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Correlation between Electrical Transport and Nanoscale Strain in InAs/In$_{0.6}$Ga$_{0.4}$As Core–Shell Nanowires

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Supporting Information

ABSTRACT: Free-standing semiconductor nanowires constitute an ideal material system for the direct manipulation of electrical and optical properties by strain engineering. In this study, we present a direct quantitative correlation between electrical conductivity and nanoscale lattice strain of individual InAs nanowires passivated with a thin epitaxial In$_{0.6}$Ga$_{0.4}$As shell. With an in situ electron microscopy electromechanical testing technique, we show that the piezoresistive response of the nanowires is greatly enhanced compared to bulk InAs, and that uniaxial elastic strain leads to increased conductivity, which can be explained by a strain-induced reduction in the band gap. In addition, we observe inhomogeneity in strain distribution, which could have a reverse effect on the conductivity by increasing the scattering of charge carriers. These results provide a direct correlation of nanoscale mechanical strain and electrical transport properties in free-standing nanostructures.

KEYWORDS: InAs nanowire, strain mapping, piezoresistance, transmission electron microscopy

Nanoscale III–V semiconductor materials are essential components in electronic and optical devices such as high-speed transistors, photovoltaics, and photodetectors.$^{1–9}$ In contrast to bulk materials, nanocrystals (such as nanowires) offer new possibilities for strain engineering by the growth of heteroepitaxial layers or by mechanical manipulation. The quasi-1D geometry combined with the high crystal quality of the nanowires often enables the nanowires to withstand more mechanical strain compared with their bulk counterparts. Significant electromechanical responses have been found in various semiconductor nanowires, although the origin of the profound electromechanical effects is still under debate.$^{10–18}$ Among the semiconductor nanowires studied, InAs nanowires possess some unique properties such as strong spin–orbital coupling, a relatively narrow band gap, and a high electron mobility.$^{7,19–21}$ Previously, considerable piezoresistive and piezoelectric effects in InAs nanowires have been reported.$^{11,22}$ Surface effects, such as surface-charge accumulation and ionized surface states, have been found to strongly affect the electrical and photonic properties as well as the electromechanical properties of bare InAs nanowires.$^{23–25}$ The change in the density of accumulated surface charge with strain was considered the main reason for the strong electromechanical response of bare InAs nanowires.$^{11}$ Nonetheless, electromechanical characteristics of InAs nanowires with suppressed surface effects have not yet been studied. Especially, studies of the correlation between mechanical properties and the electrical transport properties of single InAs nanowires with reduced surface effects are still lacking.
In this paper, we have carried out such studies and show that there is a direct correlation between electrical transport and the nanoscale lattice strain of single crystalline InAs nanowires. The InAs nanowires were passivated with a thin coherently strained In$_{0.6}$Ga$_{0.4}$As radial shell to reduce surface effects. Electrical conductivity and nanoscale mechanical strain measurements of the nanowires were performed simultaneously with an electromechanical in situ transmission electron microscopy (TEM) set up. In situ straining, mechanical measurement, and electrical characterization were enabled by an in situ TEM holder. Quantitative nanoscale strain analysis on the nanowire was carried out using scanning TEM (STEM) combined with nanobeam electron diffraction (NBED). By straining the crystal lattice, the charge carrier transport is altered by modifications in the band structure and charge carrier scattering characteristics engendering an intriguing insight into the electromechanical behavior of InAs nanowires.

The surface of the InAs nanowires used in this study is passivated with a thin In$_{0.6}$Ga$_{0.4}$As shell, as presented in Figure 1a,b. The nanowire morphology appears uniform. The nanowire has a diameter of about 84 nm and is approximately 10 μm long. The nanowires show single crystalline wurtzite structure in both the core and the shell (Figure 1). Radial growth of a larger band gap material (∼0.68 eV for bulk In$_{0.6}$Ga$_{0.4}$As) compared to the core (∼0.35 eV for bulk InAs) is used here as an effective method to reduce surface charge accumulation and ionized states for InAs nanowires. In general, a considerable lattice mismatch between the core and shell materials may result in inhomogeneities at the interface, such as misfit dislocations or change of crystal structure, which will increase charge carrier scattering and reduce the charge carrier mobility. The nominal lattice mismatch between bulk wurtzite InAs and In$_{0.6}$Ga$_{0.4}$As is about 2.7% (see Supporting Information S1). Here, we have a core-shell nanowire structure, which will affect the distributions and magnitudes of lattice strain in the InAs core (diameter ∼80 nm) and in the In$_{0.6}$Ga$_{0.4}$As shell (∼4 nm). A detailed strain analysis near the core-shell interface of the nanowire is shown in Figure S4, A thorough inspection revealed no dislocations along the nanowire length (see Figures 1 and S2). Selected area electron diffraction (SAED) analysis also confirmed the absence of stacking faults and dislocations (Supporting Information S3).

