ANALYSIS AND MODIFICATION OF AMORPHOUS AND PARTIALLY-CRYSTALLINE THIN FILMS

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ABSTRACT

Thin films of light atomic weight elements in amorphous, partially-crystalline, or crystalline forms have applications in a broad range of technologies. For example, amorphous tetrahedral carbon (a-tC) and polymeric thin films impact electronic materials technology as electron- and light-emitting device elements, respectively. A lack of crystallinity introduces complexity in the experimental and theoretical characterization of these materials but is not necessarily a limiting factor in their performance. While the growth process is clearly a major factor governing the physical properties of a film, interactions with the substrate are also important, so surface and interface analysis provides an important complement to bulk measurements. Currently, the fundamental and applied aspects of the atomic, electronic and vibrational structure of these complex materials are being elucidated by novel approaches combining several experimental techniques with theoretical calculations. This paper focuses on several approaches in the characterization and modification of thin films made possible by recent experimental advances. The structural and electronic properties of two model systems are considered as examples: a-tC thin films grown by pulsed laser deposition (PLD) and polyaniline thin films grown by vapor deposition. First, scanning probe microscopies and x-ray scattering are used to investigate the structural aspects of a-tC films as a function of PLD growth conditions. The possible connection of nanoscale surface modification and characterization with electron emission properties will be discussed. Second, the results of inelastic scattering spectroscopy and other surface techniques will be discussed to obtain information on both interfacial aspects of the growth of polyaniline thin films and microscopic and macroscopic aspects of electrical conductivity upon doping. Comparisons will be made with other studies that address properties of analogous crystalline systems as appropriate. A brief assessment of the broader problem of analyzing these systems will be given.

INTRODUCTION

Thin carbon-based films, including diamond-like carbon and polymer materials, have applications in a broad range of technologies. Key questions regarding the physical properties of thin films originate with the interface between substrate and film, and extend to the relationship of the properties of the thin film to that of the bulk material and to the surface properties of the film. The surface properties of the substrate can affect growth to varying degrees. The surface of the film controls or contributes to chemical reactivity, tribological properties, and electron emission and is accessible to surface spectroscopies. The buried interface between the film and the

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substrate is much less accessible. A central issue regarding the behavior of materials systems is the interplay between the microscopic and macroscopic properties. Obtaining as complete a picture of a thin film system as possible relies on an integrated approach in which multi-technique analysis is used to measure surface, bulk, and interfacial properties.

The structural, electronic, and vibrational properties of thin films grown on various substrate materials is dependent on a large number of variables, including the substrate material and preparation, the growth process itself, and specific processing conditions. Much of the progress in thin film analysis has come from the use of surface science techniques where growth is monitored at various stages. In many instances, as for epitaxial films, the use of in-situ diagnostics, such as electron diffraction, provides a means to monitor the evolution of the film layer-by-layer. Non-crystalline or partially-crystalline films present a greater challenge. Once the film is grown, spatially-localizing the boundary layer between film and substrate is difficult. Approaches such as cross-sectional electron microscopy, while destructive, are extremely useful in getting a picture of the thin-film – substrate interface even to the sub-nanometer level. X-ray scattering can non-destructively probe surface, bulk, and interface structure for comparison to theoretical models.

In this paper, we discuss the analysis of the surfaces and interfaces of technologically-relevant non-crystalline materials – amorphous tetrahedral carbon (a-tC) films grown by pulsed laser deposition and vapor-deposited polymers. The application of scanning tunneling microscopy (STM), scanning force microscopy (SFM), and high resolution electron energy loss spectroscopy (HREELS) are extended from conventional surface science systems. The work demonstrates the usefulness of various combinations of electron microscopy and spectroscopy with high energy or spatial resolution. We describe studies of the surface structure of a-tC that indicate thin film microstructure, the simulated conditioning of a surface which we relate to other studies in which electron emission is observed, and near-surface vibrational structure applied to the growth of polymer thin films.

EXPERIMENT

Composition, structure, electronic excitations, vibrational modes of thin films have been measured with a variety of techniques including STM, SFM, HREELS, scanning electron microscopy (SEM), and spatially-resolved electron energy loss spectroscopy and Auger electron spectroscopy (EELS and AES, respectively). The techniques are described in more detail in the next section.

a-tC films were grown by pulsed laser deposition (PLD) as described previously [1]. PLD was performed with 248 nm radiation from a KrF excimer laser incident on a rotating graphite target in an ambient pressure of $\leq 10^{-7}$ Torr with a substrate temperature of $\leq 50°C$.

