

Three Functions in One: 3D Confocal Raman AFM-Raman Tip-Enhanced Near-field Raman

Material Identification and Distribution, Analysis of Molecular and Crystal Structure, Stress by Raman Spectroscopy



Nanofinder[®]FLEXG

Three Functions in One : AFM-Raman Microscopy System Leading the Field

3D Confocal Raman Microscopy

High sensitivity: Detection of the weak 4th order Si peak High spatial resolution: < 200 nm High speed: < 3 ms/point (spectrum)

AFM-Raman

Simultaneous or sequential measurements of AFM and Raman images from the same sample area Surface topography with simultaneous 2D confocal Raman AFM resolution:0.1 nm High spatial resolution: < 450 nm(Raman) High speed: < 7 ms/point (spectrum)

Tip-Enhanced Near-field Raman

TERS (Tip-enhanced Raman spectroscopy) Spatial resolution < 50 nm by Near-Filed Raman method

Features

Stable experimental conditions

Extremely high spatial resolution requires minimizing of mechanical temporal and thermal drift, suppression even the smallest vibrations.

Nanofinder[®]*FLEXG* ensures data stability and reproducibility.

Granite Frame

 $\mathit{Nanofinder}^* \textit{FLEXG}$ is based on the high stiffness granite frame with low thermal expansion coefficient. It minimizes the defocusing and sample lateral displacement in severe environment conditions of temperature fluctuations or presence of mechanical vibration sources.

Smart Sliding Mechanism (SSM)

SSM mechanism with Weight Compensation Spring and Air Suspension Drop Protection ensures smooth long-distance moving of optical unit for objective lens or sample exchange. Displacement of laser focus point after moving of optical unit up and down is within 1 μ m.

LED Illuminator

Cool LED illumination minimizes the thermal drift of the microscope system. The sample observation can be done without heating effect.



Newly designed high throughput optical unit

• High Throughput

Raman signal throughput from the sample to the detector is within 30-40% (taking into account all losses in the microscope objective lenses, the optical unit, the spectrometer). "No moving parts" optical unit design principle makes unnecessary the system alignment procedure.

Example of 3D Raman image (Si/SiO₂ wafer)

Measurement conditions (confocal Raman)

Measurement points	: 100 × 100 × 20 (X,Y,Z)
Exposure time	: 3 ms/point (spectrum)
Measurement time	: 10 min
Raman shift	: 520 cm ⁻¹
Laser	: 0.7 mW@633 nm



Small Footprint* Optical unit is downsized to A4 paper format and is installed on top of the microscope.

The total footprint of the $Nanofinder^*FLEXG$ optical system (open type) is 30×40 cm (W × D). It can be supplied with Class 1 cover equipped with user protection interlock.

The microscope system and controller are connected only with flexible wires and can be easily adapted to user facilities.

Polarization Measurement*

The optical unit layoutallows to carry out the high quality polarization measurements with free orientation of linear laser beam polarization on the sample surface. The Glan-Taylor prism with rotation mechanism around the optical axis is installed in the detection channel.

Imaging spectrometer & Raman detector

- MS3504 focal length 35 cm, 2 exit port Up to 4 gratings, automatic exchangable. High imaging quality, high throughput to a CCD or an APD detector.
- Spectral resolution 2 cm⁻¹~ Optional spectrometers with

other focal lengths (20 cm, 75 cm) are available for different resolution requirements.

Raman detector High-end CCD detectors are selectable depending on the required wavelength range (see p.11, Fig.2 Quantum Efficiency (QE) curves).

Customer budget saving

Modular design principle of *Nanofinder*^{*}FLEXG gives a chance to save customers' budget by combining their available devices into the whole system of $\mathit{Nanofinder}^*\mathit{FLEXG}$.

Customer's spectrometer, CCD detector (ANDOR) and lasers can be used for decreasing the total system cost.

Software

The Nanofinder[®] software provides 3D display and powerful tools for practical data analysis (see p.6).

Wide working space broaden potential applications*

Wide working space under the objective lens 300x220x300 mm (WxDxH) allows to install various equipment, such as piezo stage for high-resolution confocal Raman imaging, AFM, heating&cooling crvostat. stage, high pressure chamber, magnetic field facility, etc.