The core and shell appear to be coherently strained, with the shell fully passivating the InAs core surface. To investigate the correlation between mechanical strain and the electrical transport properties of such surface-passivated InAs nanowires, a Hysitron P19S nanoindentor holder with an electrical push-to-pull (EPTP) microelectromechanical system (MEMS) device was used for the in situ TEM studies; see the Methods section and Supporting Information for a more detailed description. The nanowires were placed across the gap between a movable and a fixed part in the EPTP device and connected to an electrical circuit (see Figure 2a). Tensile stress was applied on the nanowires by a nanoindentor through the push-to-pull mechanism. Direct and quantitative measurements of the stress, strain, and electrical conductivity of single nanowires were performed simultaneously, while the nanowires were pulled in tension using the EPTP device.

The tensile stress applied on the nanowire was determined from the in situ TEM measurements as described in the following. The force applied to the nanowire (Figure 2c) was determined by the difference in load applied to the MEMS device with and without the nanowire (Figure 2b). The force-displacement curves before and after the fracture of the nanowire are both linear, indicating the elastic deformation of the EPTP device and the nanowire as well as a brittle fracture of the InAs nanowire. The nanowire fractured when the total load applied on the EPTP MEMS device was around 110 μN. A combination of scanning electron microscopy (SEM) and TEM was used to determine the original diameter and length (between the two electrical contacts) as well as the changes in dimensions of the nanowire as a function of the applied load. As a result, the stress applied on the nanowire was determined based on the force and geometry measurements.

The in situ quantitative measurements of lattice strain and nanoscale strain distribution were carried out by STEM-NBED strain mapping. STEM-NBED has been demonstrated to be a reliable method for measuring mechanical strain in crystalline materials with high spatial resolution, a large field of view, and high precision. The electron beam diameter was about 2 nm with a convergence angle of ∼2 mrad. The electron beam was scanned over an area of 2 μm × 140 nm with a step size of about 4 nm, and a NBED pattern was acquired at each beam position. The NBED patterns were acquired with the electron beam aligned along the [1−100] zone axis of InAs (Supporting Information S8). At each beam position, axial and radial strain in the nanowire was determined by measuring the displacements of the (0002) and (11−20) diffraction spots in the NBED pattern relative to those when no strain was applied on the nanowire. By measuring lattice strain at every beam position, the spatial strain distribution in the nanowire was obtained and presented as strain map for the different stress loads applied (Figure 3a,b). In situ STEM-NBED measurements were performed while the nanowire was under stress. Strain maps corresponding to different tensile stresses applied on the nanowire were then calculated. The detailed spatial strain distributions along the (0002) (defined as the x axis) and the (11−20) directions (y axis) were obtained and plotted as $\varepsilon_{xx}$ and $\varepsilon_{yy}$ respectively (Figure 3a,b). The rotation of the crystal lattices was also measured based on the intensity distribution in the diffraction patterns of the NBED maps. In general, the lattice structure of the InAs nanowires in this study shows very little rotation (less than 1°), even at the highest stress level (Figure 3c).
Figure 2. EPTP MEMS device and mechanical measurement on single core-shell InAs nanowire in the in situ TEM instrument. (a) Top: an SEM image of the EPTP device with a core-shell InAs nanowire mounted across the gap between the mobile and fixed parts of the device. The EPTP device is made of Si with four Au electrodes deposited on it. A schematic of the electrical circuit is also shown. Bottom: a magnified image of the area marked by the dashed window in the upper image. The ion beam induced deposition (IBID) of Pt (yellow) on the EPTP device (blue) was used to connect the Au electrodes and establish electrical contact. The nanowire (green) is fixed on the IBID Pt stripes by electron beam induced deposition (EBID) of Pt (orange). The gray part is the gap between the fixed and mobile parts and allows for the TEM investigation of the nanowire during in situ TEM measurements. (b) Force—displacement curves of the EPTP MEMS device obtained before (brown) and after (black) the fracture of the nanowire. The inset shows a SEM image of the fractured nanowire. (c) Force—displacement curve of the nanowire deduced from panel b.

