

Measuring the thickness of ultra-thin diamond-like carbon films

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Abstract

This paper examines the challenge posed by the measurement of thickness of sub-50 nm diamond-like carbon (DLC) films deposited onto silicon substrates. We compared contact profilometry (CP), optical profilometry (OP), contact atomic force microscopy (CAFM), tapping atomic force microscopy (TAFM) and X-ray reflectometry (XRR). Generally, CP, CAFM, TAFM and XRR give similar thickness values except for the case of the more compliant samples measured by CP and CAFM. Moreover, the theoretically precise XRR technique gives significant standard deviation due to the layering of the DLC film. For those transparent samples, OP always gives an erroneous measurement. These metrological artefacts are compared to calculations of mechanical deformation (CP and CAFM), energy dissipation (TAFM) and thin film interferences (OP). The OP artefact is used to extract the film's refractive index, in good agreement with literature values. Finally, the comparative data obtained in this study also shows that the density and refractive index of the 10 nm thick films are constitutively lower than those of the 50 nm thick films. This scaling effect, which is consistent with known growth mechanisms for DLC, further complicates the measurement of thickness by optical techniques.

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1. Introduction

Diamond-like carbon (DLC) is a thin film material used as a protective coating in, for instance, gas barrier, data storage and biomedical applications [1–5]. In many cases, the carbon layer must have a specific thickness, often in the sub-100 nm range and the measurement of such ultra-thin thickness remains a considerable challenge. To date there has been some very accurate measurements of gate oxide thickness [6,7], using spectroscopic ellipsometry (SE), principally motivated by the shrinking size of semiconductor memory chips. Some traceable sub-nanometer metrological protocols using specialised atomic force microscopes (AFM) have also been developed [8]. Transmission electron microscopy (TEM) is also routinely used to measure epitaxial growth [9]. But, in the whole, these

investigations are focused on minimizing the instrumental uncertainties rather than questioning the operating principles of the thickness measurement technique. Moreover, comparative studies between various metrological techniques are scarce, especially for DLC films. Indeed, the availability of a range of such techniques is becoming a pressing necessity as DLC is applied to a growing variety of substrates such as metals, ceramics and polymers. Hence, the present paper will examine these issues for a range of ultra-thin (10–50 nm) carbon layers. We will compare a number of commercial techniques that are relevant to the applications of DLC materials, discussing their instrumental uncertainties and operating principles.

2. Background on techniques to probe ultra-thin thickness

The thickness of a thin film can be measured using a mechanical probe (a sharp tip) of either a contact profiler (CP) or a scanning probe microscope (SPM). The measurement relies on scanning the tip over a coated/uncoated step

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Table 1
Comparison between technique measurement techniques

Technique	Advantages	Disadvantages
CP	Large lateral range, for stress measurement high Z resolution	Sample may deform, not laterally resolved
OP	Large lateral range, for stress measurement high Z resolution, fast	Not laterally resolved, requires spectrum modelling (optical constant unknown)
CAFM	Laterally resolved	Sample may deform, small lateral range, slow
TAFM	Laterally resolved	Small lateral range, surface force artefact? slow
XRR	High Z resolution, layering information, no step edge required	Not laterally resolved, requires spectrum modelling (optical constant unknown)
SE	High Z resolution, layering information, no step edge required	Not laterally resolved, requires spectrum modelling (optical constant unknown), slow
TEM	Small resolution, direct estimate	Difficult sample preparation, inefficient scattering in carbon

CP = contact profilometry, OP = optical profilometry, CAFM = contact atomic force microscopy, TAFM = tapping atomic force microscopy and XRR = X-ray reflectometry, SE = spectroscopic ellipsometry, TEM = transmission electron microscopy.

edge pre-defined, for instance, by a lithographic lift-off technique and measuring the vertical displacement of the probe. Alternatively, one can shine radiation (photons, electrons, neutrons, etc.) onto the film and measure the transmitted or reflected signal. In the later case, the assumptions behind the operating principles vary greatly from one technique to the next.

In previous works [10,11], we have compared thickness measurements made by atomic force microscopy (AFM) and energy dispersive X-ray analysis (EDX). The latter technique relies on using an electron inelastic scattering law, an equation for X-ray production and a calibration procedure on a thick film of known thickness. Both techniques give similar thickness values, however, the EDX technique has a number of advantages. It does not require the definition of a step edge; it gives ‘through thickness’ data rather than topographical differences, it has lateral resolution and can be carried out anywhere on the film, hence spotting thickness heterogeneities wherever they occur. It also has several disadvantages. It relies on a calibration done with a thicker film, hence it must assume that thin and thick films have the same properties, which might not be the case. It is not very repeatable (± 5 nm) as carbon is a low density material which does not efficiently scatter electrons. Indeed, we find that all metrological techniques have shortcomings (see Table 1). This paper will focus on a few of them, of interest for DLC applications, namely, the CP, OP, CAFM, TAFM and XRR techniques.

3. Experimental

The samples examined here are hydrogenated amorphous carbon (a-C:H) and tetrahedral amorphous carbon (t-a:C) films prepared, respectively by filtered cathodic arc deposition (FCVA) and plasma enhanced chemical vapour deposition (PECVD) onto silicon substrates. The deposition conditions are described elsewhere [12]. For each method, we prepared two nominal thickness and, therefore called the samples 10P (10 nm by PECVD), 50P (50 nm by PECVD), 10F (10 nm by FCVA) and 50F (50 nm by FCVA). As will become apparent later, these nominal thicknesses, guessed from previous deposition batches, are not always accurate. The step edge is made by drawing a thin line of photoresist on the bare substrate, the film was then deposited above and around this line and after deposition the line was lifted from the substrate by sonicating the sample in isopropanol.

These samples were analysed by Raman spectroscopy and nanoindentation to check that their bonding character and mechanical characteristics did correspond to the expected a-C:H and t-aC forms [12]. Generally, the t-aC films are smoother [13], although the average roughness of our a-C:H films on Si is only around 1 nm [14]. Therefore, these surface topographies have a small effect on the measurement of thicknesses in the 10–50 nm range. In addition, two other samples were analysed; PMMA/glass and gold/polyimide. PMMA/glass corresponding, respectively to a compliant film on a stiff substrate and a stiff film on a compliant substrate.

The CP system is a Dektak 8 instrument, the load is applied by a coil assembly and the vertical displacement is measured by a linear variable differential transformer (LVDT) displacement gauge (0.1 nm resolution). The CP profiles are scanned at varying normal loads (0.03–10 mg) for few hundred microns distance across the step edge. The thickness is calculated by fitting straight lines through substrate and film windows. The CP system uses two conical diamond tips, 2.5 μm and 12.5 μm radii, as measured by Scanning electron microscopy (SEM).

The CAFM and TAFM measurements were carried out with a Veeco Dimension 3100 SPM system. The vertical piezo-drive of the instrument does not have interferometric or strain gauge control of the vertical height [15], and this obviously limits the repeatability of the topographic measurements. The calibration of this Z piezo-drive was done by either using a standard of known step height (Veeco PN 138367, 102.8 ± 0.9 nm) or by using the pre-contact interferences present in a force curve [16]. The two methods give the same Z sensitivity within 1% (22 nm/V). Using a set standard tilt of approximately 15° , we repeated those measurements for the full Z range of the scanner and found that the non-linearity was smaller than 1%. As well as this relative error, we found an absolute error of 10 mV for the photodetected voltage, corresponding to a 0.5–2 nm deflection error. This uncertainty comes from the thermal noise of the lever ($X \sim [3K_B \cdot T/k]^{1/2}$), the laser shot noise and

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