AFM



Contol&measurement rack (including PC, spectrometer, CCD detector, piezo controller etc.) Separately placed with the microscope system



High pressure (Diamond anvil cell)



W: 300 mm





Work space

D: 220 mm

XYZ-piezo stage

Heating&cooling stage



3D Confocal Raman

High Sensitivity*, High Spatial Resolution (XY < 200 nm)* High Speed < 3 ms/point (spectrum)*

Advantage of the *Nanofinder*[®]*FLEXG* is 3D fast Raman imaging with high sensitivity and high spatial resolution. It is achieved by employing the high throughput optical system, high sensitivity CCD detector and high accuracy piezo stage .

High sensitivity

High Sensitivity Confirmation by ability to detect the weak 4th-order Si Raman peak

Si Raman spectrum with clear the 4th-order peak is shown in Fig. 1.

An intensity of the Si Raman peak at 520 cm⁻¹ (1st-order) is usually strong enough but higher overtones show much weaker signal. Therefore, detection ability of higher orders of Si Raman peak may be considered as a sensitivity benchmark of the Raman spectroscopic system.

High sensitivity of *Nanofinder*[®]*FLEXG* permits to reduced the required excitation laser power and, resulting in avoiding the modification or damage of fragile samples.

High spatial resolution

Raman Image of Si/SiO₂ 200 nm line&space

2D intensity mapping at Si Raman peak (520 cm⁻¹) of 200 nm Si/SiO₂ line&space sample is depicted in Fig. 2. The corresponding Scanning Electron Microscope (SEM) image is shown in Fig. 3.

These figures demonstrate Raman imaging with high spatial resolution, giving clear separation of the 200/200 nm line&space structure.

High speed

With using of the most advanced EMCCD

Nanofinder[®]FLEXG provides high sensitivity, high accuracy and high speed (< 3 ms/point) sample measurement, storing full Raman spectrum in every mapping point. TII advanced technology solutions give the fastest scanning procedure in the closed-loop mode.

Typically about 3 sec. are required to obtain a clear Raman image of 32x32 points (X,Y) resolution. Higher resolution requires longer accumulation time with a lot of measurement points.

Resolution vs. required mapping time:

- 32x32 (X·Y) :1024 points ~ 3 sec.
- 32x32x10 (X·Y·Z): 10,240 points ~30 sec.
- -100x100x30 (X·Y·Z) : 300,000 points ~ 900 sec. (15 min.)



Fig. 1 Si Raman spectrum of the higher order overtones

Measurement conditions

Excitation laser	:4 mW@532 nm
Objective lens	: 50 × , NA 0.8 (magnification, numerical aperture)
Measurement time	: 60 sec.



Fig. 2 Raman image of Si/SiO₂ line&space



Fig. 3 Scanning Electron Microscope (SEM) image

*TII advanced point

Examples of 3D&2D Raman Imagings

Three dimensional mapping&display* Optimal method for observation of foreign compound, their structure and chemical modifications inside transparent materials

Degradation of Li-ion battery electrode

In the Li-ion battery the repetitive cycles of charge and discharge lead to the performance degradation. In these cycles the original material of the positive electrode surface - $LiCoO_2$ transforms into Co_3O_4 .

The 3D pictures at the bottom show the Raman images of the Li-ion positive electrode surface using specific peaks of $LiCoO_2$ (600 cm⁻¹) and Co_3O_4 (700 cm⁻¹), respectively. These images offers the feature of electrode degradation in three dimensions.





Surface observation of Li-ion battery positive electrode by optical microscope

Raman spectra of $LiCoO_2$ and Co_3O_4 on Li-ion battery positive electrode surface



Measurement conditions

Sample	:	Li-ion battery positive electrode
Excitation laser	:	5 mW@532 nm
Objective lens	:	$100 \times$, NA 0.9 (magnification, numerical aperture
Measurement points	:	$32 \times 32 \times 10$ (X, Y, Z) × 18 ms/point (space)
Measurement time	:	185 sec.

3D Raman images of positive electrode in a Li-ion battery



Measurement of amorphous Si thin film crystallization

Crystallization condition of each point of amorphous Si thin film (t:60 nm) illuminated by a Nd:YAG laser (532 nm) Data courtesy of S. Horita, Japan Advanced Institute of Science and Technology

*Tll advanced point

Examples of Data Analysis

During the mapping procedure, 2D or 3D images can be observed in real time. *Nanofinder*^{*} unique software has advanced abilities of drawing and data analysis. Various data processing and presentation tools are available. *Nanofinder*^{*}*FLEXG* software can easily manipulate with a huge volume of 3D mapping data with full Raman spectrum being saved in every point.

