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High resolution determination of local residual stress gradients in single- and multilayer thin film systems

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ABSTRACT

Residual stresses and stress gradients are of great importance in all thin film systems, as they critically influence the structural stability and functionality, and thus the lifetime, of the concerned devices. In this study, an improved ion beam layer removal method is developed to determine the stress distribution in copper- and tungsten-based thin film systems. Cantilevers were prepared from single, bi- and tri-layer systems with an individual layer thickness of 500 nm using focused ion beam machining. Subsequently, residual stress profiles were determined with a depth resolution of 50 nm, employing the ion beam layer removal method. We observe that the evaluated average film stresses correspond to state-of-the-art X-ray diffraction measurements. However, depending on the layer order, different stress profiles with strong stress gradients evolve, and pronounced changes in residual stress occur across an interface within only few grains. These novel findings have profound implications when addressing the interface adhesion, fracture properties and reliability of novel thin film systems, as well as interface dominated materials in general.

1. Introduction

Patterned multilayer thin film structures are commonly used in microelectronic applications, simply because single material layers are not able to perform on the same level in terms of required functionality [1]. Notably, besides the primary functionality, the structural stability has to be fulfilled within such patterned multilayer systems as well. In such thin films residual stresses are one of the major challenges to be considered. They can cause damage, fatigue, delamination and cracking of the films, thereby limiting the service life of the whole structure [2]. Components are continually getting smaller, but at the same time new technologies, such as for example the through silicon via (TSV) technology and 3-D integration [3,4], are being developed. Therefore, it is increasingly important to measure residual stresses at the same dimensional scale as that which is used in industrially manufactured devices. With conventional methods such as X-ray diffraction (XRD) or wafer curvature measurements [5–8], the residual stresses in thin films can only be determined globally. Thus, techniques that make a high local resolution for such measurements possible are highly relevant for the modern microelectronics industry. In recent years, several methods to analyse the residual stresses and stress gradients in single and multilayer thin film systems have become available. One way is to perform cross-sectional nanodiffraction experiments [9,10] using finely focused synchrotron radiation at large-scale facilities to determine the stress distribution in thin films in a depth-resolved manner. Another X-ray technique capable of deducing stress gradients proposes measurements at fixed penetration/information depths [11]. Only single layers can be analysed with this method. Another general limitation of X-ray based techniques is the fact that they are only applicable to crystalline materials. A completely different approach suggests micromachined cantilevers where the stress profile is measured through interferometric profilometry [12,13]. Such approaches allow fabrication of many samples by lithographic means, but come at the cost of limited flexibility regarding the material systems that can be processed lithographically. A third group of methods for the local determination of residual stresses comprises incremental focused ion beam (FIB) milling to determine material relaxation due to stress redistribution upon material removal. Different measurement geometries such as slits [4,14,15], pillars [16–19] and micro cantilevers [20,21] are available. These methods are generally applicable to crystalline and amorphous, as well as to textured materials, and provide data from specific positions of a film/sample or local features on a patterned structure. In

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Table 1
Overview of investigated material systems.

Sample	1st Layer [nm]	2nd Layer [nm]	3rd Layer [nm]
Cu	Cu 483 ± 4	–	–
W–W	W 504 ± 4	W 490 ± 1	–
W–Cu–W	W 486 ± 3	Cu 492 ± 1	W 483 ± 3
Cu–W–Cu BB1[a]	Cu 483 ± 5	W 477 ± 4	Cu 452 ± 2
Cu–W–Cu BB2	Cu 484 ± 4	W 478 ± 1	Cu 474 ± 5

[a] BB . . . Bending Beam.

Table 2
Deposition and material parameters for Cu and W.

Material	Power [W]	Deposition time [s]	Purity [%]	Target diameter [mm]	Target thickness [mm]
Cu	90	4550	99.99	76.20	3.00
W	125	10280	99.95	76.20	3.18

the case of pillars and slits, the deformation field is analysed using digital image correlation. Therefore, a sufficiently structured surface in the region of interest is required. For ring milling of pillars using a simple computation of the stress field, the height h, diameter d and film thickness t should be equal to ensure complete strain relief [17]. For the more general case of d > t additional FEM analysis is necessary to determine the average stress in the film [18]. As outlined by Sebastiani et al. [19], owing to the calculation procedure, the accuracy of the stress values drops significantly for cutting depth values higher than 40% of the desired pillar height. Thus, when aiming at analysing commonly used thin films of only a few 100 nm thickness, extensive FEM analysis is unavoidable to determine residual stresses.

