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Temperature driven three-dimensional ordering of InGaAs/GaAs quantum dot superlattices grown under As₂ gas flux

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Abstract
A comprehensive microscopy analysis has been undertaken to study three-dimensional quantum dot (QD) ordering in multilayered In₀.₄Ga₀.₆As/GaAs structures grown with an As₂ flux at different substrate temperatures. Atomic force microscopy, transmission electron microscopy, and photoluminescence measurements were employed to fully understand the formation of these extended dot structures. Changes in the lateral pattern of QD ordering are correlated with their vertical alignment. These correlations are analyzed in light of the inherent transformation of the wetting and spacer layers, as well as changes in the shape, strain, and composition of individual QDs. The experimental results are attributed to the anisotropy in the thermally activated surface mass transport and the relaxation of elastic stresses.

Introduction
Versatile control of QD-based semiconductor nanostructures parameters forms the basis of modern semiconductor technologies. A variety of novel devices have been suggested where 0-dimentional structures have been exploited. Lower lasing thresholds and improved device performance have been achieved using high-density QD arrays as the active material in laser diodes and vertical cavity surface emitting lasers. InAs quantum dots in GaAs barriers were the first intermediate band single-junction solar cell prototypes which allowed for more efficient structures due to the inherent tunability of their band alignments from changes in shape, size, and quantum confinement properties [1–4]. High detectivities to infrared radiation, reduced dark current noise, and near room temperature operation are some advantages of the near-, mid- and far-infrared photodetectors based on intersubband transitions in quantum dot superlattices. The direct practical application of InGaAs/GaAs quantum dot superlattices investigated in this work is a photodetectors for visible to mid-infrared detection.

However, in order to maximize the potential application of these types of structures, three-dimensionally ordered arrays of uniform QDs are necessary. Strain-driven self-assembly has been developed into an almost ubiquitous fabrication process for these nanostructures and is well described by the classical Stranski–Krastanov growth model [5–7]. However, the growth of more complex systems, with multiple components or where the elastic constants or diffusion rates become anisotropic, requires a more elaborate model for description. In the case of multilayer structures, the strain distribution in the barrier layers plays an important role in the nucleation of QDs. At the same time, segregation towards and along the surface during capping as well as roughening of the surface must be taken into account (see for example Refs. [8–12]). The regularities of vertical QD alignment in multilayer structures have been established both theoretically and experimentally to depend on the degree of crystal anisotropy [11,13,14], the degree of mechanical inter-layer coupling governed by the thickness of the spacer layers [15–17], and the crystallographic orientation of the growth surface [18,19]. While these effects have all been studied in QD ensembles, there are also reported studies of the local structure of individual QDs by high-resolution transmission electron microscopy (HRTEM) [20,21]. However, to bring all of these results to a common basis and predict the QD evolution in anisotropic multi-component structures is very difficult because of the uniqueness of each case studied.

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In this paper, the three-dimensional QD ordering has been studied in In_{0.4}Ga_{0.6}As/GaAs multilayered structures grown by molecular beam epitaxy (MBE) at different growth temperatures. Changes in the lateral and vertical ordering of the QDs are correlated with transformations in both the wetting and spacer layers, changes in shape, degree of deformation, and composition of individual QDs. This allows us to obtain relationships between the main processes involved in the formation of 3D ordered multilayer nanostructures without having to make assumptions about the influence of any of the significant factors.

**Experimental details**

QD superlattices were grown by MBE on semi-insulating GaAs (0 0 1) substrates using an As$_2$ flux. First, the native oxide layer was removed from the substrate and a 300 nm GaAs buffer was grown at 580 °C. Then, the temperature was reduced to the superlattice growth values for each sample and a 15 period multilayer structure was grown composed of nine monolayers (MLs) of In$_{0.4}$Ga$_{0.6}$As separated by 60 ML GaAs spacers. The superlattice growth temperatures were 510, 520, 530, 540 and 555 °C. As$_2$ was used as the group V source and the V/III ratio was kept at ~15:1. In this regime the growth is highly group III limited. This procedure resulted in growth rates of 0.24 ML/s for GaAs and 0.27 ML/s for InAs. For each sample, the superlattice structure was completed with an uncapped layer of InGaAs QDs. Atomic force microscopy (AFM) was then capable of characterizing the morphology of the buried QDs with the assumption that after the first few QD layers, subsequent layers are similar. The AFM images on these uncapped QDs, were performed using a NanoScope IIIa Dimension 3000TM scanning probe microscope. In order to minimize tip-effects, the AFM was operated in tapping mode. In addition, high-resolution silicon tips were used with the apexes being well characterized with a calibration grating [22], before and after measurements.