Reduced measurement time & optimization experimental conditions*

In order to get good quality mapping images a lot of trial measurements are required to determine the appropriate experimental conditions.

$$\label{eq:states} \begin{split} \textit{Nanofinder}^*\textit{FLEXG} & \text{with its real-time display can} \\ \text{analyze data during the measurement. Fast data} \\ \textit{visualization} & \textit{allows} & \textit{user} & \textit{fast} & \textit{parameter} \\ \textit{adjustment, area exchange and the measurement} \\ \textit{restarting.} \end{split}$$

Data analysis capability*

During the mapping procedure all spectra from every measurement (X,Y,Z) point are stored in the computer memory. *Nanofinder*^{*} software provides various imaging functions, such as Raman peak intensity, peak shift, peak FWHM (Full Width at Half Maximum). Built-in image calculator provides ability even for various more complicated image functions visualization, such as image filtering, image subtraction, and Raman peak intensity ratio. Deconvolution and curve-fitting functions improve hardware specifications for spatial and spectral resolution.

Impressive imaging data*

Measured 3D data can be easy observed from arbitrary angle (Fig. a). 2D cross-sections plane position and orientation are also arbitrarily selected (Fig. b, d).

1D cross-sectioning provides an ability for distance measurement and intensity (and other imaging functions) plot presentation along the arbitrary direction (Fig. c,f). Pointing tools show Raman spectrum in the specified mapping point (Fig. g). Multiple pallets provide a powerful tool for impressive image coloration.





3D Raman image of self-assembled liquid crystal

(Intensity images of Raman with at 1136 cm⁻¹ shift)





Fig. g Raman spectrum of self-assembled liquid crystal (Measured at point A in Fig. c)

*Tll advanced point

AFM-Raman

Simultaneous Measurements at the Same Position by AFM and Confocal Raman*

AFM-Raman system $Nanofinder^*FLEXG$ allows for simultaneous measurements of AFM topography and Raman confocal image at the same sample position.

Such correlated images of the surface topography and Raman spectroscopy are an useful for various applications such as defect evaluation of semiconductor devices, and foreign materials detection. Several different AFM modes can be applied for simultaneous operation with the confocal Raman measurment.

Nanofinder *FLEXG AFM is especially designed for combined operation with the confocal Raman system. With using of IR laser wavelength at 1300 nm there is no influence of AFM feebback control system to Raman system. Design concept is focused on full device automation, easy use, easy exchange of cantilever or sample, easy combination with the confocal Raman system (by hardware and software), high stability and low drift for long time measurements.

Optical sample access with high NA objective lenses

Nanofinder *FLEX G AFM designed for combined operation with confocal Raman has the best optical access to the sample/probe area: microscope objectives of 100xNA0.7 (from top) can be applied for simultaneous combined AFM-Raman measurements. As a result, spatial resolution below 50 nm can be achieved in TERS measurement (see p.8).

Automated alignment mechanism

AFM can start measurement quickly after adjusting the laser and the position-detection system by an automated alignment mechanism.

Advanced sample position adjustment

AFM is equipped with a motorized XY sample positioning system, which ensures the possibility of easy return or a quick switch between points/areas of interest. Fine closed-loop XYZ piezo-scanner is applied to the AFM and the confocal Raman scanning. The motorized XY stage provides precise sample positioning and XYZ piezo scanner ensures fine images.

Focused laser spot/AFM probe position adjustment

A Closed loop AFM unit positioning system is applied to automatical laser spot/AFM tip apex fine coincidence. Preliminary adjustment of the overlap of the spott and the tip can be performed manually by AFM head positioning using microscrews.

True non-contact mode

A non-contact operation mode is realized by fast (190 MHz) DSP (Digital Signal Processor).The non-contact scanning mode at reduced amplitudes of probe oscillations is optimal for soft samples measurement or delicate AFM probe using, avoiding damage of the sample or the probe due to sample/probe contact.

