scheme of the experimental setup. M and DOE stand for Mirror and Diffractive Optical Element respectively, $\lambda/2$ is for half wave plate.

The detection of the elastic waves is made using a heterodyne scheme. This involves two continuous laser beams (respectively the probe and the reference beam) both incident on the sample with an optical angular frequency ω . The probe beam gets diffracted upon reflection on the sample, giving rise to diffraction orders ± 1 at optical angular frequencies $\omega \pm \Omega$. The beam at $\omega + \Omega$ is further mixed with the specularly reflected reference beam, whereas the beam at $\omega - \Omega$ is ignored. It is then straightforward to show that a signal proportional to $\cos(\Omega t)$ results from the mixing of both first order diffracted probe beam and specularly reflected reference beam. This signal is recorded with a fast photodiode connected to a broadband oscilloscope and further processed using conventional FFT techniques.

3.3. Characterization of elastic properties

We show in Fig. 4 (left panel) a typical signal recorded on the sample. The spectrum of the signal obtained by Fourier transform is shown in inset.

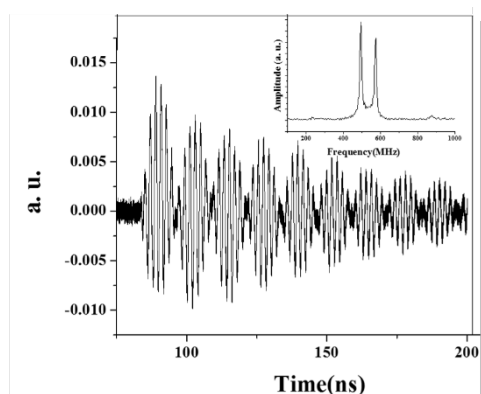


Figure 4. typical signal recorded in the sample shown in the Figure 1. The wave vector is set to $k=2\pi/\Lambda=1.07\mu\text{m}^{-1}$ ($L=5.85\mu\text{m}$). Inset: Fourier transform of the data.

The frequency spectrum features two peaks at 495 and 575MHz respectively. They correspond to the first two Rayleigh modes (the higher order Rayleigh modes are sometimes called Sezawa modes; they require a minimum layer thickness to propagate). The effective phase velocities V of these modes can be very simply deduced from the measured frequencies ν through $V = \Lambda \nu$. Regarding the dispersion, it may be recorded by tuning Λ owing to a proper choice of the angle between the two incident optical pulses. As an example of the achievements of this experimental technique, we show in Fig. 5 the dispersion for three carpets of nanowires similar to the one shown in Fig. 2 but with different curing temperatures).

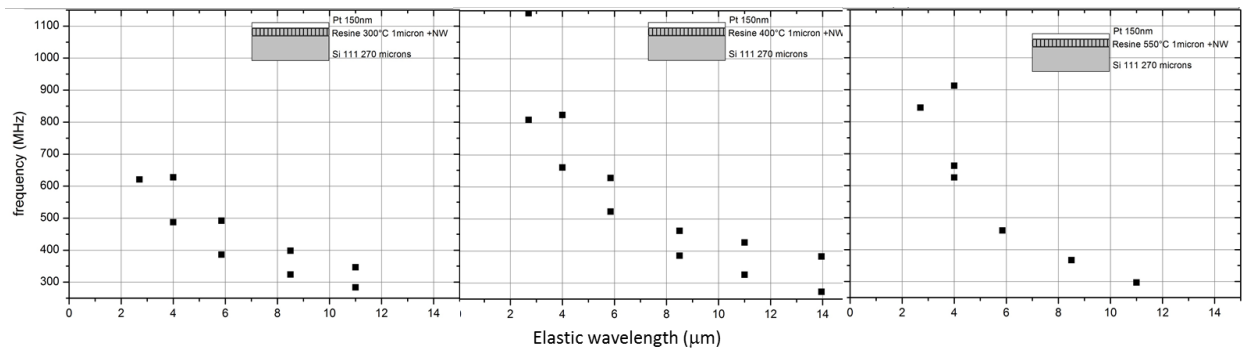


Figure 5. Dispersion of the first two Rayleigh modes propagating in layers of nanowires deposited on a silicon substrate. The samples differ each other by the annealed temperature (300, 400 and 550°C).

The stress of HSQ matrix increases with increasing curing temperature. Furthermore, HSQ may have become SiO₂ based on the change in the chemical structure and larger increase in modulus and hardness when cured above 650°C. Here, the interest is in keeping the nanowires flexible within the HSQ matrix, thus, temperatures below 650° are chosen.

Analysing these dispersion curves, one sees that with increasing temperature, the velocity of Rayleigh waves also increase, and the rapid change of frequencies from wavelength in MHz diapason allows the elastic constants to be determined with accuracy.

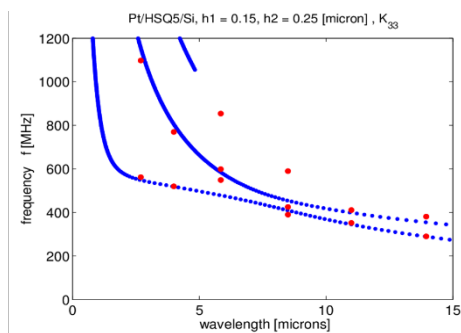


Figure 6. Dispersion curves of Pt/HSQ/Si structure: theoretically predicted (blue line); experimental results (red points).

In order to identify all modes and to determine elastic constants, theoretical investigations are carried out for a three-layers elastic half-space. Figure 6 represents analytical behaviour of dispersion curves (blue lines) and experimental data for one sample cured at 400°C (red points). The theoretical curves obtained from the cross-section of the $|K_{33}(\lambda, f)|$ surface along vertical lines $\lambda = \text{constant}$ in the range from 0 to 15 μm [6]. The elastic constant C_{11} , C_{44} as well as velocities in all layers can be deduced directly from comparison between the theory and experiment.

4. Conclusion.

Beside the dispersion properties of the nanostructures, it is worth noting that this experimental technique allows for a precise determination of the effective elastic parameters of such a layer on a substrate. It should also be noted that this non-destructive contactless technique is efficient to investigate the effective elastic properties at very high frequency (several GHz), along any directions of an anisotropic sample as the phononic crystals and the elastic metamaterials are.

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