Cross-sectional TEM samples were prepared for structural analysis. The samples were first glued face to face, with the faces rotated from one another by 90°, allowing us to take images along and across the dot chains. The electron transparent areas were obtained by standard mechanical polishing followed by low-energy ion-milling using Fischione equipment. The analysis was carried out using a FEI Tecnai TF20 FEG Transmission Electron Microscope fitted with a Gatan post-column imaging filter (Gatan Quantum 963). For the HRTEM images, the samples were oriented along the [0 1 1] and [0 − 1 1] zone axis (along and across the dot chains) and bright or dark field images were recorded using two-beam conditions with (0 0 2) reflecting planes for diffraction contrast.

Elemental mapping for In and Ga was achieved using electron energy loss spectroscopy (EELS) in scanning mode at 120 kV. Scanning TEM (STEM) dark-field images were recorded using a Fischione HAADF detector. An EELS spectrum was obtained for each pixel of the HAADF image. The InN2.3 and GaM2.3 peaks at 443 eV and 103 eV, respectively, were integrated using the Gatan Digital Micrograph package. Finally, a power-law approximation was used to extract the signal from the background contributions.

The PL measurements were performed in a variable temperature, 10−300 K, closed-cycle helium cryostat using the 532 nm line from a doubled Nd:YAG laser for excitation. The laser spot diameter was ~20 μm with power kept low enough to avoid excitation of higher order states in the QDs. The PL signal was dispersed by a 0.5 m single-grating monochromator and detected by a liquid-nitrogen-cooled OMA V: InGaAs photodiode detector array.

**Results and discussion**

**Complementary microscopies of samples**

Figs. 1 and 2 show TEM images of the prepared samples and the AFM images of the top dot layer of each sample grown at different temperatures, respectively. As expected, the size, density and the nature of the vertical and planar QD ordering depend strongly on the growth temperature chosen. All TEM images of Fig. 1 were obtained under 2-beam condition with (0 0 2) reflecting planes to enhance the contrast between the dots and surrounding GaAs matrix. All samples show good crystalline quality and extended defects were detected using diffraction contrast imaging with several reflections. Figs. 1(a) and 2(a) present the overall structure for the sample grown at the lowest temperature, 510 °C. It is rather difficult to differentiate one dot from another in the TEM image of Fig. 1(a), because the dot-separation is small compared to the average height. At the same time the organization of the dots in-plane (see Fig. 2(a)) is random. As a result, the overall structure appears to be simply a stacking of corrugated layers, because the electron beam travels through many dots contained within the sample thickness (~250 nm). However, growth at higher temperatures resulted in the formation of larger dots with larger interdot separation distances. The dots can be clearly seen in the TEM images of Fig. 1(c)−(e), for samples grown above 520 °C. A striking feature observed in these images is the quality and the nature of the vertical

![Fig. 1. Panel (a)](image-url) Conventional single-beam dark field TEM image obtained with a strongly excited (002) beam for the 15 layers QD stacking of the sample grown at 510 °C. Panels (b)−(e): bright field TEM images, taken under two-beam condition with (002) reflection for samples grown at 520 °C, 530 °C, 540 °C and 555 °C. The lines with arrows indicate the inclination of the QD stacking columns as induced by different growth temperatures.
alignment of the dots which becomes more pronounced at higher temperatures.

The vertical ordering of the QDs can be examined using TEM images collected from pairs of slices in the (0 1 1) and (0 1 1) planes. The inclination of vertical columns of QDs is clearly visible in the TEM images for samples grown at 530 °C and 540 °C as shown in Fig. 1(c) and (d), respectively. The vertical stacking is irregular for the sample grown at 520 °C since the column inclination changes from place to place (not shown here) with a maximum of ~7 degrees. It is more pronounced and more regular for the 540 °C sample where it reaches ~15 degrees. In the perpendicular plane (0 1 1), the inclinations are somewhat smaller in both samples and the dot columns are sometimes terminated before the surface layer, i.e., not extending through the entire stack of layers in the sample (not shown here). The samples grown at 555 °C did not show any inclination of the ordering in any direction except within a few small and isolated areas where it may reach a maximum of 2 degrees. TEM observations at many locations confirm that these vertical QD correlations are typical for the entire volume of superlattice.

