Focussed on "Crack Paths"

Visual Abstract

Experimental characterization at nanoscale of single crystal silicon fracture toughness

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ABSTRACT. The work reviews some preliminary recent micromechanical tests aimed at the evaluation of the fracture toughness of silicon. Pre-cracked nano specimens and alternatively notched nano specimens combined with the theory of critical distances (TCD) are compared. The results show that the fracture toughness of silicon is approximately 1 MPa·m$^{0.5}$, regardless of the procedure involved (i.e., pre-cracked samples or TCD). This value agrees with macro counterpart, i.e., 0.75-1.08 MPa·m$^{0.5}$, and therefore the K_{IC} is independent of the size and crystal orientation. However, by employing the TCD, the accurate control of the final crack tip which is currently very challenging, is overcome by using notched specimens. Additionally, the results give information about the crack propagation at the nanoscale. It seems that although the specimen axis deviates from the (011), the crack propagates along the cleavage plane (011) and the process develops very fast by breaking covalent bond at the crack tip. A brief discussion on beyond the breakdown of continuum theory and challenges toward nanometer scale fracture mechanics concludes the paper.

KEYWORDS. Fracture nanomechanics; Silicon; Nanoscale; Fracture toughness;

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INTRODUCTION

Conventional fracture mechanics [1] has been primarily investigated starting from the II World War and developed in the well-known linear elastic fracture mechanics (LEFM). However, in the last decade, new challenges have arisen. Indeed, the miniaturization of electronic devices driven by the increasing demand for high-density integrations have brought problems of material behavior at very small scales into the domain of the conventional fracture mechanics [2]. Moreover, recent literature suggests that in many applications at the macroscale, reliable engineering for entire component lifetime depends on accurate prediction of fracture from the smallest size until final failure [3]. While numerical simulations have spread rapidly [4], the small sizes of micro and nanocomponents still impose several challenges for the experimental study of their mechanical properties. The mechanical characterization at the micro and nanoscale (or even at atomic scale [5]) provides essential parameters for the design of components such as Micro/Nano-

Electro-Mechanical Systems (MEMS, NEMS), but gives us an insight into the breakdown of the continuum fracture mechanics. For these reasons, *fracture nanomechanics* has sparked the increasing interest of the scientific community. While effort has been primarily devoted to determining Young's modulus and tensile strength of silicon and other materials [6,7], the fracture properties and fracture process at the nanoscale have been marginally treated. Recent results suggest that at micro and nanoscale the material still fails by nucleation and propagation of cracks [2] and therefore some local approaches, commonly employed at the macroscale, can be directly scaled down [8]. However, there is a limit below which the continuum assumption of the LEFM has to face the discrete nature of atoms [9]. In the above background, the experimental characterization of fracture behavior at the nanoscale and the investigation of the low limit of the continuum theory are at the present relevant and challenging aspects.
Therefore, this contribution presents a synthesis of experimental tests on the evaluation of single crystal silicon fracture toughness at the nanoscale [10,11]. First, results obtained from pre-cracked samples are reviewed and crack propagation is commented. Later, results from notched nano-cantilever beams by using the theory of critical distances (TCD) are presented. At the end, the procedures are compared to highlight the pros and cons. The work also provides an insight into the micro-mechanisms of crack propagation in brittle materials.

FABRICATION OF THE SPECIMENS

The specimen fabrication is depicted in Fig. 1. The pre-cracked samples and the notched nano-cantilever beams have similar procedures of fabrication with substantial differences only in the final steps. First, a block is generally carved out from a single-crystal Si (100) plate. The block is then deposited on a flat end of a gold (Au) wire and stabilized by tungsten vapor deposition. In this phase, the orientation of the sample can be re-arranged if needed. The block is then processed by FIB (focused ion beam) to obtain the final specimen.
In the case of pre-cracked specimens, a trapezoidal plate is generated, and V-shape grooves are introduced in the vertical direction. These grooves are used as a guide for the generation of the pre-crack. A notch is introduced at the top surface, and a wedge-shaped indenter is employed to apply an opening displacement until crack propagation is detected. Finally, the top part of the specimen can be cut to obtain the desired crack-length. The process of pre-crack generation is in general quite difficult and time-consuming since precise control of the geometry with the FIB is problematic at this small scale. In the case of notched specimens, the cantilever beam is cut from the block in several steps, and a notch is finally introduced. In the generation of the notch, attention should be paid to obtain a good level of symmetry on the two lateral surfaces. This aim is achieved by tilting the stage to correct the beam angle. It should be pointed out that also in this case the control on the notch root radius is very difficult, especially when the generation of sharp notches is needed. Because of the extremely small sizes of the samples, even the smallest radius currently achievable with FIB could not be simplified and considered as "zero". The procedure is, however, faster than the fabrication of pre-cracked specimens and, as is shown in next sections, accurate control of the notch geometry is not needed when applying the TCD.

