High-throughput characterization of film thickness in thin film materials libraries by digital holographic microscopy

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Abstract

A high-throughput characterization technique based on digital holography for mapping film thickness in thin-film materials libraries was developed. Digital holographic microscopy is used for fully automatic measurements of the thickness of patterned films with nanometer resolution. The method has several significant advantages over conventional stylus profilometry: it is contactless and fast, substrate bending is compensated, and the experimental setup is simple. Patterned films prepared by different combinatorial thin-film approaches were characterized to investigate and demonstrate this method. The results show that this technique is valuable for the quick, reliable and high-throughput determination of the film thickness distribution in combinatorial materials research. Importantly, it can also be applied to thin films that have been structured by shadow masking.

Keywords: thin films, high-throughput characterization, combinatorial materials science, optical methods

1. Introduction

The combinatorial approach facilitates the discovery and optimization of new functional materials (e.g. [1–4]). In this approach, following the synthesis of materials libraries, high-throughput, fast and reliable techniques are needed for characterizing the properties of the individual materials that were synthesized. Film thickness is a fundamental parameter, which is important both for adjusting the deposition process (e.g. deposition rate and uniformity) and calculating the volume of the film for later quantitative characterization. Contact stylus profilometry is the most common technique for making high-throughput thickness measurements. It uses a mechanically-loaded stylus for determining the height variation across the scanned area with nanometer resolution and requires a well-defined step fabricated, for example, by photolithography, thin film deposition and a lift-off process. However, mechanical scanning is time consuming. For example, a 300-point scan of a 4 inch wafer can take 6–10 h, or more, depending on the length and speed of each individual measurement, and the table movement time to reach each subsequent point.

Digital holographic microscopy (DHM) analyses the interference patterns of a light beam scattered by an object, with a coherent reference beam. It has recently been used to obtain three-dimensional (3D) surface information of micro-electro-mechanical systems (MEMS) [5,6]. Compared with other optical surface analysis techniques, such as white light interferometry (WLI), DHM has similar vertical and lateral resolutions. Although the maximum measurable step height in DHM is limited by the wavelength of the light source, the absence of high-precision moving components

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shows different stages of the object and a flat surface, the surface topography of the object. By capturing the interference pattern between the information and allows quantitative 3D profiling of the object.

Digital holography (DH) enables the construction of 3D images by analyzing the phase shift of the light reflected from objects. Compared with conventional holography, the digital nature of DH provides easy access to the depth information and allows quantitative 3D profiling of the object surface. By capturing the interference pattern between the object and a flat surface, the surface topography of the object is readily obtained in digital format after computer-assisted image reconstruction. A digital holographic microscope was developed for measuring the surface profile of patterned thin films (figure 1). In this instrument, a diode laser beam (660 nm) is spatially extended by a beam expander before being separated into two coherent beams by a beam splitter. The reference beam is reflected by a mirror. The object beam illuminates the sample. The scattered light from the sample returns through the beam splitter and combines with the reference beam. The resulting interference pattern (a hologram) is recorded using a consumer-grade digital single-lens reflex camera without the focusing lens (2592 x 1728 pixels, pixel size 8.6 x 8.6 µm²). As the formation of the hologram relies on the light reflected from the sample, a sufficiently reflective sample surface is required. The hologram is then analyzed using a computer program written in MATLAB. A numerical reconstruction method, based on Fresnel transformation, is used to obtain the intensity and phase information of the object beam from the recorded hologram [8]. While the reconstructed intensity shows a 2D picture of the sample, the phase contains the 3D surface profile information. However, because of aberrations in the optical components, the phase image is distorted, resulting in a distorted profile for the reconstructed surface. A double-exposure scheme is employed to solve this problem [5]: the recording of a reference hologram from a flat reference surface on the sample close to the area of interest is followed by subtracting its phase from the original phase. Figure 2 shows different stages of the reconstruction of images of a metallic island deposited on a Si substrate through a shadow mask. Figure 2(a) shows holograms of the island and of the substrate area next to it, and figures 2(b) and (c) are the corresponding reconstructed intensity and phase images. Both phase images contain the same phase front distortion coming from the optical system. After subtracting the background phase from the object phase, a distortion-free phase image of the island is obtained (figure 2(d)), and unwrapping the phase (figure 2(e)) yields a 3D surface profile (figure 2(f)). The maximum field of view is 10 x 10 nm² for which each pixel corresponds to an area of 8 x 8 µm². The lateral resolution can be improved by inserting a microscope objective lens in front of the sample [6]. The vertical resolution of the DHM is on the order of 10 nm [6]. The entire imaging procedure, including the hologram acquisition and 3D surface profile reconstruction, is completed within seconds. Such fast measurements are ideal for the high-throughput thickness mapping of semiconductor wafers (materials libraries). Furthermore, the DHM is