The axial $\varepsilon_{xx}$ maps provide detailed and quantitative information about the spatial distribution of strain along the axial direction of the nanowire and also the evolution of the axial strain resulting from external tensile stresses (Figure 3a). When there is no stress applied, the mean tensile strain in the nanowire core is around zero, although in some small areas, there is either tensile or compressive strain (the first strain map from the top in Figure 3a). This local strain in the nanowire without any externally applied stress is likely due to the nanowire transfer process when a small amount of stress may be introduced in the nanowire during the mechanical fixing of the nanowire onto the EPTP device. As the applied load from the nanoindenter increases, strain distribution within the nanowire evolves gradually. As shown by the axial strain maps ($\varepsilon_{xx}$ Figure 3a), the tensile strain seems to initially emerge in localized areas close to the surfaces of the nanowire. Subsequently, more and more areas within the nanowire show tensile strain as the applied stress increases, while the rest of the nanowire area is still not strained. When the force applied on the nanowire reaches $\sim$7.79 $\mu$N, the entire nanowire seems to be homogeneously strained, as observed from the even contrast in the $\varepsilon_{xx}$ map. When the force applied on the nanowire is larger than 9.43 $\mu$N, a region of the nanowire close to the center always shows lower tensile strain compared to rest of the nanowire. The $\varepsilon_{xx}$ maps show spatial radial and axial inhomogeneity in strain distribution (Figure 3a). Moreover, the average strain value ($\langle \varepsilon_{xx} \rangle$) over the whole strain map is calculated. The average strain $\langle \varepsilon_{xx} \rangle$ increases with force, reaching values of 0.187%, 0.466%, and 0.837% at 1.19, 2.84, and 4.49 $\mu$N, respectively. When the nanowire experiences a tensile strain along the axial direction, the lattice in the nanowire is expected to be elastically compressed in the radial direction due to the positive Poisson’s ratio of these materials. The spatial distribution of the compressive strains within the nanowire ($\varepsilon_{yy}$) under different stresses also shows complex behaviors. As the applied stress increases, compressive strain emerges initially in some localized areas in the nanowire (Figure 3b). Thereafter, more and more regions in the nanowire show compressive lattice strain though there are always areas in the nanowire in which the lattice is not strained. From these quantitative spatial strain distribution maps, the corresponding probability histograms are calculated and displayed in Figure 3d. The lattice strain along both the $x$ and $y$ directions is inhomogeneous within the nanowire due to the inherent lattice strain existing in the nanowire due to the core-shell structure. Surface roughness at the nanometer scale may also contribute to the observed strain distributions. Further studies are needed to fully understand the cause for the strain inhomogeneity at the nanometer scale in the nanowire.

Nonetheless, strain analyses based on STEM—NBED maps allow a direct quantification of the elastic strain within the nanowire. The average strain values ($\langle \varepsilon_{xx} \rangle$ and $\langle \varepsilon_{yy} \rangle$) were calculated from the strain maps and considered as the elastic strain of the whole nanowire. In such a way, we directly determined the elastic strain within the nanowire. The Poisson’s ratio of the InAs/In$_{0.6}$Ga$_{0.4}$As core–shell nanowire was also calculated, based on the $\langle \varepsilon_{xx} \rangle$ and $\langle \varepsilon_{yy} \rangle$ values, to be $\sim$0.36. Moreover, strain mapping also clearly and quantita-
tively shows the spatial fluctuations of lattice deformation within the nanowire at the nanometer scale for different stress levels. Interestingly, despite those inhomogeneous strain distributions, the nanowire as a whole seems to deform elastically under tensile stress before fracture, as indicated by the linear characteristics of force−displacement curve (Figure 2b,c). Our strain analyses also unravel the evolution of lattice strain within the nanowire, containing rich information about the creation and development of lattice strain in such a nanosized crystal under external mechanical stimuli.