Therefore, in this paper, we demonstrate the determination of residual stresses and stress gradients using micro cantilevers analysed with the ion beam layer removal (ILR) method [20,21], an incremental layer removal method which was further improved and adapted for use in sub-micron multilayer thin film systems. This method has a high depth resolution of 50 nm in the individual sublayers and a lateral resolution within a few µm, with no limitations concerning film thickness. Thin film systems can be analysed analytically, with an error in residual stress values that is independent over the whole film thickness, and without the need for complex computer image analysis.

The technique is applied to determine the residual stresses in copper (Cu) and tungsten (W) based thin film systems. Both materials are of high interest in microelectronics applications in the context of thermal management and electric conductivity, as they show a good combination of high thermal and electrical conductivity for Cu and, in the case of W, a rather low thermal expansion coefficient for a metal, which is only a factor two higher than for Si [1,22]. To demonstrate the capability of the improved ILR-method, we compare the obtained results to standard lab-based X-ray diffraction residual stress measurements and emphasize the importance of pronounced stress gradients at interfaces that are not accessible by standard X-ray diffraction.

2. Experimental

As indicated above, the focus of the present paper was to study Cu and W films, as they are of high importance in the microelectronics industry. We investigated single layers as well as multilayer systems, the latter for different stack configurations, as there should be a difference in film growth depending on the substrate or previous layer, and thus a change in the residual stress profiles.

Four different film systems were systematically investigated. The stack configurations including precise film thicknesses are given in Table 1. We start with a single Cu film of about 500 nm on a silicon substrate. Then a sample consisting of two W layers was investigated to study the influence of an interrupted deposition. Following, a tri-layer sample consisting of W–Cu–W was prepared, to determine the stress distribution in a configuration with a soft interlayer. Finally a Cu–W–Cu sample was deposited to analyse a reversed stack configuration. All samples were deposited at room temperature on a (100) oriented single crystalline Si wafer with a thickness of 525 µm in a Mantis Sputter System (Mantis, Thame, United Kingdom) with an Ar flow of 45 sccm. Direct current sputtering with three different sputtering targets (Cr, Cu and W) was used in the same deposition chamber. Therefore, the vacuum was never broken when changing from one film material to the other. Before putting down the actual layers to be studied, in all cases 10 nm Cr were deposited as seed layer, to stimulate the growth of either Cu or W on the Si wafer. Further deposition and material parameters are given in Table 2. The films consist of globular grains with a grain size ranging from 60 to 70 nm as determined by a line intercept method from scanning electron microscopy (SEM) images (not shown here).

2.1. Sample preparation

In order to determine the residual stress profiles of thin films on a local scale, micro cantilevers were prepared. The material to be studied was extracted from an area in the middle of the wafer to avoid any influence from inhomogeneous film deposition near the wafer edge. The sample was constructed as follows: First, a narrow freestanding fillet (see Fig. 1b) on the sample is prepared using broad beam ion milling [23]. For this preparation step a

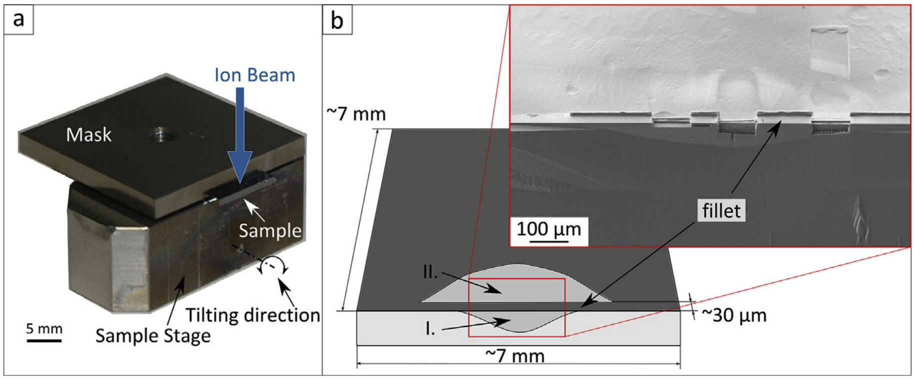

Fig. 1. a) Optical photograph of the sample setup used in the Cross Section Polisher. b) Schematic illustration of the ion-polished lamella with an SEM detail of the actual sample.