

# Correlative Microscopy in Life and Materials Science

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### Delamination buckling of thin metallic film studied by digital holographic light microscope interferometry

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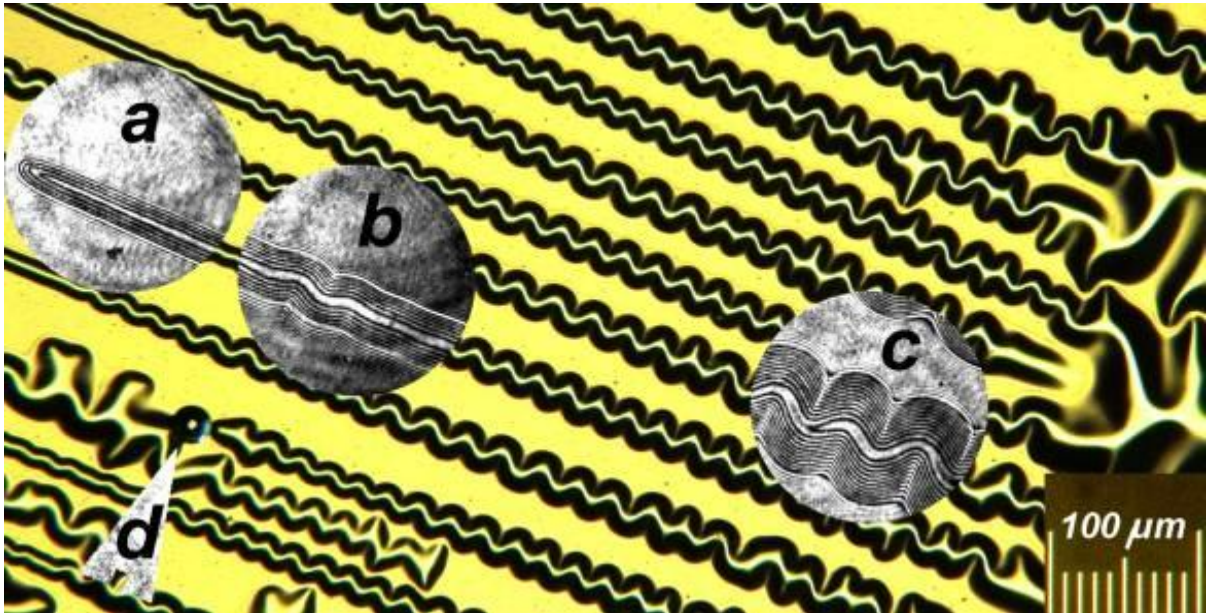
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Here, we present an application of our Digital Optical Holographic Microscope (DOHM) [1] in the study of morphology and topography of buckling patterns that can be observed on thin film, during or after its delamination from thick substrate [2]. For deposition of tungsten thin films by magnetron sputtering [3], it is well known that argon pressure significantly affects the film internal stress. In the case of tensile residual stress a network of through-thickness cracks forms in the film, while the residually compressed thin films may delaminate from substrate and buckle [4]. As it is shown in Figure 1, buckling of thin tungsten film can result in a number of topographical patterns such as disordered surface wrinkles, regular herringbone, straight-sided or telephone cord buckles and circular blisters [5]. The associated mechanics has been studied [6] and evaluated for a number of cases of all types of buckles (circular, straight-sided, ...), but for the telephone cord (TC) ones, more precise microscopic observation (complemented by topographic measurements) are still needed for testing the corresponding models [6]. Delamination usually start as straight-sided linear blisters, but then deviate to the TC periodic wavy geometry due to the fact that the compressive residual stress in the film is biaxial. In general, buckling profiles can be characterized depending on scale, by mechanical or laser-scanning profilometry, optical interferometer microscopy, or by using an atomic force microscope [7].