To correlate electrical transport properties with the stress and the strain characteristics, simultaneous transport measurements were conducted during the in situ TEM experiments. All current−voltage \((I−V)\) characteristics measured for nanowires with electrical contacts at both ends show a linear behavior, thus confirming Ohmic contact behavior (Figure 4a). \(I−V\) measurements were also performed for larger voltage range than that shown in Figure 4a (Supporting Information S9). The \(I−V\) characteristics at a larger voltage range show the same linear behavior as those in smaller voltage range. To avoid the overheating of the nanowire, the effect of stress on the \(I−V\) characteristics was measured within the relatively small applied voltage range, as shown in Figure 4a. It should also be noted that the contacts between the nanowire and the EPTP device remain mechanically stable based on the reproducible STEM, load−displacement, and \(I−V\) measurements (see Supporting Information S10). Figure 4b shows STEM micrographs of a nanowire under different tensile stresses. Piezoelectric effects in InAs nanowires have previously been reported and shown to result in asymmetric \(I−V\) characteristics.\(^{22}\) In contrast, our \(I−V\) curves are linear and symmetric, suggesting a minimal piezoelectric effect on the \(I−V\) measurements of the individual InAs nanowires. The slope of the \(I−V\) curves, i.e., the resistance of the nanowire, changes continuously with tensile stress (Figure 5a). At the maximum

Figure 3. Lattice strain distribution within the core-shell InAs nanowire under tensile stresses unveiled by STEM−NBED measurements. (a) Lattice strain distribution along the nanowire length direction with different tensile forces applied on the nanowire. (b) The corresponding strain distribution maps of the nanowire for strain along the direction perpendicular to the nanowire length direction. (c) The corresponding rotation angle maps of the nanowire. (d) Quantitative strain distribution histograms at different stress levels in the nanowire. The X axis shows stress applied on the nanowire. The Y axis is the lattice strain. The colors indicate the probability of certain strain existing in the strain maps, with red showing higher probabilities than blue.

Figure 4. Effect of tensile stress on the electrical transport properties of the core-shell InAs nanowire. (a) Current−voltage characteristics of the nanowire when different forces were applied on the nanowire. Arrows indicate the direction of the increase of force. (b) The corresponding STEM ADF images of the nanowire under the absence of stress and for different tensile stresses.
where $\rho_0$ is the resistivity of the nanowire under zero stress, $\sigma$ is the stress applied on the nanowire, and $\Delta \rho$ is the change in resistivity.

The InAs/In$_{0.6}$Ga$_{0.4}$As nanowire shows a nonlinear piezoresistive response. Figure 5b clearly shows the nonlinear piezoresistance characteristics of the $\Delta \rho/\rho_0$ curve, which is different from the electromechanical behavior of bare InAs nanowire without the shell (Supporting Information S12). The nonlinearity in piezoresistance effect is believed to be a result of the nonparabolic shape of the energy bands in nanowires.\textsuperscript{10-12} Due to the obvious nonlinearity in piezoresistance, the piezoresistance effect in the core–shell InAs nanowire can be described as:\textsuperscript{32}

$$
\frac{\Delta \rho}{\rho} = \pi_1^{i} \sigma + \pi_2^{i} \sigma^2 + \pi_3^{i} \sigma^3 + \cdots
$$

where $\pi_i$ is the $i$-th longitudinal piezoresistance coefficient. By fitting the nonlinear piezoresistance effect to the $\Delta \rho/\rho_0 - \sigma$ data with a fourth-order polynomial, the piezoresistance coefficients of the InAs/In$_{0.6}$Ga$_{0.4}$As nanowire from first- to fourth-order are found to be $\pi_1^{i} = -3.4 \times 10^{-9}$ Pa$^{-1}$, $\pi_2^{i} = 1.9 \times 10^{-18}$ Pa$^{-1}$, $\pi_3^{i} = 19.3 \times 10^{-27}$ Pa$^{-1}$, and $\pi_4^{i} = -33.5 \times 10^{-36}$ Pa$^{-1}$; see the red curve in Figure 5b.