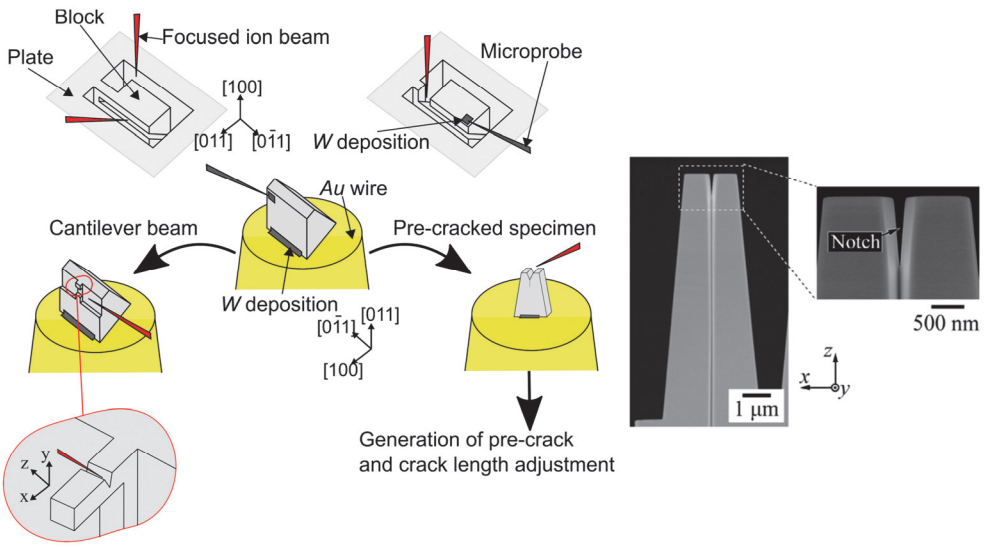

Figure 1: Example of fabrication of specimens.

EVALUATION OF FRACTURE TOUGHNESS USING NANOSCALE PRE-CRACKED SPECIMENS

Three micro-specimens were fabricated by a focused ion beam (FIB) processing system (see previous section). V-shape grooves were used as a guide for straight pre-crack introduction, and a notch was then introduced at the top of the specimen. The notches were, on average, 100 nm wide (mouth width) and 700 nm deep. Example of the specimen is depicted in Fig. 2(a) together with material properties, while geometrical parameters are reported in Tab. 1. The pre-crack was realized by applying an opening displacement with a wedge-shaped indenter according to Fig. 2(c). The upper part of the specimen was later cut by FIB to obtain shorter pre-cracks. The experiments were conducted using a sample holder with a loading device inserted in a TEM (transmission electron microscope) provided with *in situ* observation camera. The opening displacement was applied to the sample by pushing the wedge-shape indenter in the pre-crack mouth while a sensor beneath the indenter detected the applied load (see visual abstract). The stress in the specimens was later re-analyzed by finite element method (FEM) by properly considering the material anisotropy and 3D model was used. The material constants and the model employed are reported in Fig. 2(b). The fracture toughness was evaluated by the intensity of the singular field at the critical load (at crack propagation).

The results are summarized in Tab. 1 which reports the fracture toughness K_{IC}, the length of the singular stress field Λ_K and the critical crack opening displacement δ_C. From the results, it is possible to conclude that the average fracture toughness K_{IC} is 1 MPa·m$^{0.5}$. This value agrees with the bulk silicon value, i.e., 0.75-1.08 MPa·m$^{0.5}$ [12–14]. This finding proves that the fracture toughness is independent of the size. The K_{IC} is also independent of the length of the singular stress field. Results in Tab. 1 are, indeed, all very close to the same average value of 1 MPa·m$^{0.5}$. These results have been recently further confirmed by additional experiments and sophisticated first-principles density functional theory (DFT) simulations [10,15] which considered the discrete nature of atoms. In those works, Griffith criterion was applied to 4 nm stress singularity, and the fracture toughness resulted in being 0.95 ± 0.07 MPa·m$^{0.5}$, in agreement with values presented here.

