A SCANNING TUNNELING MICROSCOPIC (STM) STUDY OF THE SURFACE TOPOGRAPHY OF PLASMA-DEPOSITED NANOCRYSTALLINE SILICON

J.K. Gimzewski, A. Humbert*, D.W. Pohl
IBM Zurich Research Laboratory, 8803 Rüschlikon, Switzerland

and

S. Vepřek
Institute of Inorganic Chemistry, University of Zurich, 8057 Zurich, Switzerland

ABSTRACT

Scanning Tunneling Microscopy (STM) was used to study the surface topography of atomically-clean nanocrystalline silicon deposited at a floating potential and under ion bombardment. Nanometer scale roughness is determined by crystallographic parameters. Sharp grain boundaries are resolved. The data show a good correlation with TEM lattice images and x-ray crystallographic data. The importance of STM as a powerful tool for the investigation of plasma chemically-processed surfaces is demonstrated.

1. INTRODUCTION

Plasma processes used for deposition, etching or modification of materials offer new and interesting opportunities in surface and thin-film preparation. Properties such as surface reactivity, wear resistance, adhesion and interfacial characteristics ultimately relate to the local environment of the surface atoms and the surrounding clusters that form the bulk. Until recently, the investigation of this relationship was limited to non-local methods or by the resolution of microscopic techniques (e.g., SEM and carbon replica-TEM).

The recent development of Scanning Tunneling Microscopy (STM) by Binnig, Rohrer, and co-workers [1] has opened the way for investigation of real-space three-dimensional imaging of surfaces with atomic resolution. Applications of STM to plasma-chemical processing of surfaces should give a new insight into deposition, etching, nucleation, film growth, and other related problems on an atomic scale.

Here, we present the first experimental investigation of plasma-processed surfaces by this new method. We have chosen nanocrystalline silicon (nc-Si) because of the high degree of materials' property control that can be achieved through careful selection of plasma parameters [2]. Further details on the preparation and properties [3] are reported elsewhere.

*On leave from CNRS, UA 783, University of Marseilles, Luminy, 13288 Marseilles, France.
2. PRINCIPLE OF STM OPERATION

The principle of STM is based on the tunnel effect. An atomically-sharp tip is positioned \( \sim 1 \) nm from the sample surface. Under the influence of an applied voltage, \( V_t \), a tunnel current, \( j \), flows from the tip to the sample or vice versa owing to overlap of the wave functions of tip and surface where \( j \propto V_t \exp[-A\phi^{1/2}S] \). Here \( A \) is a constant, \( \phi \) is the tunnel barrier height, and \( S \) is the tip-sample separation. To record an STM picture, \( j \) is maintained constant while the tip is scanned in the horizontal planes. This is achieved by use of an x-y-z piezo-electric position controller and feedback circuit. The surface topography which reflects contours of constant wave-function overlap is recorded by plotting the voltages applied to the \( X, Y \) and \( Z \) piezo-electric elements. Further details of the technique are given in [1].

3. EXPERIMENTAL

Details of sample preparation are described elsewhere [4]. Samples were deposited at a floating potential and under a bias potential of -100 V. The STM used in this work is similar to the design of Binnig, Rohrer and coworkers [1]. STM graphs were recorded at \( V_t \approx +0.5 \) V and \( j = 10 \) nA using a tungsten tip. Sample transfer to the STM from the deposition apparatus was done through air in approximately seven minutes. X-ray photoelectron spectroscopy was used to show that no carbon or oxygen contamination is introduced by this procedure owing to the unique passivation of the surface by the plasma [5]. STM measurements were conducted under a vacuum of \( \sim 10^{-8} \) mb.

4. RESULTS AND DISCUSSION

In Figure 1, X-ray diffraction was used to determine the dependence of crystallite size as a function of bias potential, \( V_b \) [4]. At a floating potential (\( V_b = 0 \)), the crystallite size is \( \sim 10 \) nm. Figure 2 shows a typical STM graph of an area of the surface: sharp boundary regions separating flat areas of between \( \sim 5 \) and \( 15 \) nm are clearly resolved. Based on the correlation in size to the x-ray diffraction data, we interpret the graphs as direct observation of nanocrystallites at the surface. Furthermore, the shape and size of the crystallites exhibit excellent similarity with TEM images of the (111) lattice planes of similar samples [3].

The widths of the grain-boundary regions are close to the resolution limit of the present data (< 1 nm) consistent with previous work showing that the grain boundaries contain essentially no amorphous network [3,4].

The STM graphs show in a direct manner that the crystallite size plays a determining role on the surface roughness on the nanometer scale. The estimated vertical corrugation resulting from the nanocrystallites varies between \( \sim 1-6 \) nm.
Structural and other modifications resulting from deposition under a negative bias (ion bombardment) have been studied in detail [4, 6]. SEM reveals that granular roughness present on 'floating' samples in the μm scale disappears under bias resulting in mirror-like surfaces. Figure 3 shows a typical STM graph for a sample deposited under a bias potential of -100 V. Here, X-ray diffraction data (see Figure 1) gives a crystallite size of ~ 3 nm. From Figure 3, we estimate a crystallite size of ~ 3-6 nm in reasonable agreement with the diffraction data. The crystallites also appear more rounded than at floating potential, although the vertical corrugation induced by the crystallites is smaller (1-3 nm). (Figures 2 and 3 have different vertical scales.) Noting that ≥ 50% of the atoms are on the surface or in grain boundaries, finite crystallite size effects [7] or ion bombardment may be responsible for the rounding of crystallites.

Fig. 1. Plot of crystallite size vs negative substrate bias, $V_b$, determined from X-ray diffraction data [4].
Fig. 2. STM graph of nc-Si deposited at $T = 260^\circ C$ under a floating potential. Divisions on axes correspond to 5 nm.

Fig. 3. STM graph of nc-Si deposited at $T = 260^\circ C$ under a negative substrate bias, $V_b = 100$ V (ion bombardment). Divisions on axes correspond to 5 nm.
5. CONCLUDING REMARKS

Scanning Tunneling Microscopy has been applied to plasma-surface chemistry through the real-space observation of nanocrystallites for plasma-deposited nc-Si. The information extracted from the STM pictures has been correlated with crystallographic data. For nc-Si deposited at a floating potential and under ion bombardment, sharp grain-boundary regions are observed. The nanometer-scale roughness of the films is determined by the crystallite size. Simultaneous ion bombardment is shown to reduce the overall vertical roughness while increasing the lateral corrugation owing to the smaller crystallite size.

REFERENCES