High environment desturbance-resistant

High resonance frequencies of AFM in combination with high stiffness of the *Nanofinder*^{*}*FLEXG* granite base enables the achivement of high spatial resolution (<50 nm in TERS) even without special vibration suppression system (in a conventional laboratory environment).

AFM-Raman Simultaneous&Same Position Measurement of CNT (Carbon Nano Tube)



Tip-Enhanced Near-field Raman

TERS:Tip-Enhanced Raman Spectroscopy

High spatial resolution < 50 nm by near-field Raman spectroscopy with AFM metal coated probe

Tip-enhanced Raman spectroscopy (TERS) is a novel method for getting Raman spectra/images with a high spatial resolution power far beyond diffraction limit of light. Near-field light is generated at the tip apex of a metal coated sharp probe (10-30 nm). Tokyo Instruments, Inc. commercialized the world's first reflection TERS imaging system^{*}.

Measurement principle

The basic system configuration for TERS is similar to AFM-Raman combined system. AFM probe for TERS is coated by noble metal (Au or Ag) instead of bare Si probe for just combined AFM-Raman measurements. At first, AFM probe approaches the sample surface. Then closed-loop AFM unit positioning system realizes exact coincidence of the tip apex and focused laser spot. In an appropriate conditions the localized surface plasmon may be generated at the tip apex of the probe. It may enhance the Near Field Raman signal up to thousands times from area of 10-30 nm around the tip apex. Therefore Raman spectra and images can be measured with spatial resolution beyond diffraction limit.

Reflection optical configuration

Reflection configuration is the only way for TERS measurement of opaque samples. It may be applied to low-dimentional samples such as carbon nanotubes, distributed on Si substrate. Graphene, various nano crystals, strained Si structures are also suited to TERS measurements.

Easy optical alignment

In TERS technique the laser spot must be focused exactly on the AFM tip apex. Closed-loop AFM unit positioning system with piezoscanners and capacitive sensors (optional) of *Nanofinder*^{*}*FLEXG* automatically realizes this focusing procedure.





Exact positioning of focused laser spot onto AFM tip apex for TERS.

AFM features of the TERS:

Tip-Enhanced Raman Spectroscopy system

• Easy to exchange AFM probes with high position reproducibility due to cantilever holder and PSD (Position Sensor) motorized mechanisms and fully automatical software algorithm

- Sample exchange or position search without removing and realignment of AFM head
- Scanning speed up to 40 lines/sec
- Scanning area $100 \times 100 \times 15 \,\mu$ m with atomic resolution
- USB2.0 interface



Reflection system

TERS Imaging

Examples of Si/SiO₂ line & space structure

Si/SiO₂ (100/100 nm) line & space structure was measured with different methods and then results were compared. Figures on the right show TERS image (Fig. b), confocal Raman image (Fig. c), AFM topography (Fig. d), SEM image (Fig. e).

Fig. a Raman spectrum at point A in Fig. b (TERS image)

This Raman spectrum consists of two peaks which are separated with TII peak fitting software. The yellow line represents Si peak at 520 cm⁻¹ (it may include the Far Field confocal Raman signal and the Near Field TERS components) and the blue line around 515 cm⁻¹ shows signal from AFM Si probe, irradiated by a focused laser.

Fig. b. 2D image* of peak intensity at 520 cm⁻¹ (including the TERS component (the yellow line in Fig. a)). (Uniform background is subtracted).

Strong signal (depicted with a white color) area represents a Si area, while weak intensity (depicted with a purple color) area represents SiO_2 area. The period between two neighboring Si or SiO_2 lines is 200 nm and the edge responce between Si and SiO_2 lines is below 50 nm, beyond the diffraction limit of the excitation laser wavelength at 532 nm.

Fig. c Confocal Raman image

With an objective lens 100x NA0.7 and the excitation laser wavelength of 532 nm, the confocal resolution of the system is calculated as 450 nm and it is not expected to resolve the 100 nm line&space structure.

Fig. d AFM topography.

An AFM topography image has high spatial resolution and it confirms the TERS image in Fig.b. Besides it helps to interpret the data, supplying information about the sample topography.

