

Epitaxial growth of cobalt films on Cu(100): a crystallographic LEED determination

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Abstract. Epitaxial thin films of cobalt ranging from 1 ML up to 10 ML have been grown on a Cu(100) substrate and characterized by LEED. The cobalt is found to grow, adopting a slight tetragonal distortion of its high-temperature FCC phase. Further growth of 5 ML Cu on top of a 5 ML Co film results in an almost perfect Cu(100) surface.

1. Introduction

It is well known that below 660–690 K the stable phase of bulk cobalt is HCP, and only above 720 K does the material develop an FCC phase. It would be of considerable interest to study the growth of the FCC high-temperature phase at lower temperatures, for example at room temperature, and it has been now almost 30 years since the first attempts at growing FCC Co films on Cu substrates were made (Goddard *et al* 1968, Jesser *et al* 1968).

Many studies have since been carried out on the growth of Co on Cu(100) in UHV conditions, and the structural (Gonzalez *et al* 1981, Clarke *et al* 1987, Li *et al* 1990, Ferrer *et al* 1991, Miguel *et al* 1991), electronic (Miranda *et al* 1983, Schneider *et al* 1990a) and magnetic (Pescia *et al* 1987, Beier *et al* 1988, Miguel *et al* 1989, Schneider *et al* 1990b, Kief *et al* 1991, Kämper *et al* 1992) properties of such films have been studied in detail. However, in spite of all this effort and its critical importance for other investigations, a detailed surface crystallographic characterization of Co films on Cu(100) was still lacking.

The FCC phase of Co has a lattice parameter at room temperature of 3.548 Å. The corresponding interplanar spacing along the [100] direction is then 1.774 Å. The pseudomorphic growth of FCC Co on Cu(100) with the lateral (2.556 Å) and vertical (1.805 Å) spacings of bulk Cu would correspond to a uniform 2% expansion of the FCC Co lattice. Total energy calculations (Marcus *et al* 1988) indicate that this 2% tensile strain represents an increase in energy of 68 meV per atom with respect to the unstrained FCC Co. Although the accuracy of this calculation might be limited, we stress that this

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is a large number, specially if one considers that the calculated difference in energy between FCC and HCP Co is 27 meV per atom (Min *et al* 1986). Accordingly, there are two solutions that nature may adopt: a Co film could grow in the strained FCC phase up to a limited thickness, where it would switch to the unstrained FCC structure by introducing a lattice of misfit dislocations, or the film could adopt the lateral spacing of Cu and *contract* the vertical spacing trying to keep the volume of the unit cell. The latter possibility would result in the stabilization of a face-centred tetragonal structure, i.e. a tetragonally compressed FCC phase (FCT). Although this has been previously suggested in the literature (Ferrer *et al* 1991, Miguel *et al* 1991), this work presents clear experimental evidence of this being the case.

The present contribution yields a full determination of the structural parameters that characterize the Co/Cu films. A detailed LEED study of this growth has been made for five different Co coverages: 1 ML, 2 ML, 3 ML, 5 ML, 10 ML and Cu-5 ML/Co-5 ML/Cu(100) (see table 1).

Table 1. Structural parameters. See text for an explanation of the various quantities.

	1 ML	2 ML	3 ML	5–10 ML
$d_{12}(\text{Å})$	1.76 ± 0.03	1.77 ± 0.02	1.76 ± 0.02	1.72 ± 0.03
$d_{23}(\text{Å})$	1.78 ± 0.03	1.73 ± 0.03	1.74 ± 0.02	1.74 ± 0.03
$d_{34}(\text{Å})$			1.76 ± 0.03	1.73 ± 0.03
$d_{\text{Co}}(\text{Å})$				1.73 ± 0.03
$d_{\text{Cu}}(\text{Å})$	bulk	bulk	bulk	bulk
R_p	0.22	0.22	0.23	0.24
$\Delta E(\text{eV})$	2500	1850	2150	3530

2. Experiment

LEED intensity spectra (IV curves) were recorded with a video LEED commercial system featuring a computer controlled CCD camera. The data were acquired in two independent experimental runs carried out in a chamber described elsewhere (Schneider *et al* 1990a) for energies varying between 60–500 eV. The experimental curves were normalized to constant incident current, corrected to eliminate the background. For the structure analysis, the intensity spectra were collected at normal and non-normal incidence ($\theta = 5^\circ, 6.5^\circ, 8.5^\circ$ and 10.5°) while the azimuthal angle φ was set either to 0° or 45° . Symmetry-equivalent beams were averaged, to make the following available for each coverage: (00), (10), ($\bar{1}0$), (11), ($\bar{1}1$), ($\bar{1}\bar{1}$).