A more detailed statistical analysis of the HRTEM and AFM images shown in Fig. 3 allows us to establish a number of non-trivial features related to the formation of these QD superlattice structures. First, we will consider the lateral ordering of QDs along the [0 –1 1] and [0 1 1] directions, as shown in Fig. 2. It has been discussed in the literature [23,24] that the anisotropy in the surface diffusion rates on the GaAs (1 0 0), which results from the (2 × 4) surface reconstruction, allows for a longer diffusion length along the [0 –1 1] direction than the [0 1 1] direction. This leads to a more efficient relaxation of mechanical stress along [0 –1 1] which at the same time encourages alignment of the nucleating dots in this direction. As the number of superlattice layers increases, the anisotropy of the deformation field begins to follow the surface diffusion, thus enhancing the lateral organization within each layer. However, it requires an optimum growth condition to realize the highest degree of ordering. As can be seen from Fig. 2, temperatures below 530 °C do not provide enough thermal energy to realize the full anisotropy of the growth surface. As a result, highly ordered dots never appear even after many layers. Clear chains of QDs are formed for growths performed above 530 °C, whereas at the highest temperatures the prevailing parallel chain arrangement of QDs is subtly transformed into a pattern with hexagonal symmetry. Fig. 3(c)–(e) show typical examples of autocorrelation analysis of the AFM images of the surface QDs from our multilayered structures. It can be seen that apart from the direction of most probable QD ordering (the brighter spots in the [0 –1 1] direction), there is clearly found another slightly less probable direction of ordering at an angle of about ±24° from [0 1 1] (see Fig. 3(c)). This angle decreases slightly with temperature from 24 to 21 degrees. If we examine the threshold temperature for ordering along these two directions, we find very different values for each (see Fig. 3(a), curves 1 and 2). For deposition at 510 °C we find that the most probable distance between QDs in both
dot size covers the full range of observed sizes of the other samples, however the average value of the dot size actually decreases to ~10 nm.

By analyzing the geometrical characteristics of the buried QDs, their dimensions are in good agreement with AFM images of the uncapped topmost layers (Fig. 2). However, it should be noted that the QD size decreases with increasing depth of the buried layer within the structure and the rate of this change appears to increase with increasing temperature. This is particularly evident in the temperature range from 530 °C to 555 °C where the dimensions of the QDs in the upper buried layers exceed, by 20% to 40% respectively, the dot sizes in the first layers of the structure.

The origin of dot ordering

The formation of dots through strain relaxation is closely related to diffusional mass transport on the surface. Anisotropy in this surface diffusion and elastic deformations in the crystal lattice are particularly important processes that lead to the arrangement of these QDs into ordered arrays. In general, the QD growth mode is complex and very sensitive to conditions. Apart from the temperature, other critical growth parameters are, for example, the type of As species (As$_2$ or As$_4$), the ratio between group III to group V elements, the growth interruption time, the composition of constituents, the thickness of the spacer layer, etc. Nevertheless, it is possible to identify some general patterns having a direct bearing on the effects observed. Growth of coherent islands is self-limited due to a strain barrier surrounding the QD [28] and, for the growth process to proceed; the adatoms must overcome this barrier in order to diffuse into the islands. The energy barrier increases with the island size and, thus, fewer adatoms are able to overcome the barrier. Therefore, at low temperatures the QD growth mode is consistent with the classical Stranlki–Krastanov mechanism. But, alloying generally occurs at higher temperatures due to extensive material transfer into the QD from the wetting layer and from the substrate [29]. For the superlattice growth, some QD size reduction and In diffusion from the wetting into the spacer layers are often possible during spacer layer deposition. These effects lead to a change in the size and shape of the buried QDs and to the formation of a 1D wetting layer which connects neighboring dots within a chain during the GaAs capping as the result of anisotropic In transport from the top to the edge of existing quantum dots [30]. In the InGaAs/GaAs system, an increase of In concentration is always observed from bottom to top and from the sides to the center, which is accompanied by an increase in the local lattice parameter. The tendency to accumulate In atoms at the QD apex is interpreted as a way to minimize the strain during dot formation, while the most Ga-rich areas occurring near the lower edge of the dots are able to better accommodate the GaAs substrate lattice parameter [20].