Polyaniline films were grown by vapor-deposition onto an atomically-clean Cu(110) surface in ultrahigh vacuum ($\leq 3 \times 10^{-10}$ Torr). The polymer evaporation source is a resistively-heated quartz cell filled with emeraldine base powder housed in a preparation chamber attached to the main HREELS chamber. After outgassing the source, the Cu surface was exposed to the vapor at $\sim 10^{-8}$ Torr.

2

RESULTS AND DISCUSSION

Amorphous tetrahedral carbon films

Amorphous tetrahedral carbon films grown by pulsed laser deposition [2] (or filtered arc deposition [3]) are composed of networks of primarily three-fold and four-fold coordinated carbon atoms [4]. Such a description refines the view of considering the films as composed of structures that are purely sp^3 (diamond-like) or purely sp^2 (graphitic). On the average, the ratio of four-fold to three-fold species is dictated by details of the growth parameters. For example, analysis of PLD-grown films with Raman spectroscopy shows that growth at low laser energy densities increases the proportion of three-fold coordinated carbon [1]. In addition, growth geometry-related parameters, such as the position of the laser-induced plume with respect to the substrate, are also important. On a microscopic scale, it might be expected that the films are inhomogeneous because interfacial mixing, interfacial strain, and the growth dynamics themselves can result in vertical and lateral variations in atomic densities.

Besides the bulk properties, the surface properties of a-tC films are very important technologically in light of potential applications as cold-cathode electron emitters and as tribological coatings. One consideration, then, is to investigate the nanometer-scale structure of a-tC films in the surface region as a function of growth conditions and after *ex-situ* etching is performed. Thus, the object of our initial studies was to assess the surface structure of PLD-grown films with scanning force microscopy (SFM). The experiments were performed using intermittent-contact SFM in air.

The use of a-tC films as electron emitters has been demonstrated in several studies [5-7]. It has been noted that, in order to initiate electron emission from a-tC films, a conditioning process is typically required [7]. This involves stressing the material with an electric field, i.e., applying a potential difference between the cathode and anode which is greater than the field subsequently required to obtain emission. Talin, et al. observed discharges and the formation of craters in some instances during the initial application of the electric field [6]. More recently, Missert, et al. demonstrated that a reduced rate of increasing the electric field can induce electron emission without any observable morphological damage; emitting regions were identified by SEM as areas exhibiting reduced secondary electron emission [7]. Scanning Probe Microscopy is a rapidly-developing field that permits not only structural, force, electronic, and magnetic characterization but also manipulation on the nanometer scale [8]. We have taken advantage of some of these capabilities in combination with spatially-resolved electron spectroscopy to simulate the conditioning process and characterize the a-tC surfaces.

Surface structure of PLD-grown a-tC films

In our first series of studies, the nanometer-scale surface morphologies of a-tC films were compared as a function of laser energy density and surface treatment [9]. Initial SFM images of as-prepared films revealed little because the surfaces were found to be extremely smooth, exhibiting a root-mean-square roughness of ≤1 nm. No reproducible surface features were observed in these images. Considering the fact that the films are composed of mixtures of three-fold and four-fold coordinated carbon species, we proceeded to study the effects of three preferential etching processes designed to remove the remnants of "graphitic" carbon and leave behind "diamond-

3

like" species. The etching methods – exposure to an oxygen plasma, annealing in air, and wet-chemical etching in a chromic-sulfuric acid mixture are known to etch graphitic carbon at a faster rate than diamond for crystalline materials. When applied to a-tC films, the study specifically addressed the question of etching the surface of an amorphous film with non-uniform bonding configurations of carbon atoms.