Cu substrates, cut from a single crystal bar (1.3 cm^2) and oriented within 0.1° of the [001] direction by Laue diffraction, were cleaned by cycles of sputtering with Ne (3 keV, $2.5 \mu\text{A cm}^{-2}$) and annealing to 1000 K. The surface cleanliness was checked with Auger electron spectroscopy (AES). Scanning tunnelling microscopy (STM) reveals substrates with terraces larger than 400 Å (Schmid *et al* 1991).

The Co layers were evaporated onto the substrate from ovens heated by electron bombardment and equipped with mechanical shutters. The pressure in the chamber was typically in the low 10^{-10} torr range during evaporation. The deposition rate was 1–2 ML per minute and the thickness of the deposit was controlled by monitoring medium-energy electron scattering (MEED) intensity oscillations (Miguel *et al* 1989, Schneider *et al* 1990b) and cross-checked with AES (Gonzalez *et al* 1981, Miguel *et al* 1991). The symmetry of all LEED and MEED patterns recorded during Co growth was $p(1 \times 1)$ with

sharp spots and low background. The lattice parameter of the Co films in the plane parallel to the substrate surface is, then, visibly established by the Cu(100) lattice. Figure 1 depicts the schematic geometry of the system.

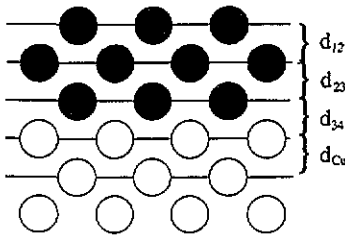


Figure 1. Schematic drawing of the geometry of Co (full circles) on Cu(100) (open circles). The Co atoms sit in the fourfold hollow site continuing the Cu lattice. d_{ij} indicates the spacing between layer i and j along the [001] direction.

3. Theory

Theoretical IV curves have been calculated by means of a multiple-scattering formalism (Pendry 1974, Van Hove *et al* 1979) and compared to the experimental curves using the Pendry R factor (Pendry 1980). A total of 101 beams were involved in the interlayer multiple scattering and up to eight phase shifts were included, calculated according to the Matheiss (1964) prescription from tabulated atomic wavefunctions (Herman *et al* 1963). Clarke *et al* (1987) have shown that Co atoms phase shifts can be obtained considering either a bulk HCP Co, or an FCC Cu structure, producing negligible changes in the R factor analysis. Accordingly, we did not try any further improvement of the phase shifts calculated considering the FCC Co structural parameters. The renormalized forward-scattering method (RFS) was chosen to treat the interlayer scattering, since it has been previously shown to be accurate enough for both Cu and Co layers (Clarke *et al* 1987). The exchange contribution was taken to be proportional to $\alpha\rho(\tau)^{1/3}$ with the α parameter taken from Schwartz (1972). Thermal effects were considered to be isotropic and we used a Debye temperature of 330 K for Co and 280 K for clean Cu. All R factor calculations included minimization with respect to the different interlayer spacings as well as the inner potential V_{or} . Since no clear trend was found when fitting this quantity individually in energy intervals, its value was kept energy independent in the analysis. Moreover, it is important to notice that there did not appear to be any important variation in V_{or} upon Co adsorption, and this is consistent with the fact that the metal's work function is not appreciably affected by this adsorption. With regard to the imaginary part of the self-energy, V_{oi} , the standard $E^{1/3}$ dependence was used (Van Hove *et al* 1979). The error bars of the structural parameters were estimated from the R factor isolines through the R factor variance calculated for each case as $\text{var}(R) = R_{\text{min}} \sqrt{8V_i/\Delta E}$ (Pendry 1980).