The observed changes in the surface QDs seen in Fig. 4 agree well with the general trends outlined above. At the lowest temperature, 510 °C, the QDs are small (see Figs. 3(a) and 4(a)) with a narrow size distribution, and STEM composition maps (EELS) show the presence of an almost continuous InAs wetting layer at the interface of each layer of the superlattice. In cross section, the dots appear only as a local increase of the In concentration, as can be seen in Fig. 5(a). For the sample deposited at 555 °C, the QDs are quite large and are obviously seen in the XTEM image of Fig. 5(b). At the same time, for growth at this high temperature, there is no observable WL between the dots. Comparing the BF XTEM image (intensity contours in Fig. 5(b)) with the In composition map we see a typical increasing trend of In concentration, from the edge to the center as expected for InGaAs QDs.

HRTEM images were used to conduct semi-quantitative analysis of the vertical strain, $\varepsilon_{||01}$, for superlattices obtained at different temperatures. The method is based upon a direct measurement of...
the distance between atomic columns in the images that represent corresponding crystallographic planes. Since epitaxial layers are coherent and there is no relaxation by dislocation generation, the lattice constants in the interface of layers are the same. The built-in strain distorts the lattice in the [0 0 1] direction according to the Poisson's ratio. This value of $\Delta d/d$ is shown in cross-section in Fig. 6. To avoid instrumental artifacts, the reference crystallographic inter-plane distance, $d$, was chosen in the unstrained GaAs spacer layer. Images of areas with uniform thickness of TEM samples only were considered. Relaxation effects due to the thinning of samples were not taken into account. From these deformation maps shown in Fig. 6 we see, in the color scale, that the strain level in the sample grown at the lowest temperature, 510°C, is almost three times higher than that found in the sample grown at 555°C. However, locally we see both tension, $\varepsilon_{001} > 0$, and compression, $\varepsilon_{001} < 0$, in the 555°C sample, whereas in the 510°C sample, the tensile strain appears to be accumulating throughout the layers. The large size and alignment of dots in the 555°C sample are such that they relax the overall strain and even stretch the lattice in the plane, thus causing compression along the growth direction. At the same time we see in both samples that the areas of highest tensile strain are at the top of the dots, which is

Fig. 5. STEM composition maps (electron energy loss spectroscopy) of the samples grown at 510°C (panel a) and at 555°C (panel b). From left to right the images are as follows: the HRTEM, the Ga composition map, the In composition map, and the In composition map overlaid with the intensity isolines from the corresponding HRTEM images at the left.

Fig. 6. Typical bright field HRTEM images of QDs, taken under multi-beam condition close to the [1 1 0] direction (left) and corresponding growth direction strain, $\varepsilon_{001}$, maps (right) for samples grown at (a) 510°C and (b) 555°C.
consistent with the observed distribution of In in the QD seen in Fig. 5. This is especially true for the surface dots.

The unusual compression seen in the sample grown at 555 °C should be analyzed by looking at some transformation in the SL topology revealed by TEM. Here we find a significant reduction in the SL period of ~8.8%. This reveals the very significant impact of In desorption for growth above 540 °C affecting both the height of the QDs and as a result, the thickness of the GaAs directly over each dot. It is known [16,31] that the In desorption rate can be considered negligible only for growth temperature below 540 °C. Similarly, the thickness of the spacer layer directly above the QD is reduced by 17%, from 15.5 nm at 510 °C to 12.8 nm at 555 °C. As a consequence, the QD height decreases and the distribution of the QD volume increases, as seen in Fig. 4.