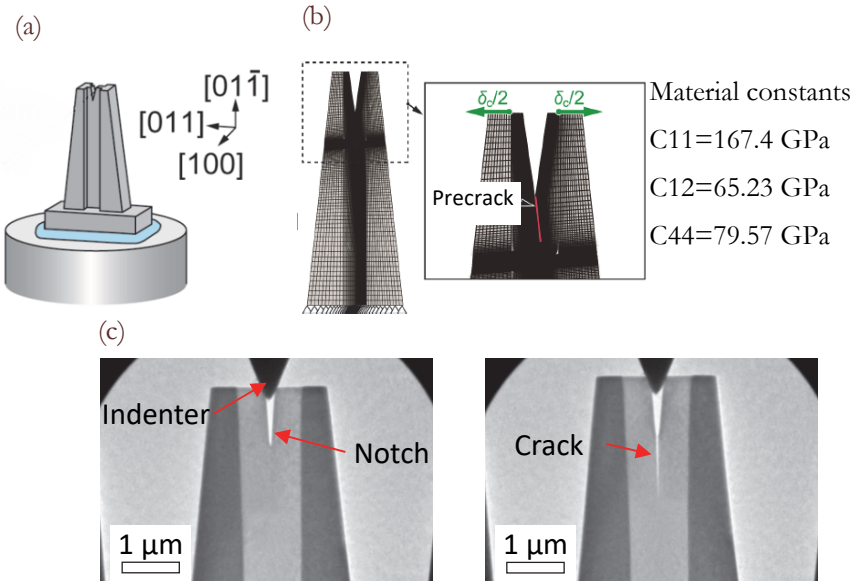

Figure 2: Example of (a) nanoscale notched specimen, (b) 3D FE model and (c) introduction of pre-crack.

	a (nm)	w (nm)	t (nm)	Λ_k (nm)	δ_C (nm)	K_{IC} (MPa·m$^{0.5}$)
Specimen 1	3982	830	580	23	205	0.83
Specimen 2	1412	600	480	25.4	62	1.09
Specimen 3	4359	474	302	57.5	237	1.08

Table 1: Pre-crack length a and width w; Thickness of the thinned region t; Synthesis of the results: length of singular stress field Λ_k and critical crack opening displacement δ_C.

EVALUATION OF FRACTURE TOUGHNESS USING NANOSCALE NOTCHED SPECIMENS

At very small scale, even a small notch radius can drastically change the stress singularity, and the fracture toughness obtained [16,17]. At the same time, the realization of crack is difficult and very challenging. For this reason, it is worth of investigating alternative procedures for the determination of those mechanical properties that would overcome these difficulties. Among the methods available in the literature, the theory of critical distances (TCD) has proved to be very versatile and simple. First developments on "material length parameters" were addressed by Neuber [18] and Peterson [19]. The recent history of the TCD, instead, was formalized by Taylor [20] and Susmel [21]. TCD in the form of the Point Method assumes that "failure occurs when the linear elastic maximum principal stress at a given distance $L/2$ from the notch root equals the inherent material strength of the material σ_0". L is the so-called material characteristic length, and it takes the following form under static loading [22]:

$$L = \frac{1}{\pi}\left(\frac{K_{IC}}{\sigma_0}\right)^2 \tag{1}$$

From Eqn. (1), it is clear that when the material characteristic length and the inherent stress are known, the fracture toughness can be quickly evaluated as:

$$K_{IC} = \sigma_0\sqrt{\pi L} \tag{2}$$

Susmel at al. [22,23] and Taylor et al. [20,24] proposed an efficient strategy to evaluate the material characteristic length and the inherent material stress. At least two static tests considering notch of different sharpnesses need to be carried out to determine the static strengths. Subsequently, the linear elastic stress fields of the two notches under incipient failure conditions can be plotted and overlapped in a single picture. The intersection of the two stress distributions permits to evaluate both σ_0 and L. Once these two parameters are defined, Eqn. (2) can be employed. The advantages tied-in to the procedure in the realization of simple geometries are relevant and worth of investigating. Therefore, to determine the fracture toughness of single crystal silicon, two notched nano-cantilever beams of different sharpnesses were realized by focused ion beam (FIB) processing system. The fabrication process reported in [11] was followed. The specimens had a notch radius ρ of 6.3, 20.2 nm and opening angle 2α of 68° and 59°, respectively. These geometries gave stress concentration factor K_{tn} (net section) of 4.9 and 2.9, respectively. The specimens were later loaded into the TEM by using an indenter provided with a load sensor (see visual abstract). Fig. 3(a) presents a sample during the experiment. Deflection at failure δ_f and load at failure P_f were obtained. The results are summarized in Tab. 2. Following the definition provided earlier, the fracture toughness K_{IC} and the material characteristic length L were determined as is shown in Fig. 3(b). The value of the fracture toughness was 1.05 MPa·m$^{0.5}$, while L was 1.8 nm. The inherent material strength σ_0 was approximately 14 GPa. These results have been further confirmed by additional tests in [11,25].

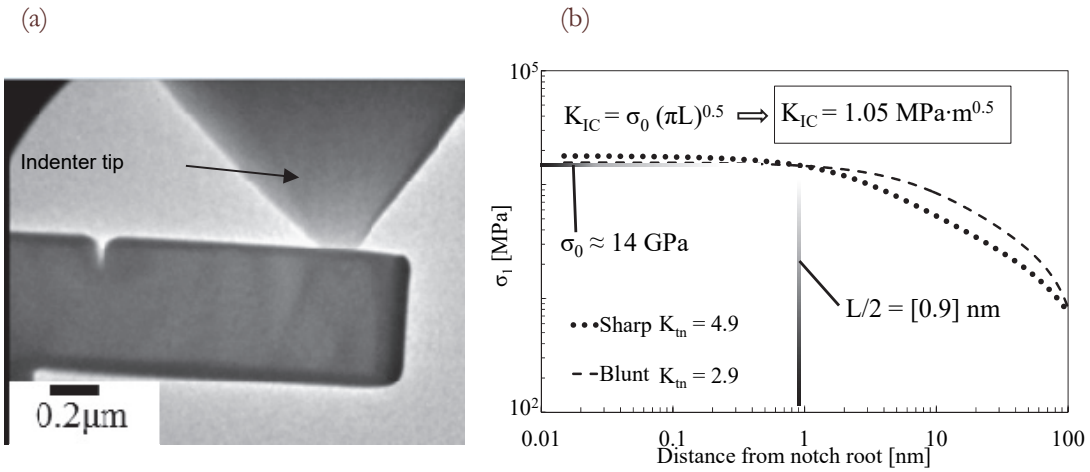

Figure 3: Example of (a) nanoscale notched specimen (TEM image) and (b) synthesis by using the TCD.

	ρ (nm)	2α (deg)	K_{tn}	δ_f (nm)	P_f (μN)
Specimen 1	6.3	68	4.9	83.74	30.84
Specimen 2	20.2	59	2.9	115.59	65.11

Table 2: Geometry of the notches, deflection at failure δ_f and load at failure P_f.

DISCUSSION: NANOCRACKING IN SILICON

As is well-known, tensile strength and fracture toughness are fundamental mechanical properties. While tensile strength has been investigated in several works/materials and its scale dependence observed [26,27], the fracture toughness has not been investigated in detail up to the present. The reasons are the well-known difficulties in the fabrication of very small specimens and in the realization of an effective pre-crack at the nanometer level. At very small scale, indeed, even a small notch radius can drastically change the stress singularity and the fracture toughness [16,17]. Ando et al. [16], for example, found that the K_{IC} changes with the crystal orientation, with maximum and minimum values of approximately 2 and 1.2 MPa·m$^{0.5}$, respectively. Li et al. [17] found a value of the fracture toughness of approximately 1.6 MPa·m$^{0.5}$, in apparent disagreement with bulk silicon value. The results presented here show that, if an effective pre-crack is realized, the nanoscale Si fracture toughness agrees with the macro counterpart. However, the realization of pre-crack is still very challenging and time-consuming. Alternatively, it is shown that by employing the TCD, the demanding accurate control on the specimens geometry can be avoided since the fracture toughness is evaluated using *at least two generic notches of different sharpnesses*, leaving the user free to employ any obtained geometry. Fig. 3(b) shows a fracture toughness in excellent agreement with macro counterpart value and the previous experiments on pre-cracked samples. Additional experiments conducted by [10,11,15,25] further confirm the results presented here.