4. Results

Prior to the LEED analysis for the different Co coverages, the Cu(001) clean surface was characterized. This structure has already been published in the literature (Davis *et al* 1983) and we did not aim to re-determine the structural parameters associated with the multilayer relaxation of a Cu(001) surface. Rather, we employed this calculation as a test to set the accuracy of the structural determination. We obtained a Pendry R factor minimum of 0.21 over an energy range of 4013 eV for a top-layer contraction of -1.4%

($d_{12} = 1.78 \pm 0.02 \text{ \AA}$), a second-layer expansion of +0.8% ($d_{23} = 1.82 \pm 0.02$), and $d_{\text{bulk}} = 1.805 \text{ \AA}$, which is in good agreement with the known structure.

Experimental and calculated IV curves for the specular beam at an angle of incidence of 5° and several Co coverages are compared in figure 2. Changes in shape and energy position for the various coverages are easily detected. The Bragg peaks clearly shift to higher kinetic energies for increasing Co thickness. This qualitatively indicates that the average interlayer distance in the Co film is *smaller* than in the Cu crystal, i.e. the Co crystal is not growing in the strained FCC phase described above.

In order to be more specific we began with a structural search for the different coverages. The experimental data set for 1 ML of Co consisted of seven non-equivalent beams ($\Delta E = 2500 \text{ eV}$). As exemplified in figure 2, the curves measured for 1 ML of Co and clean Cu(100) are similar ($R_p = 0.26$). This illustrates the known fact that LEED spectra cannot very easily distinguish between elements of similar atomic numbers if they have the same crystal structure. Our structural search first considered the growth of a *perfect* monolayer of Co that covers uniformly the Cu substrate. In all calculations only the fourfold hollow site has been considered, since Clarke *et al* (1987) have provided strong evidence to discard the bridge and top positions. The corresponding analysis then yielded a Co–Cu perpendicular distance of $d_{12} = 1.78 \text{ \AA}$ and a second-layer value of $d_{23} = 1.81 \text{ \AA}$, i.e. a geometry indistinguishable within our error bars from the corresponding clean Cu(100), as if Co atoms would simply continue the lattice of Cu in pseudomorphic growth.

It has been believed for some time that the Co/Cu(100) system is a prototype example of layer-by-layer growth. This was first concluded from the observation of breaks in the slope of the Auger intensity curves against deposition time (Gonzalez *et al* 1981), and it was subsequently confirmed by a variety of experimental techniques, such as AES (Schneider *et al* 1990a), AES and LEED (Clarke *et al* 1987, Miguel *et al* 1991), MEED (Miguel *et al* 1989) and TEAS (Miguel *et al* 1991). There were, however, a number of pieces of experimental evidence pointing to a non-perfect growth of the first monolayer, most notably that the first oscillation in TEAS was smaller than the rest, and that CO titration experiments of the covering of the Cu substrate only agree within 20% with the first AES break (Gonzalez *et al* 1981, Miguel *et al* 1991). More recently, x-ray photoelectron diffraction (Li *et al* 1990) and grazing-incidence x-ray diffraction (Ferrer *et al* 1991) have suggested that for the first monolayer the growth mode deviates strongly from layer-by-layer, with the metal atoms forming two ML-high islands, which fully cover the substrate only at 2 ML.

To explore this possibility, we have simulated these islands by averaging with proportional weights the beams calculated for clean Cu with those calculated for two complete Co ML plus the ones calculated for one perfect ML. This procedure, in which the intensities are mixed rather than the amplitudes, should be valid provided that the islands are big enough and well separated to allow the incoherent mixing of the diffracted beams. The 2 ML island percentage was varied from 0–50%, and in all cases a significant reduction in the R factor values (down to $R_p = 0.22$) was obtained. The R factor variance clearly excludes an ideal covering for the first layer, but at the same time making unlikely the purely bilayer growth mode proposed by Li *et al* (1990) or Ferrer *et al* (1991). From our calculations the most likely island percentage is in the 20–40% range. Therefore, a substantial population of the second layer occurs before the first layer is completed. The distances obtained for the structure corresponding to the best R factor value given above are $d_{12} = 1.76 \pm 0.03 \text{ \AA}$ for the first interlayer spacing of the 2 ML islands, and $d_{23} = 1.78 \pm 0.03 \text{ \AA}$ for the second interlayer spacing, which was set equal to

the first interlayer spacing of the 1 ML high islands throughout the minimization process.