Fig. e SEM image



Fig. a Raman spectrum at point A in Fig. b



10 Nanofinder **FLEX G Detailed specifications**

System configuration

- ·Upright microscope
- ·Granite frame, objective lens, TV-CCD camera
- Optical unit (Raman optical system)
- Imaging spectrometer (f=35 cm)
- ·Raman detector (cooled CCD or EMCCD detector)
- Piezo stage (X-Y-Z) or Atomic Force Microscope (AFM)
- •Excitation laser (see Table 1, CW laser)
- Computer, 19 inch LCD
- Software

3D confocal Raman

- ·Spatial resolution (Fig.1):< XY 200 nm (@100x, NA 1.4)
- •Wavenumber range:50~8000 cm⁻¹ (@532 nm) (depending on excitation lasers)
- ·Spectral resolution(f=35 cm spectrometer):2 cm⁻¹
- •High speed: < 3 ms/point (spectrum) (@EMCCD)
- •Work space: 300 x 220 x 300 (WxDxH, mm)

Units specifications

Optical unit

- ·A4 size
- ·Raman optical system, collecting system
- ·Filter set (exchangeable for different excitation lasers)
- ·Optical fiber input&output (2 m length respectively),
- ·FC connector coupling
- Imaging spectrometer:
 - ·MS3504i with 2 exit port
 - (2 detectors installation possibility, automated port

change)

- ·Focal length:35 cm
- •F number:3.8
- ·Reciprocal linear dispersion: 2.37 nm/mm @1200 G/mm
- Maximum gratings:4 (selectable, automated change) (see Table 2)

Raman detector

Cooled CCD or spectroscopic EMCCD detector (see Fig. 2)

- •Active pixels :1024 × 127 (1600 × 200 EMCCD)
- Pixel size $:26 \times 26 \,\mu$ m (16 × 16 μ m EMCCD)
- •Image area :26.6 × 3.3 mm (25.6 × 3.2 mm EMCCD)
- Maximum cooling : -100°C (water), -80°C (air)

Piezo stage (use for 3D confocal Raman)

- · Stroke (closed loop) : X-Y:100 μ m, Z : 30 μ m
- ·Resolution: 5 nm
- ·Repeatability: ±5 nm
 - (*Piezo stage is not needed in case of AFM Smart SPMTM installation)

- ■AFM Smart SPMTM 1000 (see Table 3)
 - ·For AFM-Raman measurement or Tip Enhanced Near-
 - Feld Raman measurement (TERS)
 - including X-Y-Z piezo scanner (100 \times 100 \times 15 μ m)

resolution 0.1 nm

Excitation laser (LD pumped)

- ·Wavelength:532 nm, power: 50 mW
- Power range:50
 µ W~50 mW (continuously variable)
- ·Wavelength 785 nm, power :70mW

Table 1. CW laser

473 nm 488 nm 514.5 nm	532 nm	633 nm 671 nm	785 nm	830 nm
Optional	Std.	Optional	Std.	Optional

For the excitation laser exchange the filter set in the Raman optical unit should be exchanged. The mechanical holder for the filer set is equipped with high position reproducibility mechanism

- Computer, 19 inch LCD
 - Software : Nanofinder[®] (original from Tokyo Instruments)
 - ·Control&anlalysis integrated
 - Multilanguage support, including : Japanese, English,
 - Chinese
 - ·OS: Windows XP professional
 - ·Measurement: point , 1D, 2D, 3D
 - ·Image drawing&analysis:1D, 2D, 3D imaging
 - •Various operations:displaying, rotating, magnification, arbitrary cross-sections,etc.
 - ·Various image filters,1D-2D deconvolution etc.
 - •Various imaging functions (integrated intensity, peak shift, FWHM, etc.)
 - •Spectral analysis: Filtering, smoothing, base line correction etc.

Lorentzian, Gaussian peak fitting (Max. 3 peaks)

- ·Automated wavenumber calibration
- •Fluorescence lifetime imaging with temporal resolution < 100 ps
- (with optional: TCSPC module, pulsed laser, PMT or

APD detector)

Size (Microscope system and Raman optical unit)

·Microscope: 300x400x770 (WxDxH, mm)

19 inch monitor mount desk (see picture on cover)

·Rack:600x850x700 (WxDxH, mm)

(spectrometer, CCD, piezo stage controller, PC)

Power supply

·100 V - 10 A (1000 W)

Detail Specifications

Fig. 1 Spatial resolution dependence on wavelength and objective lens NA in 3D confocal Raman microscopy