SFM analysis of films subjected to the three etching methods showed an increased the surface roughness; this resulting from randomly-distributed cluster-like structures. All three methods produced similar results, but oxygen plasma etching was found to be the most straightforward and effective. Figure 1 shows SFM images of three ~140 nm thick films grown with energy densities of 46, 27, and 11 J/cm^2, respectively. These image were acquired after all three samples were subjected to a ~4 min oxygen plasma etch simultaneously. These images show that the underlying film consists of random arrays of nanoscale clusters with in-plane diameters ranging from ~10 nm to ~25 nm. The films grown at high laser energy density exhibit smaller and better defined structures after etching than those grown at lower energy density. This is likely related to the relative proportions of three-fold and four-fold coordinated carbon in the films
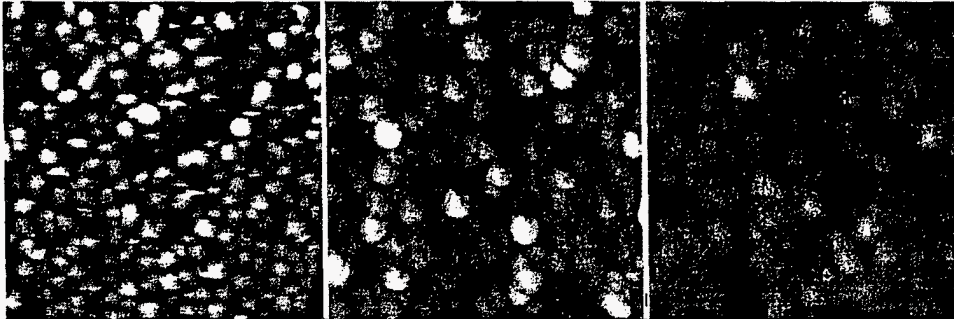

Figure 1: 250 × 250 nm^2 SFM images of PLD-grown a-tC films after O-plasma etching. The laser energy densities for growth are: left: 46 J/cm^2, center: 27 J/cm^2, right: 11 J/cm^2.

We might conclude that these PLD-grown a-tC films exhibit structural inhomogeneity indicating agglomeration of higher density and lower density carbon on the scale of tens of nanometers. That is, the clusters observed are regions of predominantly four-fold coordinated carbon that were embedded three-fold coordinated carbon prior to etching. The fact that we are presently unable to ascertain the bonding hybridization directly in these clusters prevents a more definitive conclusion. However, it should be noted that early first-principles molecular dynamics simulations of amorphous carbon predicted the evolution of dense sp^3 clusters embedded in an sp^2 background [10]. Furthermore, previous PLD-growth studies using a longer wavelength laser identified larger nodular structures [11].

Simulation of the conditioning process for emission in a-tC films

The nature of the emission process from a materials viewpoint, including the effect of the conditioning process on the surface of the a-tC film, is being studied by several groups. Recently, we attempted to simulate the electric field conditioning effect by using the spatially-localized electric field from a STM tip to both stress and subsequently view the surface of a PLD-grown a-tC film in air and in vacuum. The in-

air measurements were made with a Digital Instruments NanoScope III Scanning Probe Microscopy system and in-vacuum measurements were made with a custom-built ultrahigh vacuum STM. The films were lightly oxygen plasma etched prior to study. For the samples stressed in air, the analysis was continued with intermittent-contact and non-contact SFM to image structure and surface potential changes. Following this, a Perkin-Elmer Scanning Auger Microprobe was used to perform SEM to locate the modified region, spatially-resolved AES to rule out transfer of metal atoms from the tip, and spatially-resolved EELS to determine changes in carbon bonding hybridization.

Figure 2a shows the result of producing an array of nanometer-scale structures on a 250 Å-thick a-tC film with an STM tip. The pattern was formed after ramping the tip-sample bias from -10 V to +10 V at each point on a grid. By monitoring the I-V profiles during the voltage ramp, increased conductance between tip and sample was observed at grid points where modification was subsequently identified. The modified region was then re-positioned under a SFM probe for structural analysis. The agreement of the SFM image indicates that a structural modification occurs, i.e., not solely a change in electronic structure which could, in principle, produce contrast by itself in STM. Similar structural changes occur for modifications performed in ultrahigh vacuum as viewed by STM.
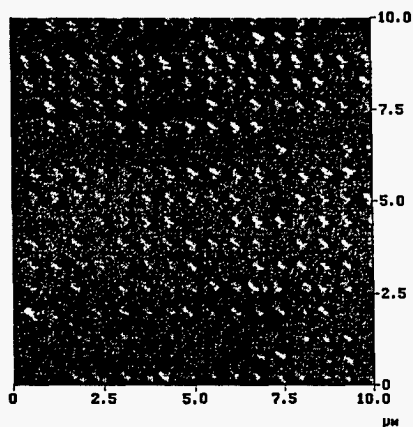