The first-order piezoresistance coefficient of the InAs/In$_{0.6}$Ga$_{0.4}$As core–shell InAs nanowire is compared to the piezoresistance coefficient obtained in bulk and other semiconductor nanowires. We found that $\pi_1^{i}$ of the core–shell InAs nanowire used in this study is 2 orders of magnitude larger than that of InAs bulk material, which has a piezoresistance coefficient of $\approx 5 \times 10^{-11}$ Pa$^{-1}$.\textsuperscript{13} It is also about an order of magnitude larger than the first-order piezoresistance coefficient of InAsP and InAs nanowires reported previously.\textsuperscript{12,22}

It is worth noting that the resistance obtained from our in situ $I$–$V$ measurements consists of contributions from the nanowire itself and the contact resistance at the two electrical contacts between the nanowire and the EPTP device. The contact resistance is insignificant compared to the resistance of the nanowire due to two reasons. First, it is known that InAs easily forms Ohmic contacts with various metals because of its small band gap and Fermi-level pinning at the surface.\textsuperscript{34} As a result, the Schottky barrier and contact resistance at the contacts between the nanowire and EPTP device are expected to be small. This is evident in the linear $I$–$V$ characteristics of the nanowire used in our study. Second, the resistance value at zero stress in our measurement is comparable with previously reported four-probe measurements on InAs nanowires.\textsuperscript{31} It also indicates that the contact resistance is not dominating in our measurements. Moreover, according to our repeated $I$–$V$ measurements, TEM imaging, and strain measurements, the contacts between the nanowire and the EPTP device are stable under applied mechanical stresses. Thus, the strain induced resistance changes are not likely due to the changes in contact conditions. The resistance changes we observed come from the nanowire.

There are several effects that the In$_{0.6}$Ga$_{0.4}$As shell may have on the charge transport in InAs nanowire. The shell has a larger band gap than the InAs core. It has been shown previously that such a shell will largely reduce surface band bending and charge accumulation.\textsuperscript{26,55} Thus, the charge carriers will populate the core instead of the InAs nanowire surface as in bare InAs nanowire. In addition, InAs has a larger electron mobility compared to In$_{0.6}$Ga$_{0.4}$As,\textsuperscript{3} resulting in preferred charge transport in the InAs core in the core–shell

Figure 5. Electromechanical properties of the core-shell InAs nanowire: (a) Resistance as a function of tensile stress and average tensile strain ($\varepsilon_{xx}$), (b) The relative change in resistivity of the nanowire as a function of tensile stress and average tensile strain ($\varepsilon_{xx}$). Red curve shows a polynomial fitting to the experimental data for extracting the piezoresistance coefficients of the nanowire.
Strain can also affect carrier transport through strain induced band structure variation. Strain can cause a band-warping effect, which can be understood by symmetry considerations. Energy bands follow the symmetry of the Brillouin zone, which is determined by the macroscopic crystal symmetry. Uniaxial mechanical stress reduces the crystal symmetry, so the shape of the energy bands change accordingly, causing band warping. Under effective mass approximation, band warping will change the carrier masses at both the conduction-band minimum and the valence-band maximum in InAs. In addition, the aforementioned band splitting in valence band will also change carrier mass due to the separation of HH and LH bands. However, the change in effective carrier mass in III–V semiconductors due to strain-induced band splitting and warping is not significant and usually smaller than 5%. Moreover, among the scattering mechanisms that affect the scattering rate of charge carriers, phonon scattering is normally considered to be dominating in semiconductors at room temperature. However, because the conduction band of InAs is singly degenerated, the scattering rate of conduction electrons in InAs is not affected significantly by strain.

Uniaxial tensile stress results in the strong inhomogeneity of lattice strain distribution in the core–shell InAs nanowire, as observed in the STEM–NBED measurements (Figure 3). As discussed above, such fluctuations in strain distribution can also affect charge transport and mobility in the nanowire. The local variation in strain can result in changes in the electronic band structure, which will induce local changes in the carrier concentration and the effective mass of the conduction electrons. In particular, the spatial variation in band gap at the nanometer scale within the nanowire will cause energy barriers for the transport of conduction electrons. Those barriers will increase the electron scattering and consequently decrease the mobility of the charge carriers. The distribution of the energy barriers is directly related to the extent of spatial inhomogeneity of strain. Strong strain inhomogeneity can give rise to confined channels for transport of charge carriers. The spatially confined charge transport will also increase the resistivity of the whole nanowire, which reduces the effect of carrier concentration change on the piezoresistance effect of the nanowire.