Useful information on crack propagation can be obtained by the experiments conducted on pre-cracked samples. Fig. 4(a) refers to the sample 2 in Tab. 1 and shows that although the not perfect specimen orientation, a stable crack propagation occurred along the cleavage plane (011). Once the critical crack opening displacement was reached (see Tab. 1), unstable crack propagation and subsequent final failure were observed. The propagation once started, is very fast and brings to the final failure rapidly. As is well-known, the fracture is usually studied on the assumption that fracture takes place from pre-existing defects of flaws. Defects, however, vary in number and severity depending on the volume and size of the sample. Ideally, at the nanoscale, the inherent material strength σ_0 can be considered to be the upper limit of fracture stress of components having no defect, i.e., *ideal fracture stress* [28]. The micro-mechanisms governing the crack propagation in such small components should be located therefore at a smaller level, i.e., atomic structure level. A more in-depth investigation can only be conducted if the discrete nature of atoms is considered, by using numerical experiments. Indeed, recent density functional theory (DFT) calculations showed that the nanocracking in silicon is due to the breaking of a covalent bond at the crack tip. When the applied stress intensity factor K_I reaches the critical value of 1 MPa·m$^{0.5}$ the bond is broken and the crack propagates instantaneously along the cleavage plane (011). This result was further confirmed with additional DFT simulations considering different models and molecular dynamics (MD) simulations, where the stress is calculated atom-by-atom using the Virial theorem. Regardless of size, geometry and loading conditions, the simulations confirmed that the nanoscale crack always starts to propagate when the atomic stress at the tip reaches its ideal (bond) strength [9,15].

Fig. 4(b) represents the fracture mode displacements at the onset of nanocracking and schematically shows the crack advancement by atomic bond breaking. In the light of crack ultimately governed by atomic bond breaking and therefore by atomic level, it is natural to wonder whether the continuum theories should be applied at such small scales, if there is any limitation to the continuum theories and how eventually evaluate this limit. Theory of critical distances lies between continuum mechanics theories and micro-mechanistic approaches. The material characteristic length should be considered therefore a representative length scale parameter that changes as the mechanisms of fracture do [29,30]. L, therefore, can somehow be representative of the low limit of continuum fracture mechanics. Validation of this hypothesis can be done by comparison with a recent relevant work by Shimada et al. [9]. Those authors proposed an interesting study based on sophisticated DFT and MD calculations on cracked specimens. Several samples were studied by varying the size. It was shown that the length of the singular stress field becomes smaller as the width of the specimen does, while the fracture process zone (defined as the zone where the discrete motion of atoms is concentrated) remains constant. When approaching a critical singular stress field length, continuum theory breaks down. This limit was quantified in 3-6 times the fracture process zone, and therefore in the case of silicon is 1.5-3 nm approximately, with a fracture process zone of 0.4-0.6 nm on average. Interestingly, L falls within the range of that limit, being 1.8 nm, while $L/2$ is close to the fracture

process zone value. With the quantification of low limit of continuum theory, it is possible to assume that method such as the TCD can be applied successfully at very small scale; however, once the limitation is reached, discrete nature of atoms cannot be neglected and other methods should be developed. It should be noted that other approaches have similitude with the TCD [31] and therefore have an excellent potential to be applied at small scales considered here. *Finite Fracture Mechanics* (FFM) [32], for example, shares several aspects with the TCD and its consideration is left as future work.