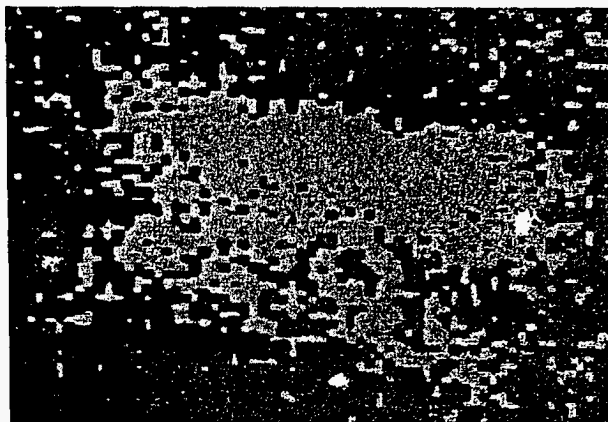Figure 2a: 10 × 10 μm² STM a-tC surface after simulated conditioning.

Figure 2b: Overlay of difference map of plasmon intensities acquired by spatially-resolved EELS over SEM image. The modified region is dark (reduced secondary emission). The light gray area highlights areas of three-fold coordination .

We note that a small surface potential variation is also observed at many grid points, some of which do not exhibit a structural change. This indicates that weaker electric field stresses can change surface electronic structure without producing a large structural change. The small magnitude of the surface potential variation (tens of millivolts) observed, while indicating some change of surface electronic structure cannot be further characterized because of the in-air analysis. The near-surface carbon hybridization was probed with EELS and SEM. Figure 2b is an overlay of an SEM image and a spatially-resolved EELS images. In the figure, reduced secondary electron emission identifies the conditioned region [7]. The EELS spectra of the conditioned regions showed plasmon excitations centered at ~26 eV while those outside the region gave losses centered at higher energy (~28 eV). The light gray area

defines the regions where the plasmon peaks at ~26 eV. We therefore conclude that we have created regions with an increased proportion of three-fold coordinated carbon near the surface.

The approach we have taken is to use a nanometer scale probe to both electrically stress and characterize the films. The results indicate that changes in the film structure and coordination do occur when a sufficiently high electron flux is produced. We infer that these observations are related to the conditioning process for which emission has been carefully studied [7]. With SEM analysis only, structural changes on the scales we have measured would not be visible.

Further exploration of the fundamental processes related to conditioning and cold-cathode emission would be enhanced by adopting a surface science strategy for analysis. This would incorporate growth and measurement facilities combining scanning tunneling microscopy and spectroscopy, SFM, SEM and spatially-resolved EELS, Raman spectroscopy and HREELS in connected vacuum chambers so as to assess geometric, electronic, and vibrational structure in a clean environment under identical surface conditions.

Vibrational and electronic excitations at surfaces and in thin films using HREELS

Vibrational spectroscopy is a key experimental methodology for the measurement of chemical bonding in materials. Vibrational spectra produce the normal vibrational modes of interatomic bonds permitting, in principle, the identification of the composition of a material. Assessment of the distribution of bond configurations in bulk amorphous materials, such as a-tC, has almost exclusively relied on Raman spectra as a diagnostic. A major disadvantage of Raman spectroscopy as typically used with visible excitation wavelengths is the fact that the excitation is resonant with the (electronic) π-π^* transition in the three-fold coordinated moiety, generating a disproportionate response between the three-fold coordinated and four-fold coordinated species and severely complicating the analysis. Recent reports have demonstrated that ultraviolet Raman spectroscopy removes the resonant behavior putting both moieties on an equal footing [12].