In summary, we have studied the intrinsic mechanical and electromechanical properties of individual InAs nanowires. Mechanical stress, lattice strain, and electrical transport characteristics of an InAs/In0.6Ga0.4As core–shell nanowire were measured simultaneously using an electromechanical in situ TEM set up. In particular, the elastic lattice strain in the nanowire was directly and quantitatively determined by STEM–NBED mapping with nanometer resolution, while mechanical stress and electrical transport properties were measured simultaneously. The resistivity of the nanowires shows a sensitive response to external tensile stress. The electromechanical properties of the core–shell nanowire differ from the bare InAs nanowire. The piezoresistance coefficient of the core–shell nanowire was found to be about 2 orders of magnitude larger than bulk InAs and also larger than the bare InAs nanowire. Furthermore, the change in resistivity in the core–shell nanowire can be largely attributed to a reduction in band gap that increases the conduction electron concentration in InAs. We also observed a nanoscale inhomogeneity in the strain distributions of the core–shell nanowire using STEM–NBED strain mapping. While an increasing overall strain increases carrier concentration, local inhomogeneity in strain
distribution can have a reversed effect through the introduction of local scattering sites. Our study shows the unique mechanical and electromechanical properties of surface passivated InAs nanowires. The properties may be utilized in applications such as ultrasensitive nanoscale sensors.

**Methods. Nanowire Growth.** The core–shell nanowires used in this study are grown in a molecular beam epitaxy system on InAs(111)B substrates in a two-step process. First approximately 10 μm long Au catalyzed InAs nanowires are grown along the [0001] direction via the vapor–liquid–solid method at a substrate temperature $T_{\text{sub}} \approx 420 \, ^\circ\text{C}$ with an In flux corresponding to a planar growth rate of 0.5 μm/h and an effective As$_2$-to-In flux ratio of 20. These growth conditions yield a pure wurtzite (WZ) crystal structure with no observed stacking faults. Subsequently, a shell of InGaAs is grown with a $T_{\text{sub}} = 360 \, ^\circ\text{C}$ and a Ga flux corresponding to a planar growth rate of 0.33 μm/h, keeping As$_2$ flux constant.

**Nanowire Transfer.** For the in situ TEM studies, individual nanowires were lifted out and mounted on EPTP device in an FEI Strata focus ion beam — scanning electron microscope (FIB-SEM) instrument. The nanowires were put across the gap with a width of about 2.5 μm between a movable and a fixed part of the EPTP device. This gap allows for the TEM investigation of the nanowires. The nanowire was connected to the electrical contacts on the EPTP device with electron beam induced deposition (EBID) and ion beam induced deposition (IBID) of Pt in FIB-SEM (Supporting Information S5 and S6).

**TEM.** High-resolution STEM imaging was carried out using the TEAM 1 microscope operated at 300 kV. A Gatan K2 direct electron detector camera attached to the TEAM 1 microscope was used to record the NBED maps. In the STEM–NBED measurements performed in this study, the electron beam was focused to around 2 nm in diameter with a convergent angle of ~2 mrad. The electron beam was scanned over an area with the size of $2 \mu\text{m} \times 140 \mu\text{m}$ with a step size of about 4 nm. At each beam position, a NBED pattern was acquired with an exposure time of 2.5 ms with the K2 camera. Each diffraction pattern was obtained with the full frame of the camera, giving rise to 1920 × 1792 pixels in each diffraction pattern. The lattice distances and, hence, the lattice strain were measured directly and accurately based on the distances between the Bragg spots in NBED patterns. The in situ tensile test and electrical transport properties measurement were performed using a Hysitron P95 nanoindenter holder with an EPTP MEMS device. The EPTP device contains a moveable part and a fixed part, which are connected by springs; see Supporting Information S5. The electrical transport properties measurements were performed by sweeping the voltage applied on the nanowire from ~0.5 to 0.5 V and measuring the current going through the nanowire. The electric current limit in the $I$–$V$ measurements was set to 100 nA to minimize the Joule heating effect. No changes in crystal structure and electrical transport properties of the nanowire due to the illumination of the electron beam in TEM were observed.

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