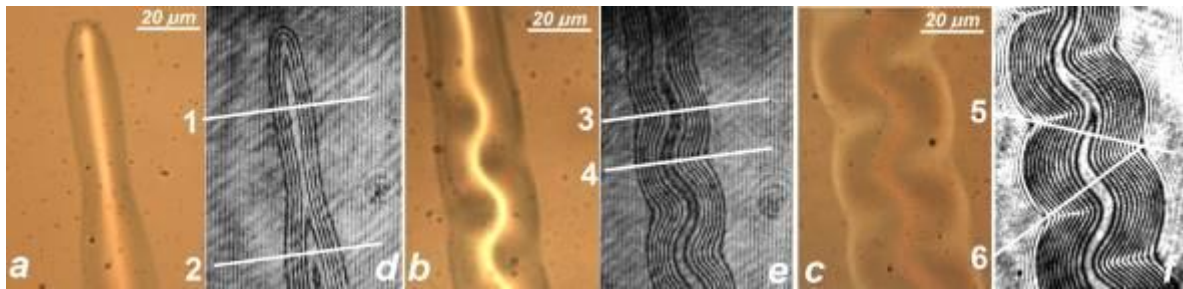
Our DOHM is a home-adapted commercial metallurgical instrument. It is an extended set-up that provides simultaneous or alternative white-light or monochromatic illumination. A parallel arm is added for the interference of object and reference beams, plus an extra arm for hologram reconstruction. The later is fitted by a LCOS display operated in real-time via PC; coupled pair of CCD cameras enables acquisition of the corresponding images [1]. Two pairs of independent sources (laser plus LED) are implemented: one for imaging, and one for hologram reconstruction. This set-up provides traditional microscopic imaging in succession with holographic imaging and the real-time optical reconstruction, as well as processing and correlation of digital images. Figure 2. displays traditional microscopic and correlative holographic interferometry data ( $\lambda/2$ -fringes) of the very same object area.

White-light microscopy of top surface of highly compressed tungsten thin film clearly reveals (at lower magnification: <500X), lateral morphology of the buckling patterns Figure 1. At higher magnification (>500X), reduction of focus depth affects imaging contrast, so that the lateral as well as vertical wrinkles` features can hardly be measured down to submicron precision, as is represented in Figure 2. By imaging and optical processing in holographic mode, one can display interferometric fringes patterns that reveal lateral and vertical buckling features with precision:  $\Delta l \approx \Delta h \approx 0.3 \mu\text{m}$ . Correlation of traditional microscopy and holographic interferometry of the very same imaging area can provide data basis for quantitative 3-D modeling of TC buckling morphology and associated stress-strain mechanics.

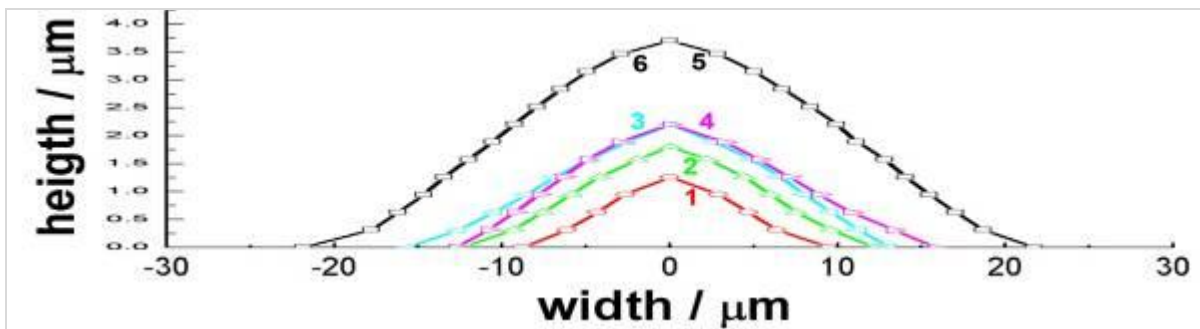
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**Figure 1.** Medium magnification (200X) top-view image (white-light microscopy mode) of W-thin film on substrate revealing straight-sided and TC buckles (insets: a, b, c), as well as disordered surface wrinkles and a circular blister (arrowhead: d). Insets (magnification: 500X) represent interferometric fringes recorded by monochromatic-light holography mode).



**Figure 2.** Correlative images of straight and TC delamination buckles in W-thin film (like in Fig.1.), with the corresponding interferometric fringes patterns (d, e, f) as recorded by reconstruction and optical processing of holographic images. White lines (1 to 6) indicate traces for height profile characterization. Consecutive dark fringes reveal trajectories of constant height in steps of  $\Delta h \approx 0.3 \mu\text{m}$ . Observation magnification (1000X).



**Figure 3.** Height profiles of straight-side and telephone-cord buckles from Fig.2. Labels 1 to 6 correspond to traces marked in Fig.2. Profile symmetry along the median line for straight buckle starts to deviate with lateral waviness in the case of TC buckles as revealed for traces 3 & 4.