Vibrational spectroscopy at or near surfaces can provide direct information on surface chemical bonding and reactivity. Inelastic electron scattering, or HREELS, provides surface sensitivity for surface measurements [13]. Depending on the interaction mechanism, the surface sensitivity can vary over the range ~5-100 Å depending on the electron impact energy and penetration of the electric field of the electron. This technique has developed over the last twenty-five years to the point at where energy resolution comparable to optical techniques is now possible. Two of the advantages of the technique are the wide spectral range, from the far-infrared to the electronic regime allowing the simultaneous measurement of vibrational and electronic excitations, and multiple interaction mechanisms. One of the disadvantages of the technique is the need to operate in ultrahigh vacuum. The surface-sensitive HREELS technique provides a complement to bulk-sensitive infrared, Raman and optical absorption spectroscopies which can also be configured to increase surface sensitivity. We note that interface-specific vibrational spectroscopy is a means to understand local bonding at the abrupt transition between materials – a sort of atomic view of adhesion.

Although HREELS is typically used to study excitations at surfaces of well-characterized crystalline materials prepared and processed in ultrahigh vacuum, recent work on a-tC materials and polymer thin films show the usefulness of the technique for systems of increased complexity.

For a-tC materials, Lopinski and Lannin have recently studied the vibrational [14] and sub-band gap spectra [15] of *in-situ* PLD-grown a-tC films . Vibrational spectra were used to assess the relative contributions from three-fold and four-fold coordinated carbon as a function of probe depth. The phonon density of states for these forms of carbon was obtained using HREELS measurements of sputtered carbon (three-fold coordinated) and neutron scattering measurements of diamond (four-fold coordinated) as references. Matrix-element-weighted surface-sensitive impact scattering data suggested 60% four-fold coordinated carbon at the surface of their films.

The growth and structure of polymer thin films is also of considerable technological interest. Bulk optical spectroscopies, which routinely exhibit high resolution, exhibit little broadening due to disorder and distinct vibrational modes. For an HREEL spectrometer, the challenges are to perform at high enough resolution to resolve the close-lying vibrational modes of an organic film while maintaining that resolution even with surface disorder and inhomogeneity. With a new-generation HREELS spectrometer [16], we have succeeded in performing a detailed analysis of the initial stages of the *in-situ* growth of polyaniline by vapor deposition in ultrahigh vacuum on Cu [17] and Ag [18] surfaces. Polyaniline is a model conducting polymer with conductivity that can be changed with doping. The growth of thin-film polyaniline on a well-characterized substrate is interesting here from a fundamental point of view, but it is desirable to assess as much as possible the role of the substrate in the polymerization and quality of the resulting film. Furthermore, the electronic excitation spectrum can produce a direct signature of conduction by the observation of plasmon excitations as a function of doping. The results, as described below demonstrate that it is possible to identify interface modes and assess substrate-mediated polymerization, both microscopic phenomena, while also addressing some aspects of macroscopic conductivity of these films upon *ex-situ* doping.

Initial stages of growth of polyaniline thin films

The use of HREELS to study vibrational and electronic spectra of polymer surfaces was first demonstrated on spin-cast polyimide films [19] and metal-polyimide interfaces [20]. Due to limitations of energy resolution, this early work could only focus on the analysis of a subset of vibrational modes. However, it was concluded that no intrinsic limitations exist to prevent the achievement of higher resolution with improved instrumentation. More sophisticated electron-optics design is incorporated into the design of the present LK-3000 HREEL spectrometer so that energy resolution of ≤ 16 cm^{-1} is achievable. We focus here on the *in-situ* growth of polyaniline on a Cu(110) surface by vapor deposition. The unique combination of high energy resolution (2-3 meV), high throughput, and broad spectral range from the far-infrared to the ultraviolet-visible spectral regions has provided a new picture of the interaction of ultrathin films with a surface as a function of thickness.

Figure 3 illustrates a HREELS spectrum of a vapor-deposited ≤ 100 Å polyaniline film on a Cu(110) surface. The rich spectrum in Figure 3 and other sets of spectra