These transformations must necessarily affect the nature of the vertical correlation between dots which depends on the degree of influence that the strain field from the lower superlattice layers has on QD nucleation and on the growth of subsequent layers. As reported in [16], for the selected growth conditions, temperature 540 °C and As flux, as the spacer thickness increases the lateral distance between QDs increases and the ratio of interdot distance to the spacer thickness approaches 3.4 in the presence of an anisotropic strain field. This value is in good agreement with the prediction of Tersoff, Teichert, and Lagally using their simplified model [11]. In our case, the ratio of lateral distance between QDs to the spacer thickness is 3.4, 4.7 and 7.9 in samples grown at 510 °C, 540 °C and 555 °C, respectively. On the other hand Holý et al. [32] found that for the case of QD superlattice growth on GaAs (001), the vertical alignment angle of the QDs is 23° off normal toward the [1 1 0] and [–1 1 0] directions. This is due to the fact that the minima in the surface strain energies with buried QDs do not coincide with the position of the QD. Due to strain anisotropy they propagate at a certain angle with respect to the growth normal. Modeling for treating these propagation angles has been improved and predict much smaller off-normal angles. In particular, Meixner et al. [33] predicted a sharp transition between correlated and anti-correlated growth as a function of the spacer layer thickness separating the QD layers. The authors of Ref. [34] have reported the dependence of the relative QD position on the spacer thickness between adjacent layers for InGaAs QDs grown at 510 °C, for thicknesses between 75 ML and 112 ML. They found QDs vertically correlated for the smaller thickness and anti-correlated for the larger thicknesses. In our case, the increase in the tilting angle of vertically correlated QD columns, in SL samples grown at 530 °C and 540 °C, indicates an increase in the elastic deformation anisotropy which leads to vertical QD alignments displaying a more anti-correlated character. The increasing values of the QD aspect ratio, for these temperatures, indicate a significant enhancement of strain level as can be seen in Fig. 3(a), curve 3. At the highest temperature, 555 °C, the QD nucleation becomes again vertically correlated and the aspect ratio H/D of QDs reaches a minimum value.

We find, therefore that as the temperature increases its contribution to the processes of nucleation and growth of QDs is not monotonically changing. We look to the QD aspect ratio to explain this. In samples grown at 520 °C and 555 °C, the energy reduction associated to QD formation is minimal (see Fig. 3(a), curve 3) due to the large interdot distances. Also, strain relaxation resulting from the formation of the QDs is minimal and, therefore, the temperature dominates over strain effects in controlling the dot nucleation process. As a result, a qualitative change in the nature of the QD localization takes place. At 520 °C, the surface diffusion increases and results in a sharp increase in the distances between QDs along the direction 23° away from [0 1 1] (Fig. 3(a), curve 1). This naturally leads to an overall decrease in the level of strain but also an increase in anisotropy, because the diffusion barrier for transport in the [0 – 1 1] direction has not yet been exceeded. Further increase in the growth temperature enhances slightly the adatom surface diffusion in both directions leading to a small increase in the inter-dot distances (same slope of the curves 1 and 2 between 520 °C and 540 °C). At the same time, the degree of anisotropy of the QD surface distribution increases and the somewhat random dot distribution pattern transforms to clear chain-like structures with a maximum reduction of strain energy. This is also evident by the increasing tilt angle of the QD vertical alignment, as seen in Fig. 1(d). At 555 °C, the temperature finally exceeds the diffusion barrier in the [0 – 1 1] direction, thus contributing to a significant increase in the distance between QDs along this direction. This again leads to a drastic decrease in the level of strain and, moreover lowers the degree of anisotropy. The reduction in the overall level of strain appears to reduce the QD aspect ratio, and returns the vertical alignment to a fully correlated character as seen in Fig. 1(e). At the same time, the pattern of dot-chains on the surface transforms into a pattern with near hexagonal symmetry, as shown in Fig. 3(e). We recall that strain considerations alone [16], indicates a ratio between QD spacing to spacer thickness, in this sample, twice as large as what would be expected for formation of vertically correlated dot columns.