Our LEED results can also be understood in the light of recent STM observations (Schmid 1991), which give a real-space description of the density and characteristics of the islands at a level of accuracy not likely to be attained by LEED. From the analysis of these data, it seems that the island density at 1 ML of Co is $6 \times 10^{12} \text{ cm}^{-2}$. Up to $\frac{1}{3}$ ML the islands have only a monolayer height. At 1 ML, around 20% of the deposited material has condensed on top of the first-layer islands, with the same percentage of the substrate still uncovered. In this way, the 1 ML film can be considered granular with islands of some nm diameter separated by 1 nm gaps.

The analysis of the data set for nominally 2 ML, which consists of seven beams spanning an energy range of 1851 eV, indicates that the overlayer is basically perfect at this stage of growth. This conclusion is obtained after analyzing the consistent growth of the R factor from the minimum value at 0.22, for no islands present in the surface, to a value of 0.26 for a fully forming islands model; the latter value is excluded by the R factor variance of 0.03. This is also confirmed by STM images showing that the formation of third-layer islands only rarely occurs before the second layer is near completion (Schmid 1991). For the analysis, the Cu-Cu spacing was kept fixed at 1.805 Å and d_{12} and d_{23} for the Co overlayer were changed by 0.01 Å steps from 1.66 to 1.80 Å. The optimized interlayer spacings are $d_{12} = 1.77 \pm 0.02$ Å and $d_{23} = 1.73 \pm 0.03$ Å, with the minimum R factor value mentioned above. The degree of agreement between theory and experiment can be judged visually from figure 3.

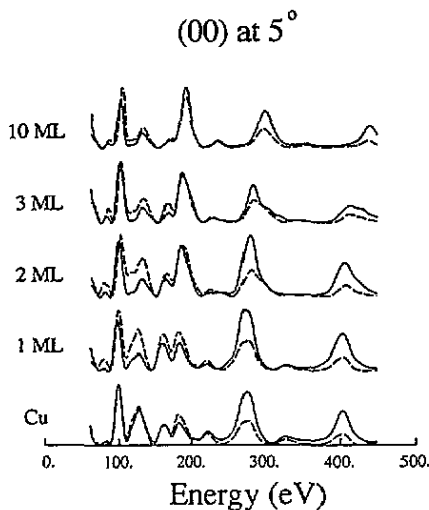


Figure 2. IV curves for the (00) beam at a polar angle of incidence of $\theta = 5^\circ$, $\varphi = 45^\circ$ and Co coverages ranging from 1–10 ML (experiment: full curve; theory: broken curve).

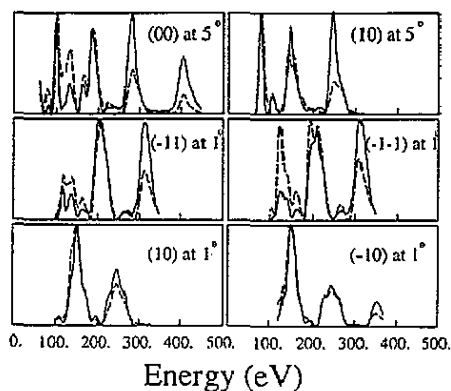


Figure 3. IV spectra for several beams ($\theta = 5^\circ$ and $\theta = 1^\circ$, $\varphi = 45^\circ$) recorded for 2 ML Co/Cu(100). Experiment: full curve; theory: broken curve.

It is remarkable that d_{12} is larger than d_{23} , showing an outward relaxation that we are not used to in metals. It has been shown that, due to the coverage dependence of the Curie temperature, films of 2 ML just reach T_c near room temperature (Schneider *et al* 1990, Kämper *et al* 1992). In fact, while 1 ML is not ferromagnetic at room temperature, magnetization settles in when the film reaches 2 ML. One may speculate that the expansion in d_{12} is related to a magnetic effect at the surface. In fact, it seems

to be established by theoretical calculations (Janak *et al* 1976) that an increased lattice constant is associated with an increased magnetization, and we speculate that, *vice versa*, the magnetization may produce a sort of magnetic *pressure* that increases the interlayer spacing.