acquired for different vapor exposures permit a very detailed vibrational analysis of the growth of the film as a function of thickness, as given elsewhere [21].
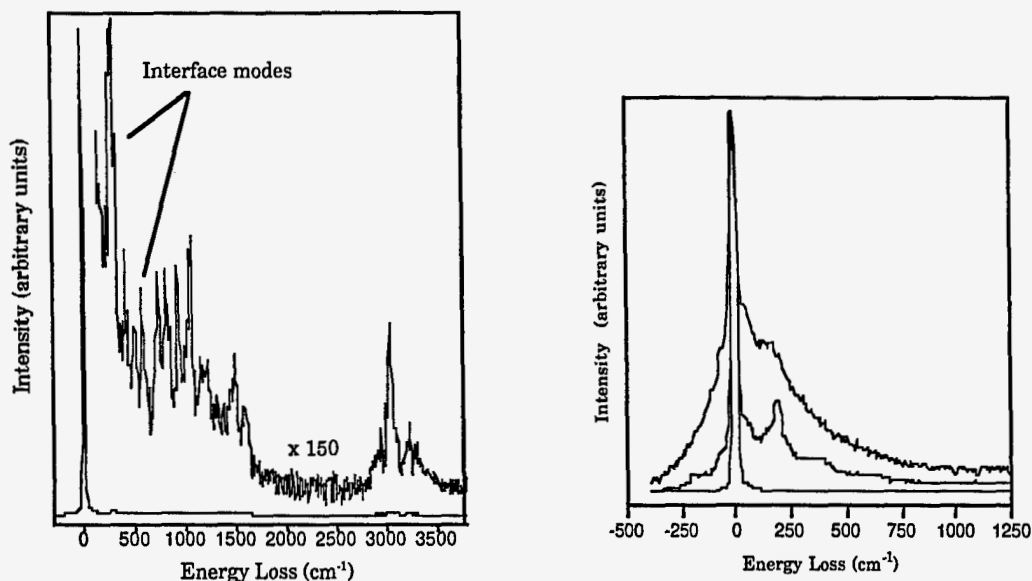

Figure 3: HREEL vibrational spectrum for a ≤100 Å polyaniline film vapor-deposited on a Cu(110) surface at room temperature.
Figure 4: HREEL spectra for ≤100 Å polyaniline films. Bottom: undoped; Middle: HCl-doped T=125 K, Top: HCl-doped T=300 K.

Analysis of the relative intensities of CH out-of-plane bending modes related to monosubstituted and paradisubstituted benzene rings indicates that the initially-adsorbed oligomers from the vapor are about three monomer units in length; a polymerization reaction occurs upon increased emeraldine exposure to form higher molecular weight species. This suggests that the oxidation state of the thin film polymer is that of the starting emeraldine powder and simultaneously-acquired electronic excitation spectra support this conclusion. Two new interfacial vibrational modes at ~295 cm^{-1} and ~585 cm^{-1} as shown in Figure 3 are observed, and these are attributed to the bonding of a fraction of the polyaniline chains to the surface through amino groups, as we found for aniline/Cu(110) [22].

Figure 4 shows that *ex-situ* doping with HCl produces major changes including a broad tail and a new loss at ~190 cm^{-1}. Based on optical studies of polyaniline films, the discrete far-IR loss is assigned to a plasmon originating from free electrons distributed across ordered and disordered regions of the material [23]. The appearance of this plasmon indicates that the film is highly conducting due to ordering of the polymer chains near the polymer-metal interface. In contrast, spectral analysis of ≥1000 Å films indicate that, compared to the ≤100 Å films, the surface region becomes highly-reduced with shorter average conjugation lengths due to cross-linking. Doping the thicker films does not produce a resolvable plasmon loss indicating a lower conductivity.

This work represents the one of the first analyses of polymer structure as a function of thickness by vibrational spectroscopy and the first direct observation of a plasmon loss of the doped ultrathin (≤100 Å) polyaniline film. We conclude that the substrate plays a major role in the quality of the film growth.

8

CONCLUSION

We have discussed studies that approach the problem of non-crystalline thin films by using multiple techniques to assess and control nanometer-scale structure and measure interatomic vibrations with great detail. We have obtained major results related to a-tC surface structure upon etching and simulated surface conditioning and in polymer thin film growth. A limitation of such a multi-technique approach is that growth and analysis instrumentation are typically not available within a contiguous system. This is particularly evident for the a-tC films grown in a high-vacuum environment with subsequent transfer to other facilities for analysis. The information obtained from the more fundamental polyaniline work is obtained with fewer uncontrolled parameters because growth and analysis are done *in-situ*; only doping is performed *ex-situ*, but control experiments indicated that the ambient environment did not alter the films. The future direction of these types of studies is expected to incorporate multi-technique analysis with an approach familiar to experimental surface science so as to achieve a higher level of experimental control and accuracy.

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