|                     | Lab-built DHM | SP  | WLI      | AFM  |
|---------------------|---------------|-----|----------|------|
| Vertical resolution | ~10 nm        | Higher | Comparable or higher | Higher |
| Imaging speed       | <1 s          | Slower | Comparable or Higher | Much slower |
| Equipment cost      | <USD 3000     | Much higher | Comparable | Much smaller |
| Field of view       | 1 cm²         | Comparable | Yes | No |
| Applicable to non-reflective surfaces? | No | Yes | Yes | Yes |

Figure 1. Schematic diagram of the digital holographic microscope.

In this study, DHM was applied to the high-throughput thickness mapping of thin films patterned by shadow masking [7]. In the case of deposition at temperatures above 200°C, photoresist layers for lift-off are not applicable and only shadow masking allows the direct patterning of thin films. In contrast to film thickness steps prepared by a lift-off process, the steps resulting from shadow masks are more gradual and therefore difficult to measure in an automated high-throughput manner by stylus profilometry, whereas DHM is suitable for such measurements. Therefore, this fast and non-contact technique is expected to play an important role in high-throughput thickness characterization of thin film materials libraries.

2. Digital holographic microscopy

Digital holography (DH) enables the construction of 3D images by analyzing the phase shift of the light reflected from objects. Compared with conventional holography, the digital nature of DH provides easy access to the depth information and allows quantitative 3D profiling of the object surface. By capturing the interference pattern between the object and a flat surface, the surface topography of the object.
equipped with a computer-controlled x–y translation stage that allows the automatic scanning of a 4 inch wafer. Other sample geometries are also possible. Scanning and image acquisition are controlled by a computer via LabVIEW software.

3. Preparation of thin-film materials libraries

Combinatorial thin-film materials libraries can be prepared in different ways. In this study, we used the wedge-type [1–4] and mask-manipulation [9,10] deposition techniques in a combinatorial magnetron sputter system (DCA, Finland). In the wedge-type approach, films are deposited on a substrate by controlling the speed and position of a moving shutter located between the target and substrate, creating a wedge-shaped thickness gradient. Typically the substrate would then be rotated to a different angular orientation with respect to the shutter, followed by the deposition of another material, for instance by 180° to make opposing binary wedges, or by two rotations of 120° to make ternary wedges. A multilayer film is obtained by rotating the substrate back to the starting orientation and repeating the process of gradually opening the shutter while sputtering each material. To evaluate the DHM technique, in this study we deposited only a single wedge layer of Cu (figure 3(a)). The film was patterned by depositing Cu through a micromachined Si shadow mask. The isolation between neighboring samples is beneficial for non-destructive thickness measurements. Discrete samples also allow calculation of the volume of individual samples, which is essential for measuring volume-dependent material properties (e.g. magnetization). The best definition of the mask pattern on the substrate is realized when the mask is directly clamped to the substrate, i.e., the mask-substrate distance is minimized, and ideally no material is sputtered underneath the mask. However in practice this is very difficult to realize, with gaps of even a few microns leading to deposition under the mask and blurring of the sample edges.