Finally, PL techniques are well suited to characterize the nature of the buried QD layers and how the growth temperature affects their structure. Fig. 7(a) shows the PL spectra of our samples collected at low temperature and low excitation power to avoid populating excited states of QDs. It is immediately evident that the change in QD size is represented here by the energy shift of the PL peak position. Since height is the smallest dimension in this type of QDs, it is the height that mostly affects the confinement and the energy of PL emission. There is a good agreement in trend between the PL peak position, which is plotted in Fig. 7(b) on the left axis, and the QD height, which is plotted in Fig. 4(a), except for the two lowest temperature samples. However, when the PL peak position is compared directly with the QD aspect ratio (Fig. 3(a), curve 3 and reproduced in Fig. 7(b), right axis) we find an obvious
anisocorrelation between these parameters. This is an indication that strain plays a significant role in the PL peak position as well. It is also obvious that PL shifts of the two samples that show one-dimensional ordering (QD chains) stand out from the rest of the samples. The 1D wetting layer that forms under the QD chain slightly increases the total height of the structure and therefore causes an additional shift of the PL peak [30].

The PL linewidth reflects the size (height) distribution of the QDs, assuming that the In composition is uniform inside the QD. There is a good agreement between the size distributions in Fig. 4(a) and PL linewidths in Fig. 7(a) except for the highest temperature sample. Despite the wide size distribution of the surface QDs grown at the 555 °C the linewidth of the PL peak for this sample is very narrow. This is likely due to the even In distribution within the large QDs and its localization predominantly in the upper part of QDs. In this case the PL behavior is equivalent to a reduction in size (height) of the QD structure.

Conclusions

We have investigated systematically the effects of different deposition temperatures on the three-dimensional alignment of self-assembled arrays of In₀.₃Ga₀.₇As QDs grown under As₂ flux with GaAs barriers. The threshold nature of the 3D QD ordering was established through cross-sectional TEM analysis correlated with AFM images of the final uncapped layer of QDs. The essential observations were taken in two different temperature ranges: 510–520 °C and 540–555 °C. In each of these temperatures ranges, we found a significant decrease in strain in the system with a sharp increase of the lateral distances between QDs in one of the characteristic [0 1 1] directions. Increased anisotropy in the strain of the system leads to the appearance of vertically aligned QDs exhibiting some anti-correlated character, i.e., aligned along a slope. At the same time, we detected a more pronounced in-plane ordering of the QDs along preferred directions. The temperature induced three dimensional reordering of the QDs is accompanied by transformations in their size and shape. Stranski–Kratonan self-limiting QD growth takes place within the growth temperatures of 510 °C and 520 °C. The volume of the QDs increases as the temperature reduces the strain energy due to increased inter-dot distance along [0 1 1] direction. Compared to those grown at 510 °C, the height of the QDs grown at 520 °C are very similar, but the volume increases due to an increase in diameter. This leads to a decrease in aspect ratio. Further increase in temperature allows more material to overcome the strain barrier around the QDs from both the wetting and spacer layers. Thus, the volumes and heights increase for QDs grown at 530, 540 °C. This further reduces the strain, but not enough to leave the dots vertically aligned. Thus there is still some inclination of the vertical QD stacking. The increase in the distribution of the geometrical parameters with temperatures is due the increasing In desorption rate and intermixing of the QD’s composition. It is shown that these changes are accompanied by: a transition from a nearly continuous wetting layer of InAs (at low temperatures) to large QDs (six-fold increase in QD volume at high temperatures); a change in the superlattice period; and a decrease of the thickness of the spacer overlaid QDs. Of particular note is the reduction in the SL period by ~9% for samples grown at 555 °C due to the increased In desorption resulting in a decrease of the thickness of the spacer over the QDs by 17%. These changes in growth and alignment of the QDs are reflected in the QD photoluminescence taken at low temperature and low excitation power. Finally, the observed regularities can be explained by the competing processes of anisotropic mass transport or diffusion in the plane of the growing film and the influence of strain distribution on the nucleation of 3D islands at the surface of the spacer layer. Elevated temperatures allow for adatoms finding a lowest energy nucleation site consistent with both the surface anisotropy and the residual fields from the previous QD layers. This provides a mechanism for controlling the 3D ordering in complex QD superlattice systems allowing for the design of samples with specific optical or transport or even mechanical properties.

Results obtained here have practical application for both multilayer structures with high-density QD arrays and structures with the low-density QDs that have potential to contribute to the fields of spintronics and quantum information processing. Novel devices, such as quantum light emitters, sources of entangled photons or few-to single-QD lasers, could be envisioned. In such devices detailed information about the QD’s local structure and how to control surface positioning is very important.

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