Andor Technology High sensitivity cooled detector

 DU970P EMCCD (FI, BV) High-end model with electron multiplying ability High speed 3MHz ADC Good for high speed measurement by signals gaining
 DU920P CCD (OE, BV, BR-DD)

High speed 3MHz ADC. High performance model

- ●DU401A CCD (BV、BR-DD) 、DU420A CCD (OE、BV、BR-DD)
- ●DU40TA CCD(BV、BR-DD)、DU420A CCD(100kHz ADC.Low cost model

7 6 5 4 3 2 1 0 350	488 NA=0.3 NA=0.45 NA=0.9	532 m mm 500 55	n 632	650
350	0 400 450 5 Wave	500 55 elengt	0 600 h(nm)	650

Table 2 Grating (4 selectable, automated change)

1 ())

Groove Density (G/mm)	Blaze Wavelength (nm)	Wavelength Range (nm)	Resolution (cm ⁻¹)
2400	400	265 ~ 645	1.5
1800	750	500 ~ 860	2.0
1200	600	400 ~ 1200	3.0
600	600	400 ~ 1200	6.0
300	600	400 ~ 1200	12.0
200	700	465 ~ 1400	18.0
150	700	465 ~ 1400	24.0
100	700	465 ~ 1400	36.0

*MS3504i (35cm), CCD detector (26 µm pixel size) 1.5 pixels, entrance slit 30 µm.

1.2 cm⁻¹ @1800 G/mm,EMCCD(16 μm pixel size)

Supported objective lens (high NA 0.7, 100 ×)

Scanning type by sample Scanning range **100 x 100 x 15** μ m (+/-10 %, closed loop capacitance sensor) Sample size 40×50 mm 15 mm height **Topography noise** < 0.1 nm(X-Y), 0.04 nm(Z) **Control method** XYZ digital closed loop Scanning speed up to 40 lines/sec (Typ.) Sample positioning Motorized, 5 \times 5 mm, positioning resolution 1 μ m Laser wavelength 1300 nm Interface **USB 2.0** Laser alignment Full automated with stepping motors Electronics High speed DSP (190 MHz) Digital, analogue input-output · AFM Semicontact Contact **True non-contact** Lateral force microscope (LFM) Magnetic force microscope (MFM) **Measuring modes**

- Kelvin probe microscope (KPM)
- Electric force microscope (EFM)
- Phase imaging
- Force curve measurement
- · Conductive AFM (Optional)
- Nanolithography
- Nanomanipulation
- Scanning tunneling microscope (Optional)
- · Photocurrent conductive mapping (Optional)
- · V-I characteristic measurement (Optional)

Table 3 AFM basic specification - smart spm™1000 -

cooling : -100°C (water cooling), -80°C (air cooling), 16bit dynaimc range

Measurement Features in Raman Microscopy

- · All types of samples (solids, liquids, gases and living systems)
- Non-destructive, non-contact measurements
- Measurement in air (No vacuum requirement)
- No special sample preparation requirement
- Microscopic observation
- Measurement in various gases
- · Easy in-situ and cross-sectional measurements

Typical applications in Raman spectroscopy

Chemical structure analysis

- Strain&stress in crystal, doping effect, superlattice, lattice defect, chemical binding observation Various samples:
 - semiconductor, organic material, carbon nanotube, fullerene, diamond, jewel, oxide, various compound semiconductor, thin-film, L-B film, glass, liquid crystal, solar cell and others.

Foreign compound detection

·Food, insoluble material in liquid, Si sample, photo mask, plastic, thin film, glass inside and others.

Crystal structure analysis

·Crystal layer change, crystallization, amorphous crystallization

Erosion evaluation

·Oxidization detection of various metal samples like stainless steel

Investigation& research on painting

·Photochromic material, functional group identification, painting inspection

Reaction process monitoring for production process control

·Reaction of polymerization, dissociation, chemical&catalytic reaction, diffusion, melting

Archeology

·Ancient potters glass products

Security

·Detection&inspection of faked print, counterfeit, drug, chemicals

Gas detection

·Detection of various gases such as hydrogen and methane, gas compound in air bubble

Biotechnology&medical

•Detection of blood serum, indifferent cell, tumor, analysis&diagnosis of bone quality, bone cell, skin change, viable yeast, protein

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