The R factor analysis for the experimental data (six beams ($\Delta E = 2155$ eV)) for 3 ML included variations of the three interlayer spacings in the Co overlayer, while the Cu–Cu distance was again set to 1.805 Å. The optimized perpendicular distances were found to be $d_{12} = 1.76 \pm 0.02$ Å, $d_{23} = 1.74 \pm 0.02$ Å and $d_{34} = 1.76 \pm 0.03$ Å, yielding a Pendry R factor minimum of 0.23.

Due to the limited escape depth of electrons in the energy range considered here, the LEED IV curves for 5 ML (five beams, $\Delta E = 1706$ eV) and for 10 ML (five beams, $\Delta E = 1823$ eV) were found to be highly similar, the Pendry R factor between them being 0.23. The calculation for these two coverages was carried out varying the first three interlayer spacings from 1.60 to 1.80 Å in steps of 0.01 Å, while assuming that the other Co layers kept the same perpendicular value, d_{Co} , varied within the same limits. Figure 4 shows the R factor contour map corresponding to this case. A common R factor analysis yielded the following values for the structural parameters: $d_{\text{Co}} = 1.73 \pm 0.03$, $d_{34} = 1.73 \pm 0.03$, $d_{23} = 1.74 \pm 0.03$ and $d_{12} = 1.72 \pm 0.03$, corresponding to an R factor minimum of 0.24. Thus, the interlayer spacing is not in agreement with the predictions for strained FCC Co (1.805 Å), and neither is it in agreement with the relaxed FCC Co (1.774 Å). However, these values are in good agreement with those reported by Clarke *et al* (1987) for a nominal 8 ML thick Co film on the basis of a smaller experimental data set. The 4% tetragonal compression of the interplanar distance clearly established by the LEED analysis has a measurable influence on the electronic structure of epitaxial Co films. It is responsible for moving the minority spin band away from the Fermi level with respect to band structure calculations for undistorted bulk FCC Co (Schneider *et al* 1990a, Schneider 1990).

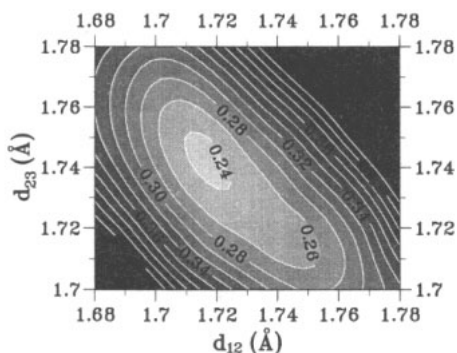


Figure 4. Pendry R factor contour map for the common analysis of 5 ML and 10 ML against d_{12} and d_{23} .

For 10 ML of Co/Cu(100) we have also calculated the IV spectra for unstrained relaxed FCC Co. Figure 5 reproduces the experimental IV curve for the (00) spot at 5°, as well as calculated curves for the optimized tetragonal phase ($d_{\parallel} = 2.55$ Å, $d_{\perp} = 1.73$ Å) and the hypothetical relaxed FCC phase where the Co film has its own lateral and vertical distances ($d_{\parallel} = 2.50$ Å, $d_{\perp} = 1.774$ Å). The visual agreement is much worse for this latter case, a fact that is confirmed by the poor value of the R factor ($R_p = 0.37$).

The fact that the Co(100) film grows with the same lateral parameter as Cu(100) is further confirmed by the growth of Cu on one of the described thick Co films. Figure 6 shows IV spectra of the (11) beam for clean Cu(100), a 5 ML-thick Co film grown on