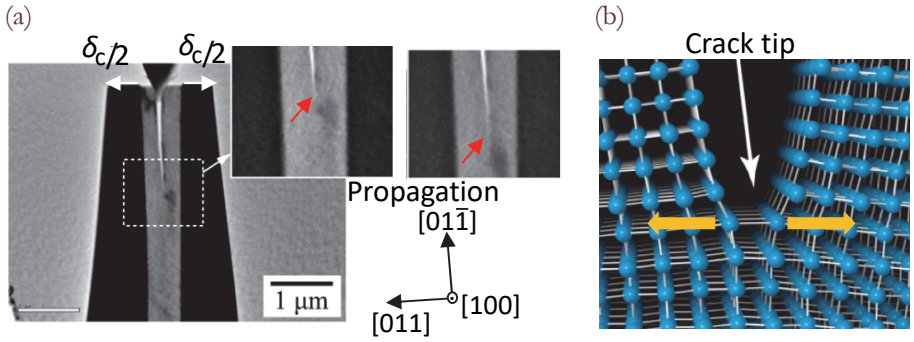

Figure 4: TEM image of (a) crack propagation of specimen 2 and (b) fracture mode displacements at the onset of nanocrack.

BEYOND THE BREAKDOWN OF THE CONTINUUM THEORY

This contribution has shown that some continuum theories could be directly extended to micro and nanoscales until their low limit is reached but it does beg the question of how to approach lower scales, where the discrete nature of atoms must be considered. As is shown before, experimental testing is very challenging at the microscale, becomes tough at nanometer-scale and extremely tough at lower scales. However, atomic simulations have developed considerably in the last decade supported by the advancement of computing power and numerical techniques. These simulations, based on interatomic potentials, have drawn considerable attention in the scientific community because of their versatility in the reproduction of qualitative phenomena and are nowadays a reliable tool. When approaching atomic scale and in general beyond the breakdown of continuum theory, the validity and definitions of usual macromechanics parameters (e.g., stress, strain, elastic constant) should be reconsidered. Approaches based on energy, instead, seem to be very versatile and their concepts showed to be easily extensible and valid at the atomic level. If one considers the brittle fracture of silicon, the Griffith criterion, Energy Release Rate (ERR) and others have been reformulated successfully to take into account the atomic structure and validated by using atomic simulations [9,15,33]. As a straightforward extension of the continuum concepts, for example, ERR has been reformulated based on atomic potential energy [34], showing to be more general than continuum counterpart and valid regardless of considered scales. A similar idea is under development by the present authors by considering the strain energy density and preliminary results should be soon available. The silicon, however, is relatively easy to be treated because of its intrinsic "*ideal*" brittle behavior: the fracture process does not involve dislocations but develops clearly by atomic bond breaking along the cleavage plane. In the fracture nanomechanics, fracture of silicon represents the most fundamental and straightforward research target. Challenges toward nanometer scale fracture mechanics rapidly increase when plastic phenomena (e.g., emission of dislocation [35]) and more complex load conditions (e.g., Mode II, Mode III, mixed mode) have to be considered. Ultimately, the definition of these primary mechanisms will depend on the realization of atomic-level fracture mechanics experiments with *in situ* observation. This final task will require a total reconsideration and improvement of current experimental equipment and techniques.
The investigation of atomic fracture mechanics might bring great advancements not only in several academic fields (e.g., multi-physics) but also in industrial miniaturization of future electronic devices.

CONCLUSIONS

This contribution has presented a synthesis of some recent micromechanical tests aimed at the evaluation of the fracture toughness of silicon by using pre-cracked nano specimens and alternatively notched nano specimens combined with the theory of critical distances (TCD).

The results can be summarized as follows:

- The fracture toughness of silicon is 1 MPa·m$^{0.5}$ and is independent of the size and crystal orientation;
- While accurate control of the geometry in the specimen fabrication is challenging, small crack tip radius has a relevant influence on the fracture toughness value when pre-cracked samples are employed;
- The TCD simplifies the experimental procedure for the evaluation of the fracture toughness by employing notched specimens instead of pre-cracked samples;
- The crack propagation at this small scale, where ideally there is no defect, is due to the breaking of atomic bonds along the cleavage plane;
- The TCD also yields a good approximation of the magnitude of the fracture process zone, defined as the zone near the crack tip or notch root where the discrete motion of atoms is highly concentrated;
- The results obtained here agree with those reported in the literature that define the breakdown of continuum fracture mechanics when the *K*-dominant region critical size is in the range of 3 to 6 times the fracture process zone.

Acknowledgments

Videos of experiments are available in the visual abstract.
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