In the mask-manipulation approach, the mask is held in a carrier, which is placed on top of the substrate. A computer-controlled robotic arm handles the mask carrier inside a vacuum chamber so as to place or remove the mask, exchange it with different masks that can be stored inside the machine, or to lift the mask, rotate it in 90° increments and put it back on the substrate. This allows the application of various masks and mask orientations during the same deposition series without breaking the vacuum. By rotating a particular mask or by using masks with different patterns during the deposition, discrete multilayer materials libraries can be fabricated [9]. Since the mask is held in a carrier to enable these manipulations, the mask-substrate distance is larger than what can be achieved by clamping, typically being in the range of tens, to a few hundreds of micrometers.

In this study, the same mask used in the clamped approach was held by a carrier during the deposition of a wedge layer of Cu (figure 6(a)). However, to evaluate the ‘worst case’ situation, the mask-to-substrate distance was
allowed to be as large as approximately 300 µm. This results in a wafer with patterned Cu films having a thickness wedge, similar to that produced by the first approach. However, because of the larger mask-substrate distance, the edges of the film islands are less sharp due to deposition into the gap under the mask.

4. High-throughput thickness mapping

Figures 3(b) and (c) show the reconstructed intensity image and 3D surface profile of one of the Cu islands prepared by the wedge-type approach. The thickness of the circular

Figure 3. (a) Optical image of a wafer prepared with a shadow mask which was clamped to the substrate to define the dot pattern, and a moving shutter to create a thickness gradient. (b) Intensity and (c) 3D surface profile of the deposited Cu films measured by DHM. (d) A cross section of the 3D profile.

Figure 4. A thickness map of the patterned Cu films deposited by the wedge-type approach through a Si shadow mask.

Figure 5. (a) Thickness variation in the rows, i.e. direction parallel to the shutter movement vector. (b) Thickness variation of the Cu islands across the columns (perpendicular to the direction of the shutter movement).
film patterns can be obtained by plotting a line profile across the surface feature (figure 3(d)). Because the mask was directly clamped onto the wafer, the edges of the islands are relatively sharp. A program written in MATLAB was developed to automatically perform the line scan and output the corresponding thickness. Once the image acquisition of the whole wafer is finished, the program analyses each circular island one by one and stores the thickness values in a spreadsheet. The overall time of image acquisition and data analysis for a 4-inch wafer is less than 15 min.

Figure 4 shows a thickness distribution of deposited Cu over a 4 inch wafer. The thickness decreases from the top to the bottom of the figure because a shutter was gradually opened from the 11th row to the first row during the deposition. The thickness variation along the 5th column is plotted in figure 5(a). Although the shutter was opened at a constant speed, the thickness distribution is not linear. This could be explained by the geometrical limitations of the sputter chamber: the limited size of the sputter target coupled with the target to substrate spacing produces a slightly inhomogeneous deposition profile across the substrate wafer. This inhomogeneity is also observed across the columns (figure 5(b)); it is more pronounced near the edge of the wafer and in the middle rows (4th to 6th rows).

For the wafer prepared using the mask-manipulation (unclamped mask) method, the mapping revealed a similar thickness variation (not shown) to that of figure 4. However, the lateral size of the thin-film circles is larger (figure 6(a)) because of the unwanted deposition under the mask. Consequently, the edges of the samples are sloped rather than step-like. This can be observed in the 3D surface profile and the corresponding line scan (figures 6(c) and (d)). The blurring at the edges inhibits thickness measurement by conventional automated stylus profilometry, because the difficulty in defining the exact top and bottom of the film, coupled with the curvature of the wafer over long scan lengths makes large uncertainties in the baseline leveling and definition of the step height. In DHM, the background is already subtracted during the image reconstruction, and thus the substrate bending effect is automatically compensated.

5. Summary

High-throughput characterization of film thickness in thin-film materials libraries by digital holographic microscopy was demonstrated. The fast and non-contact nature of this optical technique is advantageous over conventional automated stylus profilometry. The simple design and low cost of a holographic instrument makes DHM a promising alternative to optical interferometry. This technique can be applied to map the thickness of films deposited by various methods.

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