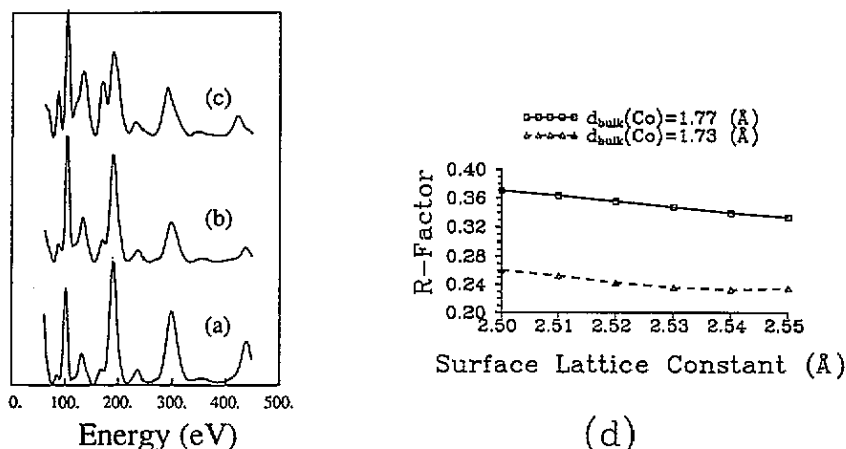


Figure 5. (a) Experimental IV spectra for 10 ML Co/Cu(100); (b) calculated IV spectra for FCC Co, ($d_{\parallel} = 2.55 \text{ \AA}$, $d_{\perp} = 1.73 \text{ \AA}$); (c) calculated IV spectra for FCC Co ($d_{\parallel} = 2.50 \text{ \AA}$, $d_{\perp} = 1.77 \text{ \AA}$); (d) the Pendry R factor for these cases against d_{\parallel} .

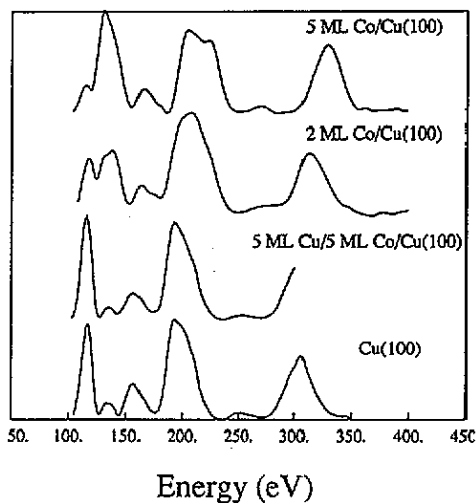


Figure 6. Experimental IV spectra for the (11) beam at normal incidence for different coverages.

Cu(100) and a 5 ML-thick Cu film grown on the 5 ML Co film. The optimized structure for the upper Cu(100) film was found to agree well with the pure Cu one ($d_{12} = 1.77 \text{ \AA}$, $d_{23} = 1.83 \text{ \AA}$, $d_{\text{Cu}} = 1.805 \text{ \AA}$, $R_p = 0.14$, $\Delta E = 400 \text{ eV}$), confirming the fact that Cu grows on top of Co(100) with the lateral and vertical spacings of pure Cu. This is possible thanks to the coherent growth of the tetragonally compressed Co overlayer. Of course, the high surface sensitivity of LEED does not allow us to detect the persistence of the tetragonal distortion in Cu-covered Co films. Recent EXAFS data confirm its persistence in sandwiches and single-crystal superlattices grown by MBE on Cu(100) (Cebollada *et al* 1989), an important fact in understanding their fascinating magnetic properties (Miguel *et al* 1991).

Information on the equilibrium (zero-pressure) lattice parameter of FCC Co can be obtained from the measured tetragonal distortion and the Poisson ratio of Co. Under epitaxial strain, biaxial expansion in this case, the ratio of parallel strain ($\Delta a/a$) to perpendicular strain ($\Delta c/c$) is related to the Poisson ratio by

$$\frac{\Delta a/a}{\Delta c/c} = \frac{(a_{\text{meas}} - a)/a}{(c_{\text{meas}} - c)/c} = \frac{1 - \nu}{2\nu}$$

where $a_{\text{meas}} = 3.62 \text{ \AA}$ and $c_{\text{meas}} = 3.47 \text{ \AA}$ are the lattice parameters of FCT Co and $a = c$ is the lattice parameter of FCC Co. Since the value of the Poisson ratio for Co is 0.31, one obtains $a = 3.543 \text{ \AA}$, in excellent agreement with the lattice parameter (3.548 \AA) determined by x-ray diffraction for powders of FCC Co (Owen *et al* 1954).

To summarize, Co films grown at room temperature on Cu(100) have been found to have a tetragonally distorted FCC structure with $a = 3.62 \text{ \AA}$ and $c = 3.47 \text{ \AA}$. A smaller, but otherwise similar, tetragonal distortion ($c = 3.54 \text{ \AA}$) has been recently reported in a LEED study of 12 ML films of FCC Fe on Cu(100) (Lu *et al* 1989).

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